



Les systèmes métallogéniques hydrothermaux à tungstène et métaux rares (Nb-Ta-Li-Sn) de la période Jurassique-Crétacé au sud de la province de Jiangxi (Chine)

Hélène Legros

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Hélène Legros. Les systèmes métallogéniques hydrothermaux à tungstène et métaux rares (Nb-Ta-Li-Sn) de la période Jurassique-Crétacé au sud de la province de Jiangxi (Chine). Sciences de la Terre. Université de Lorraine, 2017. Français. NNT : 2017LORR0266 . tel-01822836

HAL Id: tel-01822836

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Ecole Doctorale RP2E « Ressources, Procédés, Produits, Environnement »
Collegium Sciences et Technologies
Université de Lorraine CARNOT ICEEL – CARNOT BRGM
UMR 7359 GeoRessources

THESE

Présentée pour l'obtention du grade de
Docteur de l'Université de Lorraine
Mention « Géosciences »

Par **Hélène LEGROS**

Les systèmes métallogéniques hydrothermaux à tungstène et métaux rares (Nb-Ta-Li-Sn) de la période Jurassique-Crétacé au sud de la province de Jiangxi (Chine)

Tungsten and rare metal (Nb-Ta-Li-Sn) hydrothermal metallogenic systems of the Jurassic-
Cretaceous period in the southern Jiangxi province (SE China)

Soutenance publique le 14 Décembre 2017

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Résumé

Le tungstène est défini comme “ressource minérale critique” par la Commission Européenne. En effet, plus de 60% des réserves mondiales sont localisées en Chine où plus de 80% de la production mondiale est distribuée. La province de Jiangxi, située au sud-est de la Chine, dans le bloc Cathaysia, représente 90 % des réserves en tungstène chinoises. Ces ressources sont principalement observées sous la forme de veines hydrothermales à quartz-wolframite-cassiterite peri-granitiques, associées à des granites jurassiques et crétacés. Cette thèse a pour but de (i) développer des traceurs pétrographiques et minéralogiques de processus minéralisateurs à travers des études paragénétiques détaillées et la géochimie des micas lithinifères, (ii) définir les paramètres nécessaires à la précipitation de la minéralisation par une étude d’inclusions fluides et, (iii) de développer des approches de datation associées à ces systèmes. Ce travail est basé sur l’étude des gisements hydrothermaux à W-Sn de Maoping et Piaotang, tous deux situés dans le district de Dayu (sud de la province de Jiangxi), par des approches de pétrographie et minéralogie détaillées, de géochimie minérales et isotopiques, d’inclusions fluides et géochronologiques.

L’étude détaillée des gisements de Maoping et Piaotang démontre l’implication de processus régionaux permettant la mise en place de ces gisements à W-Sn « géants » entre 165 et 130 Ma (Jurassique-Crétacé) dans le sud de la province de Jiangxi. L’étude géochimique des micas lithinifères et d’inclusions fluides convergent vers un modèle impliquant des fluides multiples se chevauchant dans le temps et associés à plusieurs épisodes distincts de mise en place de minéralisations en métaux rares. Au sein du communément appelé « stade à silicate-oxydes » où l’essentiel de la minéralisation se met en place. L’étude d’inclusions fluides a permis de caractériser des paramètres distincts de précipitation des minéraux de gangue et de la minéralisation et donc une cristallisation séquencée. Le cas exceptionnel des gisements de Maoping et Piaotang permet de définir deux processus fluides comme seul responsables de la précipitation de la minéralisation dans ces gisements « géants » : la différenciation magmatique de granites peralumineux et des processus de mélange. Le premier âge sur wolframite (U-Pb) a permis de montrer que la minéralisation en tungstène se met en place aux alentours de 160 Ma, antérieurement à la plupart des âges obtenus sur les minéraux de gangue datés dans cette zone et défini alors une remise à zéro majeure des systèmes isotopiques par de multiples circulations fluides entre 150 et 155 Ma. De plus, les stades post-minéralisations ont pu être définis pour la première fois et révèlent l’implication de magmatisme peralcalin impliqué dans la précipitation de minéralisations à Nb-Ta-Y-REE aux alentours de 130 Ma. A la lumière de cette observation, cette thèse s’est aussi tournée vers le développement de méthodes de datation in situ sur columbo-tantalite.

Abstract

Tungsten is defined as a “critical mineral resource” by the European Commission. Indeed, more than 60% of the world reserves are located in China where more than 80% of the worldwide production is distributed. The Jiangxi province, located in the southeastern part of China, in the Cathaysia block, represents 90% of the Chinese tungsten resources. These resources are mainly observed as peri-granitic quartz-wolframite-cassiterite hydrothermal vein systems associated to Jurassic and Cretaceous granites. This thesis aims to (i) develop mineralogical and petrological tracers of ore-forming processes through detailed paragenetic sequences and geochemistry of Li-micas, (ii) distinguish prerequisite parameters involved in ore-forming processes by fluid inclusions studies and, (iii) develop dating approaches associated to these systems. This work is based on the study of the Maoping and Piaotang W-Sn hydrothermal deposits located in the Dayu district (southern Jiangxi), using petrography and detailed mineralogy, mineral and isotope geochemistry, fluid inclusions and geochronology.

The detailed study of the Maoping and Piaotang deposits demonstrate the implication of regional processes emplacing giant W-Sn deposits between 165-130 Ma (Jurassic-Cretaceous) in the southern Jiangxi province. Li-micas geochemistry as well as the fluid inclusion studies converge to multiple overlapping fluids associated to several and distinct rare-metal mineralizing stages. Within the commonly called “silicate-oxide” stage where main of the ore-bearing minerals occur, fluid inclusions permitted to characterize distinct parameters of precipitation for the gangue and W-Sn minerals and therefore a non-coeval crystallization. The exceptional cases of the Maoping and Piaotang deposits allow to define peraluminous magmatic differentiation and mixing processes as only prerequisite for the formation of these giant deposits. First U-Pb dating of wolframite in this region show that the W mineralization formed at ca. 160 Ma, prior to most ages obtained on gangue minerals in the area, defining a major resetting of isotopic systems due to multiple fluid circulations around 150-155 Ma. Moreover, post-“silicate-oxide” stages have been defined for the first time and reveal the implication of peralkaline new fluid sources involved in the precipitation of Nb-Ta-Y-REE minerals at ca. 130 Ma. In the light of these results, this thesis gives new developments for *in situ* direct dating of ore-bearing minerals such as columbo-tantalite.

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Remerciements

A la lumière de ces trois ans de thèse au sein du laboratoire GeoRessources, je tiens à remercier un grand nombre de personnes qui m'ont apporté leur soutien professionnel et/ou personnel, tout au long de ce périple qui restera gravé dans ma mémoire.

Tout ceci a commencé lors de mon stage de fin d'étude qui m'a conduite dans ce laboratoire sous la supervision de Michel Cuney et Julien Mercadier. Merci à eux d'avoir pris le temps de m'introduire au monde de la recherche, ce stage a été l'élément déclencheur qui m'a conduite à ce qui fut une formidable expérience de trois ans.

Je remercie tout particulièrement Julien Mercadier qui a été le leader de ce projet de thèse, qui m'a soutenu durant cette thèse, qui m'a poussé dans l'espoir de devenir un jour chercheuse et qui a monté la collaboration avec le BRGM au travers des financeurs Carnot ICEEL et BRGM, que je remercie également. Cette collaboration m'a notamment permis de rencontrer Nicolas Charles qui fut mon superviseur au sein du BRGM. Merci à toi Nicolas pour ta bonne humeur constante, ton soutien et les fous rires en Chine ! J'espère que cette collaboration perdurera. Je remercie d'ailleurs l'ensemble des collaborateurs chinois qui nous ont permis de nous rendre en Chine pour cette expérience inoubliable : Ru-Cheng Wang, Xu-Dong Che et Zeying Zhu. Enfin, je te tiens aussi à remercier Marc-Yves Lespinasse qui a accepté de devenir mon directeur de thèse et a ainsi rendu ce projet possible.

Durant cette thèse, j'ai eu le privilège de pouvoir satisfaire ma curiosité et de développer des aspects scientifiques divers qui m'ont amené à voyager et rencontrer de nombreux chercheurs qui m'ont suivie, soutenue et appris beaucoup, merci à vous. Parmi eux, j'aimerais notamment remercier Christian Marignac, Antonin Richard et Rolf Romer, qui ont pris le temps de m'enseigner une partie de leur savoir. Je vous suis très reconnaissante. Travailler avec vous fut un plaisir et une superbe expérience. Je m'attacherai à conserver au mieux la rigueur scientifique que vous avez su m'enseigner.

D'autre part, je remercie grandement l'ensemble de mes stagiaires : Thomas Tabary, Renaud Mulot, Alexis Locatelli et Simon Hector pour m'avoir épaulé durant cette thèse et avoir fourni un travail de qualité qui m'a permis d'avancer.

Je tiens aussi à remercier l'ensemble des chercheurs et co-auteurs de ces chapitres pour leur aide et leur implication, toutes les personnes avec qui j'ai travaillé au SCMEM, au CRPG mais aussi à GeoRessources et qui m'ont notamment beaucoup aidé d'un point de vue analytique. En particulier, je tiens à remercier : Christian Marignac, Nicolas Charles, Ru-Cheng Wang, Julien Mercadier, Michel Cuney, Antonin Richard, Marc-Yves Lespinasse, Alexandre Tarantola, Kalin Kouzmanov, Torsten Vennemann, Laurent Bailly, Rolf Romer, Marc poujol,

Remerciements

Matthieu Harlaux, Alfred Camacho, Johan Villeneuve, Etienne Deloule, Stijn Dewaele, Xudong Che, Marieke Van Lichtervelde, Marie-Camille Caumon et Chantal Peiffert.

Enfin, je remercie l'ensemble des membres de mon jury de thèse pour avoir pris le temps de lire ce manuscrit et de s'être déplacé pour ce qui a été un grand moment pour moi. Je tiens donc à remercier David Dolejs, Philippe Boulvais, Marieke Van Lichtervelde, Nicolas Charles, Gaston Giuliani, Rolf Romer, Stanislas Sizaret et Christian Marignac.

Sur une pointe plus personnelle, je tiens à remercier l'ensemble des thésards et post-doc qui ont croisé ma route tout au long de cette aventure. Je ne le dirai jamais assez, mais cette réussite je vous la dois aussi. Vous avez été là dans les bons et les mauvais moments, toujours à l'écoute de mes blagues douteuses et de mes coups de gueule. C'est un plaisir de venir chaque jour vous retrouver au labo et partager ces journées avec vous. C'est cette ambiance qui m'a permis de tenir et de me tirer vers le haut. Je n'aurais jamais pensé pouvoir vivre une telle expérience. Vous êtes plus que mes collègues, vous êtes mes amis.

Parmi eux j'aimerais notamment remercier Roland Salardon, qui m'a faites mourir de rire aux pauses clopes avec ses histoires à la « père castor » ; Matthieu Harlaux, avec qui j'ai passé des moments difficiles mais aussi de rire à G'nève et en Chine (j'espère que la pipe va bien) ; Marion Grosjean, pour avoir marqué ma voiture à vie (elle va bien !) ; Maïa Reilh, pour avoir fait concurrence à ma touffe ; Raphael Vasseur, pour ta sagesse de dandy et nos discussions moto ; Héloïse Verron, pour avoir partagé la garde de notre petite Chewie ; Joséphine Gigon, pour sa folie qui me fait toujours autant rire et sa bonne humeur constante ; Maxime Dargent, pour sa patience à chaque fois que je viens de le déranger dans son bureau alors qu'il n'a rien demandé ; Benoit Quesnel, qui a toujours une bonne histoire à raconter et notamment celle de son tatouage ; Yoram Teitler, pour avoir proposé mainte fois de venir se les cailler pour ramasser des champignons (je viendrai un jour !) ; Eleonora Carocci, pour sa touche exotique qui sait toujours faire rire ; et bien d'autres...

Je tiens aussi particulièrement à remercier mes co-bureaux Guillaume Barré et Christophe Scheffer qui m'ont soutenue et supportée pendant ce long périple. J'ai passé de très bons moments de rigolade avec vous. D'autre part, merci d'avoir accepté « mon œuvre » au sein de votre bureau, un travail de longue haleine qui ne s'est toujours pas écroulé ! Bientôt trois cent boîtes de café, un reflet de cette période riche en émotions !

Un merci tout particulier à mes deux compagnons de rédaction et de soutenance : François Turlin et Pierre Martz (#Chaton). Cette dernière ligne droite a été très difficile mais on a réussi ensemble et je suis contente d'avoir vécu cette expérience avec vous ! Je n'aurais jamais été capable de pondre ça sans vous, ça ne fait aucun doute. Vous êtes deux personnes que j'apprécie énormément et je n'oublierai jamais ces moments que nous avons passés

Remerciements

ensemble. Je n'oublierai pas les craquages et blagues douteuses de fin soirée en bas de la 3B, les diners à la cafet' avec l'ensemble de la gamme de plats à réchauffer Super U, les cafetières lancées la nuit, les parties de cartes, les kinder pingui, les coups de gueules, les rush, les fous rires, les répliques et chansons cultes, les angoisses et bien d'autres choses qui sont maintenant de bons souvenirs.

Enfin, merci à Julien Bonnet, mon pilier durant ces trois ans. Tu as su m'écouter tout au long de cette thèse et me reconforter lorsqu'il le fallait. J'ai eu la chance de te connaître lors de cette thèse et cette amitié restera ! Merci pour avoir passé environ une journée sur deux dans mon salon à me faire découvrir le terroir, à m'avoir faites rire, à avoir partagé des choses douteuses, mais aussi pour avoir regardé des navets avec moi et m'avoir accompagnée aux quatre coins du monde ! Une magnifique rencontre.

Merci à tous.

Introduction

Cette thèse fait partie d'un projet collaboratif entre l'Université de Lorraine et le Bureau des Ressources Géologiques et Minières (BRGM). Ces deux laboratoires ont dédiés de nombreuses années à l'étude des gisements à tungstène (W)-étain (Sn) en Europe (Massif Central, Panasqueira, les Cornouailles...) et dans le monde. Cette thèse a pour but de proposer de nouvelles contraintes et outils pour la compréhension de ces gisements à travers l'étude de la province la plus riche en W-Sn au monde : la province de Jiangxi (Chine), en collaboration avec le State Key Laboratory for Mineral Deposits Research (Nanjing, China). Afin de répondre à cette problématique, cette thèse de doctorat comporte quatre objectifs scientifiques :

(i) **Le premier objectif** est de définir les aspects pétrographiques et minéralogiques de ces gisements afin de combler le manque d'informations présentées dans la littérature. La réalisation d'une étude pétrographique détaillée est cruciale pour la compréhension de la formation de ces gisements et un préalable à toute étude plus approfondie.

(ii) **Le deuxième objectif** concerne l'identification de sources possiblement impliquées dans les stades pré-, syn- et post-minéralisation. Les micas sont connus pour être de bons traceurs de processus magmatiques et de bons transporteurs de métaux rares tels que le W, Sn, Nb et Ta. Les micas sont ubiquistes dans les gisements à W-Sn et cristallisent généralement sous forme de plusieurs générations successives et représentent donc un marqueur potentiellement de processus hydrothermaux et de sources pouvant ensuite être liés à une étude d'inclusions fluides.

(iii) **Le troisième objectif** est de caractériser la nature des réservoirs fluides impliqués dans la formation des gisements à W-Sn chinois. La nature des fluides minéralisateurs est encore débattue dans la littérature mondiale. L'implication évidente d'un fluide météorique tend à cacher l'information concernant les fluides porteurs de métaux qui pourraient être de

nature magmatique, directement exsolvés d'un magma granitique ou externes, lixiviant le granite sous-jacent.

(iv) **Le quatrième objectif** est de définir l'âge de mise en place des minéralisations hydrothermales à W-Sn dans le sud de la province du Jiangxi. Bien que de nombreuses études de datation ont été effectuées à ce jour, les analyses de datation directe sur les minéraux porteurs de minéralisation restent rares voire inexistantes sur wolframite. La complexité de la paragenèse exposée associée à de multiples impulsions fluides pouvant altérer les systèmes isotopiques met en avant la nécessité d'effectuer des études de datations dites « directes ». L'absence de standards appropriés à W (wolframite), Sn (cassitérite) et Nb-Ta (columbo-tantalite) pour des méthodes de datation in situ soulève la nécessité de développer de nouveaux standards afin de valoriser les méthodes de datation directes sur des phases minéralogiques de tailles réduites.

Ces objectifs ont pour but de répondre à trois grandes questions : (i) Quels sont les processus responsables de la mise en place ces gisements géants ?, (ii) Pourquoi ces gisements se sont-ils mis en place spécifiquement dans cette région et à cette période ? et, (iii) Pourquoi cette région, en comparaison à de nombreuses autres régions à W-Sn, a-t-elle été si productive ?

Ce manuscrit de thèse s'organise en trois parties, subdivisées en six chapitres rédigés comme des articles indépendants.

Le premier chapitre (**Chapter 1**) n'a été incorporé dans aucune de ces grandes parties. Ce chapitre a pour but de proposer une revue du contexte géologique du Bloc Cathaysia où sont situés les deux gisements étudiés dans cette thèse (Maoping et Piaotang). Ce chapitre donne une vue simplifiée des événements géodynamiques successifs observés dans le Bloc Cathaysia jusqu'à l'arrivée des événements Yanshanien (Jurassique-Crétacé) où la majeure partie des granites associés à des gisements à W-Sn sont mis en place. Ce chapitre offre aussi une compilation et interprétation des données géochimiques sur roches totales effectuées sur les formations magmatiques de la période yanshanienne. Ce chapitre tend à

fournir une vision complémentaire à l'ensemble des chapitres suivants qui sont concentrés sur l'étude des veines minéralisées.

La première partie de ce manuscrit est dédiée à des études de pétrographie détaillées et de géochimie des micas lithinifères comme traceurs des processus hydrothermaux à travers l'étude des gisements de Maoping (**Chapter 2**) et Piaotang (**Chapter 3**). L'objectif est d'utiliser les micas lithinifères comme marqueurs des processus hydrothermaux et d'interpréter les différents réservoirs fluides associés à l'évolution de leurs compositions chimiques au cours du temps. Les inclusions fluides peuvent parfois être difficiles à observer et caractériser. Ces deux chapitres ont pour but de fournir une seconde approche d'identification des fluides impliqués dans la formation de ces gisements.

La deuxième partie de ce manuscrit se concentre sur l'étude d'inclusions fluides sur les deux gisements d'intérêt (**Chapter 4**). Ce chapitre présente les cas similaires du gisement de Maoping et Piaotang comme représentatifs de la dynamique des fluides régionaux lors de leur mise en place. Une étude intégrée pétrographique, géochimique et isotopique a permis de caractériser de multiples fluides impliqués dans la formation de ces gisements polyphasés complexes. Les multiples approches de ce chapitre ont été effectuées en collaboration avec le Mineral Resources and Geofluids laboratory (SSTE-UNIGE Geneva, Switzerland), le Institute of Earth Surface Dynamics (IDYST Lausanne, Switzerland) et le Centre de Recherches Pétrographiques et Géochimiques (CRPG Nancy, France).

La troisième partie de ce manuscrit est dédiée au développement de méthodes de datation et leur application. Le premier chapitre de cette partie (**Chapter 5**) se concentre sur l'application de méthodes de datation multiples au cas du gisement de Maoping : ^{40}Ar - ^{39}Ar sur micas, U-Pb sur xénotime et U-Pb sur wolframite, le but étant de documenter l'agencement dans le temps des multiples circulations de fluides identifiées dans les chapitres précédents. Ce chapitre est le fruit de collaborations avec le Department of Geological Sciences of the University of Manitoba (Canada), Géosciences Rennes (France) et le German Research Centre for Geosciences (GFZ Potsdam, Germany). Le second chapitre (**Chapter 6**) présente

le développement de standards de columbo-tantalite pour datation U-Pb *in situ*. Pour cela des collaborations ont été mises en place pour les différents aspects analytiques avec le Centre de Recherches Pétrographiques et Géochimiques (CRPG Nancy, France) et le German Research Centre for Geosciences (GFZ Potsdam, Germany). Les échantillons fournis pour cette étude ont été collectés en collaboration avec le Royal Museum of Central Africa (Tervuren, Belgium), le State Key Laboratory for Mineral Deposits Research (Nanjing, China), le Bureau pour la Recherche Géologique et Minière (BRGM Orléans, France) et le German Research Centre for Geosciences (GFZ Potsdam, Germany).

Ce manuscrit développe ensuite une discussion basée sur les trois grandes questions posées précédemment. Cette discussion reprend les différents modèles de la littérature afin de les discuter à la lumière des nouveaux résultats de cette thèse. Elle se veut indépendante des différents chapitres et tend à raccrocher cette étude à une réflexion plus large qui est celle de la formation des gisements à W-Sn.

Etat de l'art

1. Pourquoi étudier le tungstène ?

Le tungstène et les métaux rares (e.g. Sn, Ta, Nb) sont des ressources d'avenir car couramment utilisés dans les appareils et systèmes de « haute technologie » tels que l'aéronautique, l'armement, les ordinateurs, les téléphones, les appareils photo et bien d'autres. Ces éléments sont principalement extraits de minéraux tels que la wolframite ou la scheelite (W), la cassiterite (Sn) et la columbo-tantalite (Nb-Ta), tous observables dans les gisements hydrothermaux dits de type « veines », associés à des intrusions granitiques. A première vue, ces gisements ont l'air d'être également repartis sur la surface du globe, suggérant une répartition équitable des ressources (Figure 1).

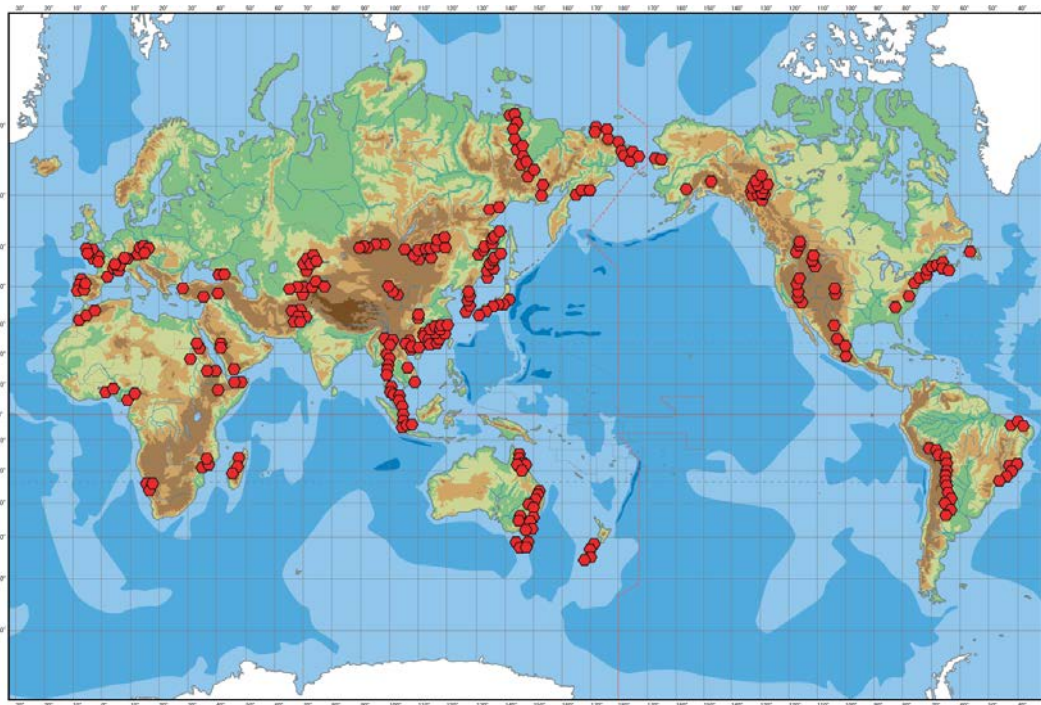


Figure 1. Carte de localisation des gisements à tungstène et/ou étain et pegmatites de type LCT (Li-Cs-Ta), modifié d'après Romer et Kroner (2016).

Néanmoins, le tungstène et les métaux rares (e.g. Sn, Ta, Nb) ont été reconnus « matériaux critiques » par la Commission européenne en 2014. Ces « matériaux critiques » sont définis par leur importance économique (importance dans les secteurs industriels

combiné à leur valeur brute) et leur risque d'approvisionnement (stabilité politique, efficacité du gouvernement, qualité réglementaire, droit...) (Figure 2).

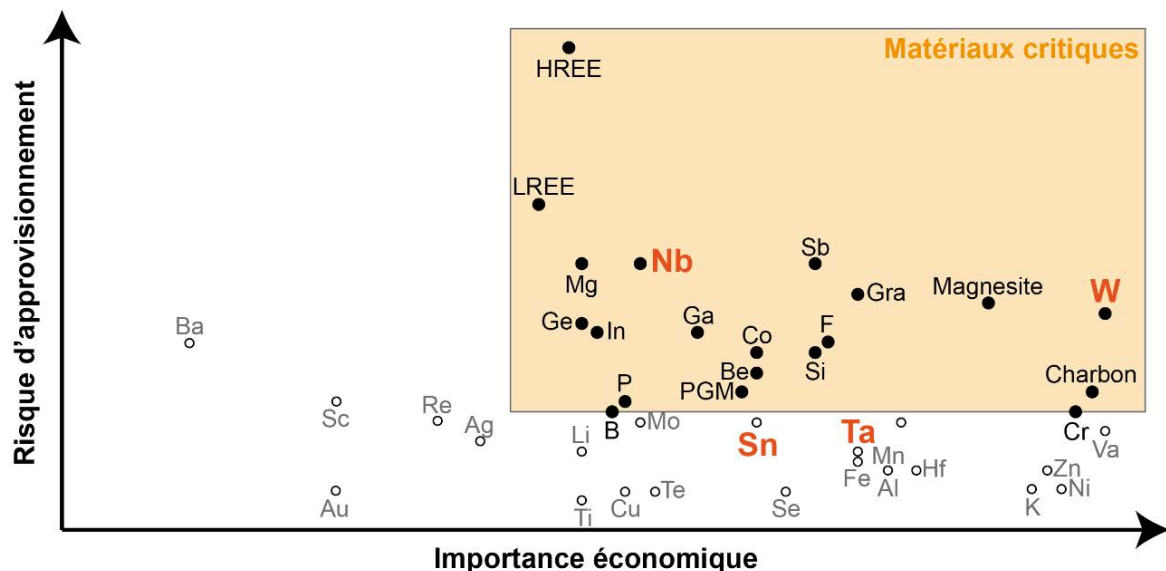


Figure 2. Diagramme binaire indiquant la criticité de différents matériaux en fonction de leur importance économique et leur risque d'approvisionnement (modifié d'après La Commission Européenne 2014).

Pour cause, malgré des réserves de plus de 3 millions de tonnes de tungstène dans le monde, la Chine en possède presque les deux tiers soit 1 million neuf cent mille tonnes (USGS, 2017). De plus, la Chine représente 83% de la production mondiale de tungstène (USGS, 2017). Même si la consommation de ce minerai par la Chine représente tout de même plus de la moitié de la consommation mondiale, il reste impossible pour les pays européens de subvenir à leurs propres besoins (Figure 3).

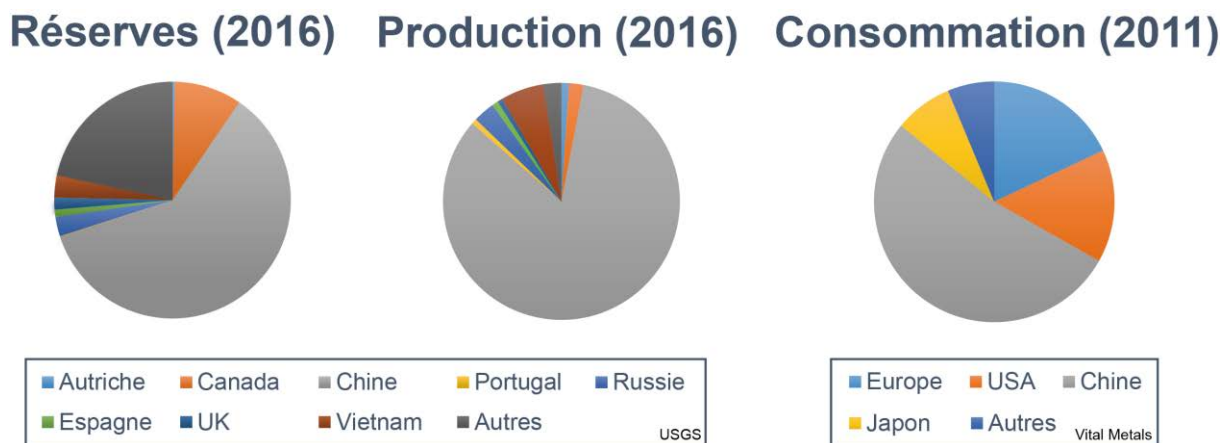


Figure 3. Diagrammes représentant la répartition des réserves (USGS, 2017), des pays producteurs (USGS, 2017) et consommateurs (Vital Metals, 2011) de tungstène.

2. Le tungstène: l'élément

Le tungstène naturel est composé de cinq isotopes stables et sa valence peut varier de 2 à 6. La forme la plus courante est le W^{6+} formant les tungstates.

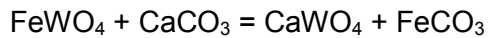
Les concentrations les plus importantes de tungstène sont associées à des processus hydrothermaux. La forme du tungstène dans ce type d'environnements dépend autant des conditions de pressions et températures que de la composition chimique dans laquelle il est mis en solution (dépendant du pH et du Eh). Le minéral le plus courant dans ce type de contexte est la wolframite, qui est un tungstate de fer et manganèse ($(Fe, Mn)WO_4$). Ce minéral est stable entre 250-450°C dans des gammes de hautes pressions (550 à 1700 bars). Deux types de solutions peuvent déposer la wolframite : des solutions hyper salines et des solutions aqueuses diluées d'acides carboniques. Des études thermodynamiques sur les wolframites ont permis de déterminer que les différences de composition de la wolframite se déterminent à température constante par variation de la solubilité des phases. $FeWO_4$ (ferberite) étant de faible solubilité, les wolframites tardives devraient être plus riches en manganèse que les wolframites précoces d'une même solution (Beus, 1986). $MnWO_4$ (hübnerite) précipite dans les solutions neutres à alcalines (Higgins 1985).

Les solutions à tungstène sont généralement accompagnées de sodium, potassium, calcium, fluorine, chlore et dioxyde de carbone. La proportion de tungstène dans ces solutions avoisine généralement les 10^{-5} à 10^{-4} mol/L (Beus, 1986).

Plusieurs substitutions isomorphiques sont possibles dans les minéraux à tungstène : W-Mo (surtout observé dans le cas de la scheelite $CaWO_4$), W-Nb-Ta (dû à la similarité entre les structures de la columbo-tantalite et wolframite) et Fe-Mn-Ca. La substitution entre ferberite ($FeWO_4$) et hübnerite ($MnWO_4$) se fait aux alentours de 400°C (Beus, 1986).

Burt (1981 ; 1978) a démontré que la teneur en F_2O^{-1} et CO_2 contrôle le dépôt de la scheelite et/ou la wolframite. La wolframite se dépose dans des environnements riches en fluor et pauvres en CO_2 . C'est pourquoi les gisements pauvres en fluorite contiennent

préférentiellement de la scheelite. La wolframite, bien que premier tungstate à cristalliser, est rapidement transformée en scheelite lors de l'arrivée de carbonates (Higgins 1985) tel que :



Le coefficient de partage du tungstène entre le magma et la phase fluide n'est pas bien contraint. Néanmoins, quelques expériences ont permis de déterminer un faible coefficient de partage d'environ 1 (Manning et Henderson, 1984 ; Keppler et Wyllie, 1991).

3. Les gisements à W-Sn : cas des gisements périgranitiques de type « veines »

3.1. Morphologie

La plupart des gisements à W-Sn sont régulièrement associés à des intrusions magmatiques de type felsiques qui sont généralement interprétées comme étant la source de ces métaux (Romer and Kroner, 2016). Une grande variété de gisements à W-Sn ont été identifiées par de nombreux auteurs : les gisements disséminés de type greisen, les skarns, les gisements de type veines (qui sont au centre de cette étude) et moins communément les gisements de type porphyre et pegmatitiques (Romer and Kroner, 2016). Ces différents types dépendent de l'évolution du magma granitique associé, de la profondeur de mise en place de celui-ci, de l'encaissant, du contrôle structural régional permettant la circulation de magmas et de la nature et l'évolution de fluides hydrothermaux au sein du système ainsi que leurs possible interactions avec l'encaissant (Romer and Kroner, 2016).

Au niveau des gisements de type « veines », les intrusions sont décrites comme multiphasées et enrichies en éléments incompatibles (dans les plus récentes et plus petites). La morphologie de ces structures dépend des conditions locales de perméabilité et du rapport entre la pression du fluide générée par le magma et la pression lithostatique. Ces paramètres permettent de passer de conditions de tectonique radiaire, au sommet d'une intrusion pour les gîtes précoces, à des contraintes régionales pour les plus tardifs (Jébrak et Marcoux, 2008) (Figure 4).

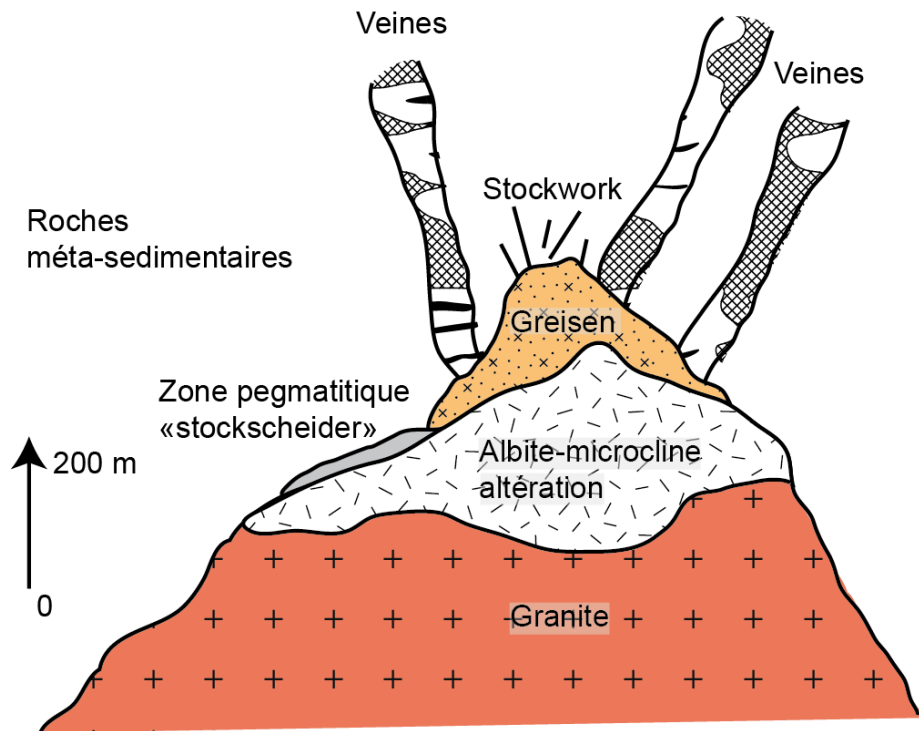


Figure 4. Représentation schématique de la morphologie des gisements à W-Sn de type "veines" (modifié d'après Pirajno, 2009).

Dans la zone supérieure du granite ou à son contact, des stockscheiders pegmatitiques zonés asymétriquement peuvent apparaître. Ceci indique une interruption ou une intrusion de magmas différenciés (Cerny et Ercit, 2005).

3.2. Magmatismes associés

Les intrusions magmatiques associées à ces gisements à W-Sn de type « veines » sont généralement très fractionnées et montrent des signatures en métaux rares remarquables par leur enrichissement en Sn, W, Be, Cs, F, B, Li, Rb, Ta, et U et un appauvrissement en Fe, Ti, Mg, Ca, Sr, Eu, Ba, and Zr (Romer and Kroner 2016).

Les roches magmatiques associées aux gisements à tungstène, et plus particulièrement à étain, sont souvent caractérisées par des valeurs de fugacité de l'oxygène associées à la série de l'ilménite (faible fO_2) (Romer and Kroner 2016).

Dans la chaîne acadien-varisque-appalachien, il y a des signatures en métaux rares remarquables par leur enrichissement en Li, Rb, Cs, Nb, et Ta et un fort appauvrissement en

Ca, Sr, and Ba (Romer and Kroner 2016). Ces signatures peuvent être obtenues par une cristallisation fractionnée extrême. Néanmoins, dans le cas d'une cristallisation fractionnée moins poussée, ce type de signature peut être observée car héritée du protolithe. Par exemple, une altération extrême peut produire des enrichissements résiduels similaires (Romer and Kroner 2016).

Les granites associés aux gisements de type Sn-W-U sont connus pour avoir un taux initial d'H₂O particulièrement élevé (Cuney et Kyser, 2008). Ces granites sont définis comme étant issus de magmas de type S, formés en milieu réduit (Figure 5). Généralement, les magmas associés à la formation de gisements à W-Sn se mettent en place dans la croûte moyenne à profonde par fusion partielle d'une roche mère contenant une proportion importante de matériel (méta)sédimentaire. Ce matériel contient de la matière organique qui va rendre le granite peralumineux et réduit lors de l'équilibration du magma avec les métasédiments (Robb, 2005).

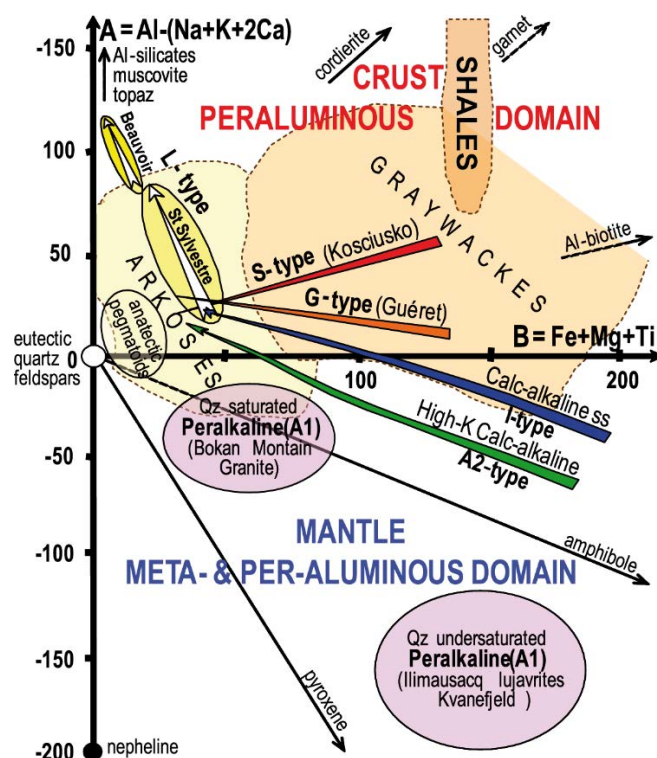


Figure 5. Index de peraluminosité ($A = \text{Al} - [\text{Na} + \text{K} + 2\text{Ca}]$) en fonction de l'index de différenciation ($B = \text{Fe} + \text{Mg} + \text{Ti}$) d'après le diagramme de Debon et Lefort (1988) et discrimination des trends de fractionnement magmatiques parmi les roches ignées peralumineuses, metalumineuses et peralcalines (modifié d'après Cuney, 2014).

Le tungstène se comporte, en milieu réduit (magmas de type S), comme un élément incompatible. Par conséquent, sa proportion augmente dans le magma résiduel lors du processus de cristallisation fractionnée. Le tungstène sera donc associé à des magmas très différenciés (Robb, 2005).

Les altérations sont dans ce type de contexte sont diverses. La greisenification, par exemple, indique une métasomatose à potassium et lithium du sommet du système (Jébrak et Marcoux, 2008).

3.3. Les veines

Généralement, les minéralisations à W-Sn sont associées à un enrichissement en éléments lithophiles de type Ta, Li, B, Be, In, Ge, et Ga (Romer et Kroner 2016).

Les magmas saturés en eau se situent au sommet des intrusions granitiques. Lorsque le magma devient trop saturé, une exsolution de fluides aqueux se produit, formant une nouvelle phase plus différenciée. Le plus souvent, la percolation de ces fluides chauds à travers les fractures entraîne la formation de veines de quartz potentiellement minéralisées (Robb, 2005). Dans le cas d'un fluide riche en chlore, Cu, Mo, Sn et W vont alors préférentiellement dans la phase fluide. Durant cette transition magmatique-hydrothermale, les fluides précoces sont généralement aqueux et circulent dans des conditions allant de 400 à 600°C et de 500 à 600 bars. Les filons sont alors remplis par des pulsations successives de fluides (Jébrak et Marcoux, 2008). Les fluides fluorés ont tendance à plus facilement se mêler aux magmas silicatés qu'aux fluides aqueux et par conséquent, le fluor se présente rarement comme un agent complexant pour les métaux dans la phase aqueuse.

3.4. Modèle général de génèse

Les premiers modèles établis furent en majorité basés sur des contextes de subduction d'une plaque océanique sous une plaque continentale et discutaient de la distribution des intrusions à métaux vis-à-vis de leur distance à la fosse (profondeur de fusion de la croûte subductée et/ou angle de subduction) interprétant que les gisements à W-Sn étaient corrélés

à des subductions « plates » (Romer et Kroner 2016). Le point commun à ces modèles est que la distribution des gisements est strictement dépendante de la zone de subduction, la croûte océanique subductée représentant la source ultime des métaux et le manteau servant de moteur thermique faisant fondre la croûte. Ce type de modèles restent très limités car nombreux sont les gisements à W-Sn formés en zones externes de types bassins arrière-arc, dans les zones de collision ou encore hors limites de plaques lorsque la minéralisation est associée à des granites de type A. D'autre part, les gisements à W-Sn peuvent tout aussi bien être associés à des contextes d'extension crustale ou de rifting. La distribution des minéralisations semble être contrôlée par d'anciennes zones de marges continentales et en particulier, liée à des protolithes granitiques à métaux rares et des volumes très importants de roches sédimentaires intensivement altérées chimiquement (Romer et Kroner, 2016).

Ces gisements se forment en conditions réduites (alors que le tungstène se comporte en élément incompatible) et le tungstène va donc s'enrichir dans le magma résiduel pendant la cristallisation fractionnée. Comme ce type de magma (S, peralumineux) est formé à partir de la fusion partielle de matériel metasédimentaire, le magma sera relativement hydraté et aura tendance à cristalliser assez profondément dans la croûte. Quand la phase fluide s'exsolve, celle-ci rentre alors en interaction avec un magma très différencié et enrichi en tungstène. La phase fluide va alors récupérer une partie de ce tungstène mais le faible coefficient de partage de cet élément induit que la majeure partie de la concentration en tungstène sera atteinte avant la saturation en eau (Robb, 2005).

3.5 Données d'inclusions fluides

3.5.1 Généralités

La grande majorité des inclusions fluides observées dans les gisements à W-Sn sont biphasées, comprenant une phase liquide et une phase gazeuse (Naumov et al., 2011). Il n'est néanmoins pas impossible d'observer certains solides pouvant s'avérer être des sels. Le contenu en gaz est néanmoins minime. La majorité des inclusions sont de type aqueuses ou à faible quantité de CO₂. Il est tout de même possible d'observer des inclusions à CH₄ et/ou

N₂ suivant le rapport moyen suivant : 56.4% CO₂, 30.9 % CH₄ et 13.3 % N₂ (Naumov et al., 2011).

Les diagrammes ci-dessous indiquant des rapports de température, salinité et pression ont été compilés par Naumov et al. (2011) selon des milliers de publications abordant tous types de gisements à W-Sn (veines, skarns, porphyres...) et tous types de minéraux porteurs d'inclusions fluides dont les proportions d'études sont relatées ci-contre. Malgré cette diversité de contextes de formation, la majorité des données restent contraintes dans des gammes très restreintes : 150-400 °C, 0-15 wt.% NaCl equiv. et 0-250 MPa.

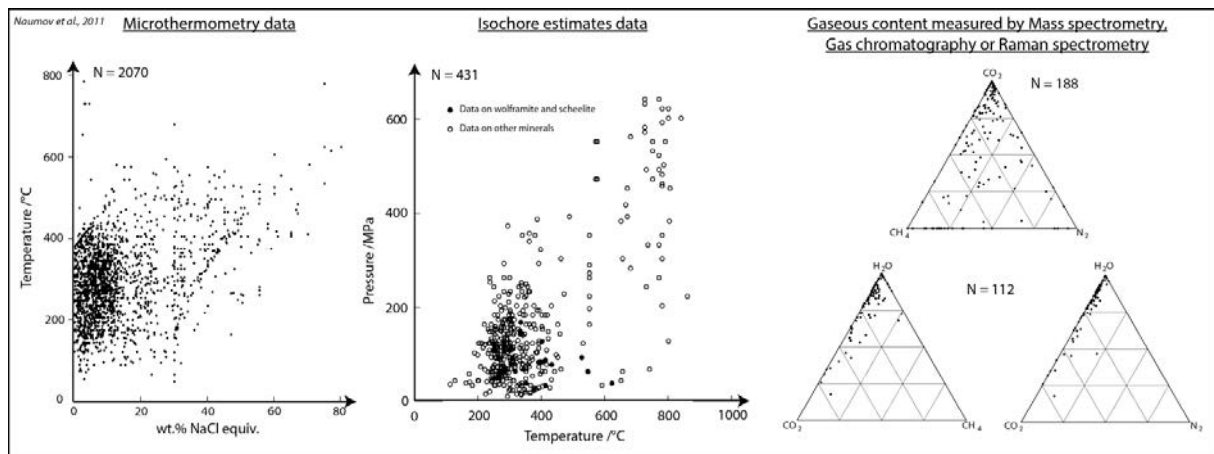


Figure 6. Données microthermométriques, pression-température et compositionnelles compilées par Naumov et al. (2011) concernant tout type de gisement à W-Sn et tout type de minéraux hôtes.

3.5.2 Propriétés des fluides minéralisateurs

Les gisements à W-Sn sont généralement polyphasés, comprenant rarement plusieurs stades minéralisateurs. La compilation des données de la littérature sur les fluides associés au stade minéralisateur (à tungstène ou étain-tungstène) n'est pas très différente de l'ensemble des données compilées sur ce type de gisements. Les conditions connues de cristallisation des minéraux à W-Sn sont donc entre 250 et 450°C, 50 et 150 MPa, avec un contenu en gaz variable voire absent. D'autre part, une faible salinité (entre 0 et 15 wt.% equiv. NaCl) des fluides est souvent attribuée à ce stade minéralisateur.

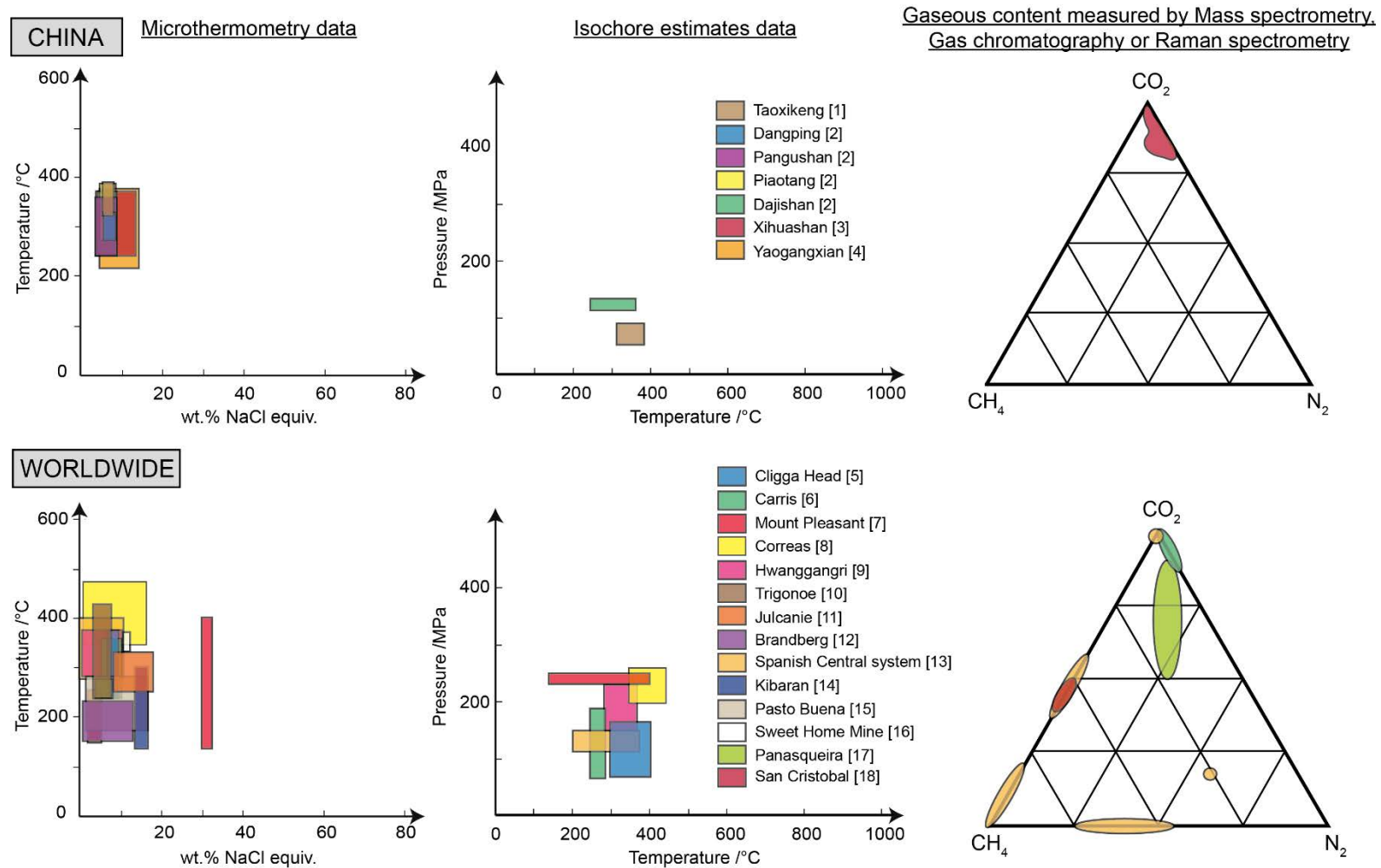


Figure 7. Représentation des données microthermométriques, pression-température et compositionnelles interprétées comme représentatives des fluides minéralisateurs dans des gisements à W-Sn en Chine et dans le Monde, superposé sur la compilation de Naumov et al., 2011. [1] Wang et al., 2012; [2] Ni et al., 2015; [3] Wei et al., 2012; [4] Hu et al., 2012; [5] Smith et al., 1996; [6] Moura et al., 2014; [7] Davis et Williams-Jones, 1985; [8] Bettencourt et al., 2005; [9] So et Yun, 1994; [10] Krylova et al., 2012; [11] Deen et al., 1994; [12] Macey et Harris, 2006; [13] Chicharro et al., 2016, Vindel et al., 1995; [14] Pohl et Gunther, 1991; [15] Landis et Rye, 1974; [16] Lüders et al., 2009; [17] Norohna et al., 1992; [18] Beuchat et al., 2004.

Néanmoins, une grande partie des données de la littérature ont été mesurées sur des inclusions fluides contenues dans des quartz, minéral sensible à la déformation (Figure 8). Il est donc possible que les informations retranscrites relatent des évènements postérieurs.

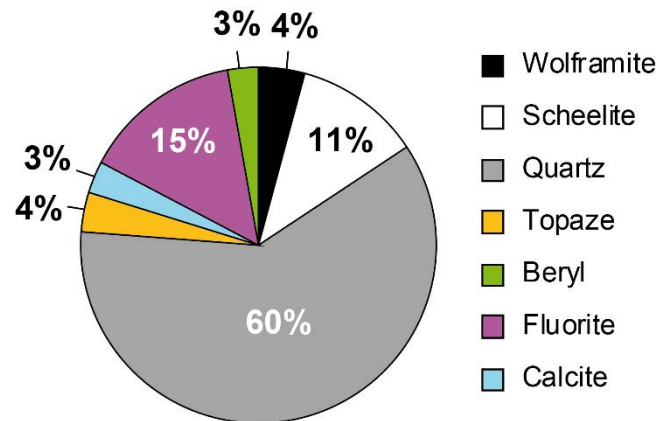


Figure 8. Répartition des types de minéraux aillant fait l'objet d'études d'inclusions fluides dans les gisements à W-Sn, d'après les données de Naumov et al. (2011).

D'autre part, il a récemment été montré que les fluides identifiés dans la gangue, interprétés comme contemporains de la minéralisation, ne sont pas toujours équivalents aux fluides observés et interprétés comme primaires dans les métaux (Wei et al., 2012 ; Ni et al., 2015).

3.5.3 Source des fluides minéralisateurs

Ces gisements sont systématiquement associés à des environnements perigranitiques où des processus magmatiques-hydrothermaux sont invoqués. Néanmoins, trois grandes catégories de fluides ont pu être distinguées par Marignac et Cathelineau (2009) parmi l'ensemble des données de la littérature (Figure 8).

- Les systèmes magmatiques-hydrothermaux

L'évolution de ces fluides et leur compositions ont pu être contrainst par de nombreuses études (Audétat et Pettke 2003; Kamenetsky et al. 2004; Thomas et al. 2005). Les fluides hydrothermaux sont de haute température (<600°C), à forte salinité (50 % pds. equiv. NaCl), les ratios K/Na sont proches de 1, le rapport Fe/Ca est élevé et les teneurs des fluides en métaux rares sont élevées.

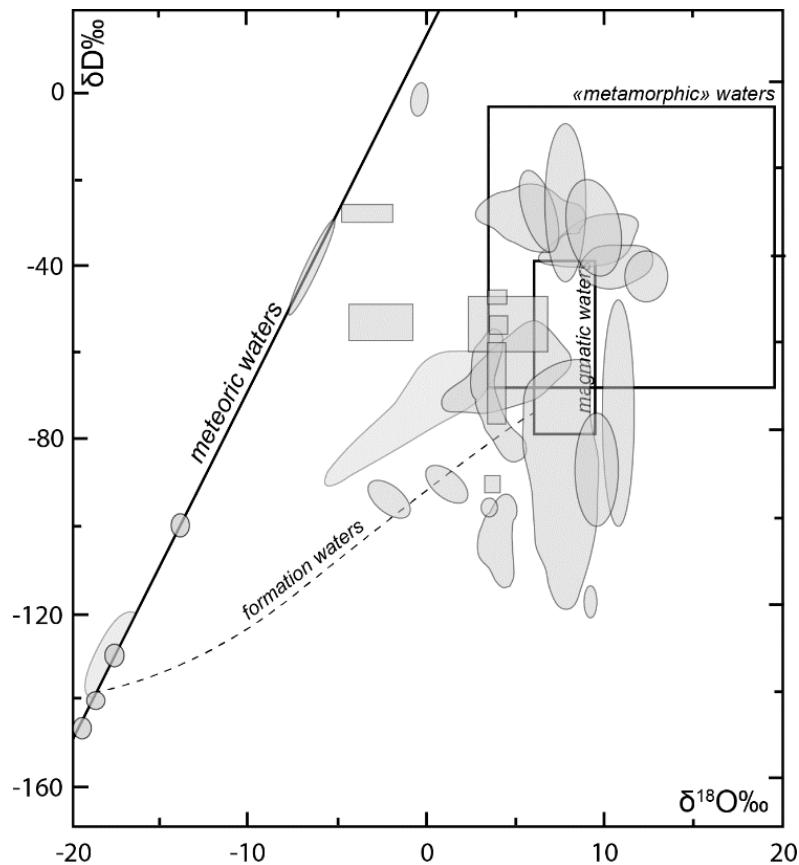


Figure 9. Diagramme δD - $\delta^{18}O$ simplifié des fluides associés aux gisements à W-Sn dans le monde (modifié d'après Marignac et Cathelineau, 2009, voir détails dans publication citée). Les zones grisées indiquent des champs de compositions isotopiques de fluides analysées dans des inclusions fluides de minéraux de gangue cristallisés dans des gisements à W-Sn.

- Les systèmes à dominante métamorphique

Ces fluides sont caractérisés par la présence de volatiles dans les fluides minéralisateurs (e.g. Weisbrod, 1988 ; Norohna et al., 1992 ; Vindel et al., 1995). Leurs caractéristiques sont les suivantes : pas de fluides hypersalés, les fluides précoces sont de haute température (<500°C) avec une forte abondance d'éléments volatiles (principalement du CO₂), la salinité est modérée (<15 % pds equiv. NaCl) et la précipitation des minéraux à W-Sn se fait à plus de 300°C par mélange avec des fluides plus froids et très peu salés.

- Les systèmes à dominante granitique

Ces fluides sont caractérisés par une absence d'éléments volatiles et des salinités variables (5-15 % pds equiv. NaCl). Les fluides les plus salés sont associés à la précipitation des minéraux à W-Sn, à plus haute température (400°C) et avec des enrichissements en Br.

4. Exemple de gisement fini-varisque : le Massif Central français

Les gisements de tungstène associés à des systèmes de veines à quartz-wolframite-cassiterite sont à ce jour bien connus dans la Chaîne varisque. La Chaîne varisque européenne est de loin la plus importante en occurrences de métaux rares (Nb, Ta, Li, Be) avec des districts à métaux rares et W-Sn de classe mondiale tels qu'en Cornouailles, dans le Massif ibérique, le Massif de la Bohême, le Massif armoricain et le Massif Central Français (FMC). Ce dernier abrite de nombreux gisements à tungstène avec un potentiel économique de plus de 45 kt WO_3 , principalement sous forme de minéralisations disséminées dans les granites ou sous forme de veines hydrothermales peri-granitiques (Marignac et Cuney, 1999). Le principal événement à tungstène eut lieu à la fin de la collision continentale entre les blocs Gondwana et Laurussia, au Paléozoïque supérieur. Les conditions géodynamiques de mise en place sont complexes et caractérisées par des contraintes simultanées de compression et extension, typiques de processus de délamination de la lithosphère. A la fin du carbonifère, des pulsations magmatiques successives ont permis la mise en place de granites à profondeurs variables et sous divers régimes tectoniques. Des granites d'origine crustale profonde et moyenne ont été produits. Le premier est caractérisé par de fortes interactions avec des magmas mafiques issus de la fusion partielle du manteau lithosphérique subcontinental métasomatisé. L'autre est typiquement peralumineux et spatialement associé à des minéralisations à W-Sn-Nb-Ta.

Des recherches réalisées sur le Massif Central Français et la province ibérique ont montré que les fluides dominants associés aux veines minéralisées font partis d'un système fluide de type C-O-H-N à signatures isotopiques caractéristiques de fluides externes tels que météoriques et/ou métamorphiques (Polya et al., 2000, Harlaux 2016). La majorité des travaux d'inclusions fluides sur les gisements à W-Sn du Massif Central Français a été menée sur le gisement d'Echassières (Aïssa et al., 1987b; Cuney et al., 1992). Quatre types d'inclusions fluides ont pu y être décrits : (i) des inclusions fluides magmatiques L1 précoces de haute température (490-590°C), fortes salinités (25-30 pds% NaCl equiv.) et enrichies en lithium, (ii)

des inclusions vapeur V2 et liquides L2 issus de l'ébullition de ces premières inclusions fluides à des températures comprises entre 400 et 520°C et des salinités variant de 3 à 12 pds% NaCl equiv. pour les inclusions vapeurs et de 30 à 50 pds% NaCl equiv. pour les inclusions liquides, (iii) des inclusions fluides aqueuses L3 à faibles salinités (2-6 pds% NaCl equiv.) pour des températures entre 330 et 420°C et (iv) des inclusions fluides aqueuses tardives de températures n'excédant pas 350 °C mais de salinités variables (0 à 60 pds% NaCl equiv.).

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Chapter 1: Assessment of the geodynamical and magmatic settings of the Jurassic-Cretaceous period in the Cathaysia Block (SE China): a preliminary review

Article in preparation for Earth-Science reviews

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1. Introduction

The Mesozoic activity in South China is responsible for the major sources of W, Sn, Sb, Ta, Cu, Hg, As, Tl, Pb, Zn, Au and Ag resources of China and worldwide. Most of these deposits are associated to hydrothermal processes related to felsic intrusions as vein-type, porphyries, skarns, MVT and epithermal deposits (e.g. Hu R.z et al., 2017). Recent studies consider the Trias-Cretaceous period (Indosinian-Yanshanian) is mostly responsible for the formation of these deposits (e.g. Hu R.z et al., 2017). With 1.7 Mt tungsten, the Jurassic-Cretaceous (Yanshanian) “E-W” trending granite belts in the Nanling Ranges are the repository for the largest tungsten province worldwide and this magmatism is also responsible for the largest antimony province and with 1.2 Mt Sn for one of the main tin provinces worldwide (e.g., Sun W.d et al., 2012; Hu and Zhou, 2012; Li Z. et al., 2014).

The South China Craton (SCC) resulted from the (late) Grenvillian suturing between two terranes, the Yangtze craton, to the north, and the Cathaysia Block, to the south, through the

Jiangnan (or Sibao) orogen, which is considered to be part of the present-day Yangtze Block, the Jiangshan-Shaoxi-Chenzhou-Yulin fault zone (JSCYFZ) being often thought to represent the boundary between the two blocks. In the SCC, the Cathaysia Block suffered the most pervasive magmatic activity in the Yanshanian times and therefore is the richest part in term of economic resources (Figure 10).

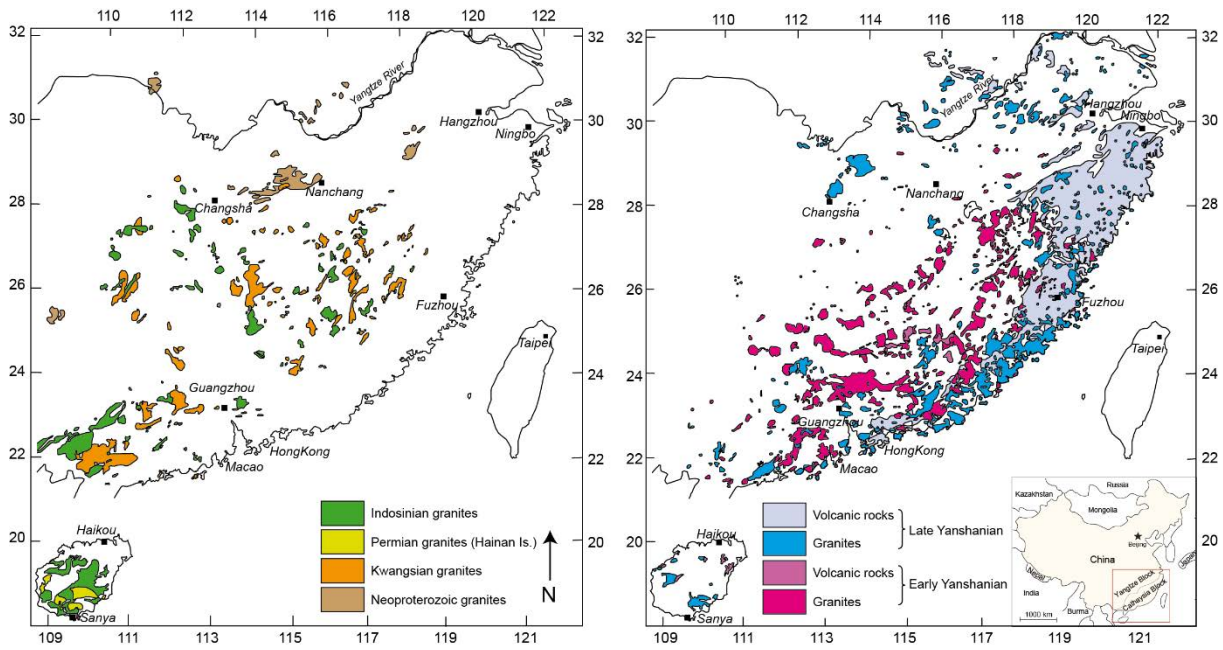


Figure 10. Maps of the granitic rocks emplaced in the South China Craton between the Neoproterozoic to the Cretaceous period (modified from Wang Y. et al., 2013).

Many petrological, geochronological, geochemical and structural studies have provided a large amount of data improving the understanding of the SCC evolution. It is recognized that from the Neoproterozoic continental-scale rifting, to the formation of the Nanhua rift and the oblique Jiangnan suture, many inherited structure played a major role to control the Palaeozoic and Mesozoic events in the SCC.

This study aims to propose a preliminary review of the geodynamical and magmatic settings of the Jurassic-Cretaceous period when the large Nanling metallogenic belt was emplaced. For this purpose, we tried to enlight the major geodynamical settings endured in the Cathaysia Block from the Paleoproterozoic to the end of the Cretaceous period in order to attest the global structural settings emplaced prior to the Jurassic-Cretaceous magmatism.

This magmatism is then studied in detail through the compilation of multiple bulk-rock analysis on mafic and felsic rocks, plotted in interpretative binary and ternary diagrams in order to deconvolute the main magmatic mechanisms involved in the Cathaysia Block in the Jurassic-Cretaceous period where world-class rare metal deposits were emplaced.

2. The Cathaysia basement

Precambrian outcrops are scarce in the Cathaysia Block, being restricted to the west and to the related Hainan Island. Their identification is somewhat complicated due to a strong overprinting by younger tectono-metamorphic and magmatic events. They consist in (meta)granites, metasediments and orthoamphibolites, found in several domains, from N to S: Chencai, Wuyishan (N: Badu Complex, and S: Mayuan Complex), Nanling, Yunkai and Hainan Is (Figure 11). Until the 2000's, they were considered to be mainly Paleoproterozoic rocks with a few Neoproterozoic remnants, but recent investigations lead to the inverse conclusion, that up to 90% of the exposed rocks are indeed Neoproterozoic and part of the (metamorphosed) cover of the Cathaysia, whereas older rocks, although present, are indeed very rare (Zhao and Cawood 2012, and references therein).

2.1. Paleoproterozoic remnants in the Chencai and Wuyishan domains

The Chencai Complex is known to be derived from Meso-Neoproterozoic tholeiite, gabbro, marble and turbiditic mudstone and sandstone protoliths. Li Z.x et al., (2010) yielded a zircon U–Pb age of 1781 ± 21 Ma for amphibolites from the Chencai complex in central Zhejiang. The protolith of these amphibolites is a gabbroic sill intruding the Chencai complex. Therefore, the deposition age for the protolith of the Chencai complex is older than 1780 Ma.

In the northern Wuyishan domain, evidence for a ca. 1.9 Ga metamorphic event was found in the Badu Complex. Yu J.h et al., (2012) document a metamorphic event at 1884 ± 2 Ma on leucosomes of a migmatitic gneiss, and (from inherited zircons) consider that the sedimentary protolith was deposited not long after ca. 2.5 Ga. Two peraluminous gneissic

metagranites that were intrusive in the metamorphic rocks and of which compositions are close to the migmatite leucosomes, yield slightly younger U-Pb (Zrn) ages of 1888 ± 7 Ma (Xiaji granite) and 1875 ± 9 Ma (Lizhuang granite) (Yu J.h et al., 2009), i.e., a ca. 1883 Ma age. Two A2-type monzogranites yield still younger ages of 1856 ± 10 Ma (Tianhou granite) and 1855 ± 9 to 1869 ± 10 Ma (Danzhou massif) (Yu J.h et al., 2009), i.e., a ca. 1862 Ma age., with inherited cores in the 2.1 to 2.9 Ga range, while metamorphism at ca. 1.9 Ga affected protoliths formed in the 2.5 to 1.9 Ga range (Xia Y. et al., 2012).

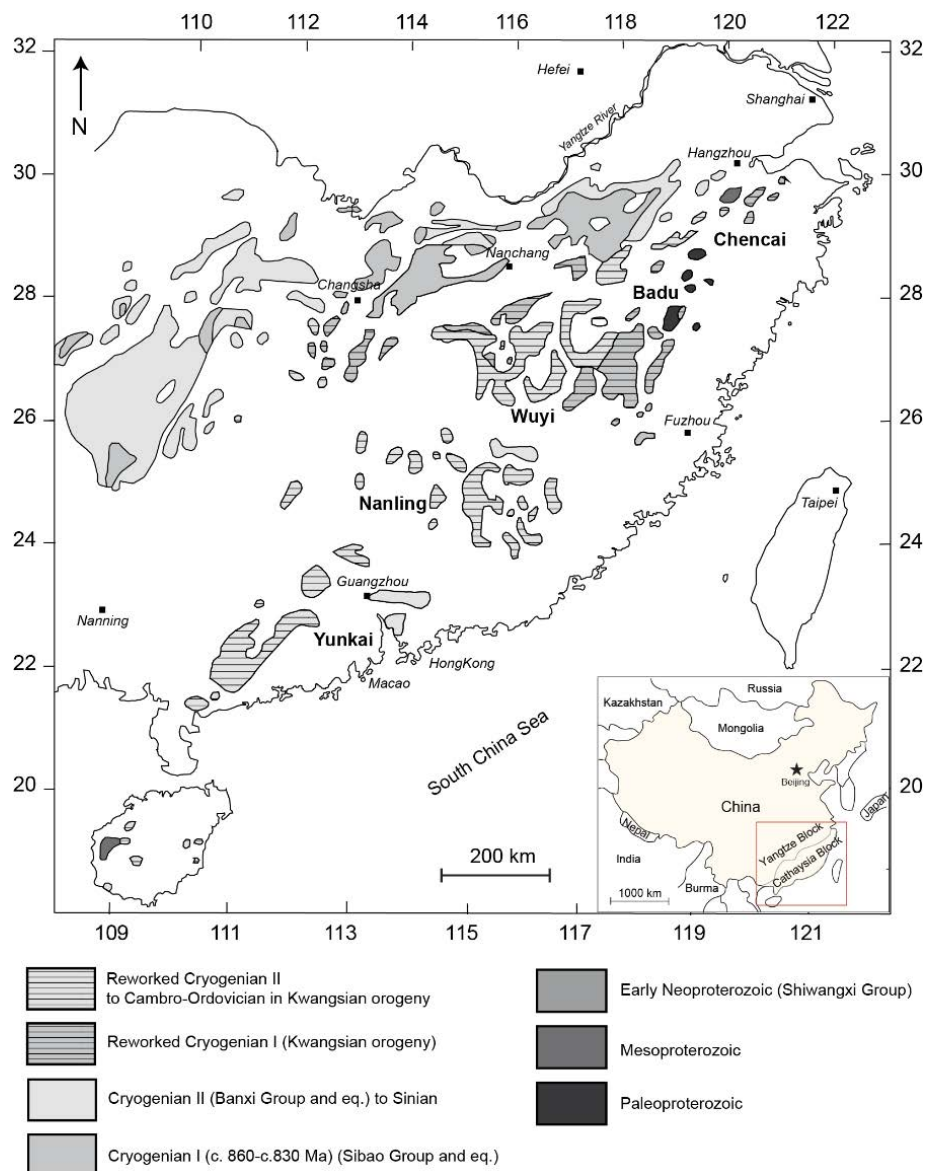


Figure 11. Map of South China showing the remaining evidences of formations dated from the Paleoproterozoic to the Neoproterozoic (modified after Zhao J.h and Asimow P.d, 2014)

In the southern Wuyishan domain (Mayuan Complex), the so-called Tianjinping Formation contains amphibolites with protoliths dated at ca. 1.9 Ga, associated with paragneiss, the youngest detrital zircon in which is dated at 1.76 Ga, although most of the “Formation” is indeed by far younger (Wan Y.s et al., 2007). These data are taken as evidence for the existence in the W Cathaysia of a complete collisional cycle of Paleoproterozoic age, with the 1.77 to 1.78 Ga amphibolites and “A-type” granites representing a post-orogenic intracontinental rifting stage (e.g., Li X.h et al., 2014). In addition, within the so-called “Mayuan Group”, some rocks with ca. 2.1 to 1.8 Ga “protolith” ages, display a ca. 1.4 Ga metamorphic age (Xu X.s et al., 2005). Metakeratophyres of this group were dated, using conventional zircon U-Pb methods, at 1438 ± 41 Ma (Gan et al., 1993) and amphibolites were dated by Sm-Nd whole rock isochron methods at 1376 ± 82 Ma and 1347 ± 39 Ma (Zhou H.w et al., 1997). Li X.h et al., (1998) obtained a SHRIMP U-Pb zircon age of 1766 ± 19 Ma for amphibolites of Mayuan group, which was interpreted as the age of their protolith.

2.2 Mesoproterozoic rocks in the Hainan Island and in the Nanling domain

In the Hainan Island, the high-grade Baoban Complex is made of a gneissic granite dated at ca. 1430 Ma and metamorphosed in the amphibolite facies at ca. 1.3-1.1 Ga, while the low-grade platformal metasediments of the Shilu Group were deposited at 1440-1430 Ma, and they are both unconformably covered by the ≤ 1.2 Ga sediments of the foreland Sihuiding Formation (Li Z.x et al., 2008). An older basement is also probably underlying the Island, inferred from gneissic charnockite-amphibolite assemblages in Qiongzhou and have a zircon U-Pb age of ca. 2560 Ma (Zhang K.j et al., 1997).

In the Nanling domain, very scarce observations were made. Although Nd model ages of granitoid rocks implied that Mesoproterozoic basement was present beneath parts of the Caledonian belt (Chen and Jahn, 1998; Wang and Shen, 2003), the only evidence for Mesoproterozoic basement in the Cathaysia was made with the Lanhe orthogneiss that display a 1.5 to 1.3 Ga magmatic age and a Neoarchean source, would have suffered metamorphism at ca. 1.0 Ga (Xu X.s et al., 2005).

2.3 Evidences from inherited zircons

Other evidences for an old basement include dating of inherited zircons, either xenocrysts or cores of magmatic zircons in Phanerozoic granites and mafic rocks, and geochemical modelling (bulk rock NdTDM or zircon HfTDM). Most of these data point to Paleoproterozoic and Archean basement, but evidence for a Mesoproterozoic event is also found.

The old ages obtained on some zircons may be *a priori* interpreted either as recycling through sedimentary cannibalization or as representative of old magmatic and/or metamorphic rocks presently concealed in the basement.

Compiling the ages obtained for detrital zircons in (meta)sedimentary rocks, Li X.h et al., (2014) note that most Neoarchean zircons (peaking age at ca. 2.5 Ga) are smoothed and must therefore be interpreted as sourced in “exotic” Archean continents, not as records of presently unexposed Cathaysia basement, whereas to the contrary the zircons with ages comprised between 1.9 and 0.8 Ga display euhedral to subhedral morphologies and are clearly of magmatic origin, testifying for proximal sources. As cogently argued by Wan et al., (2011), from the Neoproterozoic onwards, the Cathaysia (meta)sediments could not be fed by the Yangtze craton, and consequently the reworked zircons testify for a Cathaysian provenance. Beside the Neoarchean peak, there are two other peaks, one at ca. 1.85 Ga, confirming the importance of the Paleoproterozoic event, the other at ca. 970 Ma, clearly recording the Jiangnan orogeny, with minor peaks at ca. 1.43 Ga, 1.07 Ga, 780 Ma and 590 Ma. Regarding the 1.43 Ga Mesoproterozoic age, which is similar to the dates obtained in the Nanling and Hainan domains, it may be recalled that in the Neoproterozoic sediments of the Kangdian Rift (Yangtze craton), detrital zircons with a ca. 1.4 Ga age are considered to be sourced in Cathaysia (Li Z.x et al., 2002). From these data, Li X.h et al., (2014) conclude that “(...) the oldest crustal basement rocks in Cathaysia were most likely formed by generation of juvenile crust and reworking of recycled Archean components in Late Paleoproterozoic at ~ 1.9-1.8 Ga

(...)”, although, strictly speaking, they only testify that since the Paleoproterozoic no Archean rocks never outcropped in the Cathaysia block.

On the other hand, xenocrysts or inherited zircon cores in Jurassic and Cenozoic basaltic dykes and diatremes (E Guianxi: Pingnan, Pingle, i.e. SW of the W Cathaysia block), which provide a direct insight on the deeper crust (Zheng J.p et al. 2011), mainly yield Archean ages with four age populations: 3.85 Ga, 3.55 Ga, 3.3 to 3.2 Ga and the main 2.9 to 2.5 Ga group. These Archean zircons display euhedral to subhedral morphologies and, being clearly of magmatic origin, indubitably testify for a magmatic source (or a source in proximal sediments). The inescapable conclusion is that “(...) the western Cathaysia block is an old continent and has a lower crust that is significantly older than the exposed upper crust” (Zheng J.p et al., 2011, p. 254). From the oldest zircon data, it could be deduced that Eo- to Paleoproterozoic nuclei included in Mesoproterozoic rocks would constitute a large part of the Cathaysia lower crust (Zheng J.p et al. 2011). These conclusions are reinforced by Nd (whole rock) and Hf (zircon) isotope geochemistry and age modelling.

In conclusion, the origin of the W Cathaysia terrane was likely the amalgamation and reworking of Neoproterozoic nuclei in the Paleoproterozoic, as part of the formation of the Nuna supercontinent. It may be proposed that W Cathaysia was part of a large Paleoproterozoic belt connecting E Antarctica and Laurentia (Li X.h et al., 2014), which would be consistent with a proposal by Li X.h et al., (2014) that the Mesoproterozoic (ca. 1.4-1.5) granites of Hainan and the Nanling Ranges were in the prolongation of western Laurentia.

2.4 The Yunkai domain

In the Yunkai domain, Paleoproterozoic (“Guangzhou Group”) and Neoproterozoic (“Yunkai Group”) metasediments coexist. The inventory of detrital zircons show that in both “groups”, the youngest zircons are not older than 0.8 or even 0.6 Ga (Wan Y.s et al., 2010; Yu J.h et al., 2010), thus indicating the predominance of Nanhua rift-related sedimentation in the area. These findings are corroborated by geochemistry, with in addition strong indication of the presence of some Neoproterozoic rocks at depth. Evidence comes from the study of both

Phanerozoic granites (whole rock Sm-Nd, U-Pb dating of zircon xenocrysts and/or inherited cores, zircon Lu/Hf) and the detrital zircon populations in metasediments of all age.

Pioneering work by Chen and Jahn (1998) demonstrated that most Phanerozoic granites in Cathaysia were sourced in a Paleoproterozoic crust, with a small component of recycled Neoarchean material, to the exception of a NE-trending central belt with Mesoproterozoic Nd model ages (1.6 to 1.4 Ga). Further work fully validated these views. Nd and Hf model ages for a large number of granites and metasediments of all ages are in line with a Paleoproterozoic-Neoarchean basement (e.g., Wan Y.s et al., 2007, 2010; Zhang F.f et al., 2012). Moreover, Hf model ages confirm the existence of a belt with a dominantly Mesoproterozoic signature.

U-Pb dating of either inherited cores in magmatic zircons or detrital zircons yield the same results. For instance, in the Mayuan Complex, the Neoproterozoic volcano-sedimentary metasediments of the Nanshan Formation contains detrital zircons ranging from 2.7 to 1.75 Ma (Wan Y.s et al., 2007) as do the “Caledonian” migmatites (Liu R et al., 2010). Similar results (2.77 to 1.72 Ga) were obtained in the late Neoproterozoic Yunkai metasediments (Wan et al., 2011).

2.5 Cathaysia subcontinental lithospheric mantle

Insights on the Phanerozoic Cathaysia SCLM are provided by mantle xenoliths found in mafic volcanic and subvolcanic rocks of Mesozoic or Cenozoic ages. Results point to the presence of a newly accreted SCLM in the Phanerozoic times, mostly beneath the major translithospheric faults, remnants of the Proterozoic SCLM being however recognized outside these faults (Zheng J et al., 2004; Li X.y et al., 2014, and references therein). Removal of the Proterozoic SCLM and replacement by a thin lithospheric mantle (≤ 80 km) was already achieved in the Jurassic according to Liu C.z et al., (2012).

Olivine compositions indicate that “most of the peridotites of the W Cathaysia block plot in the Tecton field or overlap the field between Tecton and Proton”, some rare xenoliths contained in

far-fault intrusions being close to the Archon field (Zheng J et al., 2004). According however to a geochemical study of the Paleoproterozoic amphibolites in the Wuyishan domain (interpreted as representative of post-collisional rifting) the SCLM at that time was strongly depleted (Li X.h et al., 2000) and likely still of the Archon type. It is therefore inferred that the residual Proterozoic SCLM in the Phanerozoic was derived from metasomatic transformation of an Archean depleted mantle (Zheng J et al., 2004; Li X.y et al. 2014), when the major part of the pristine SCLM was delaminated and replaced by new lithospheric material directly evolved from the asthenospheric mantle. Re/Os studies of sulphides in xenoliths from Cenozoic basalts allowed Liu C.z et al., (2012) to propose a time scale of the primitive Cathaysia SCLM. There were numerous infiltration events that reset the Re/Os ages of the mantle sulphides. The older ages are Neoarchean (2.7 to 2.5 Ga), likely providing evidence for the existence of remnants of the Neoarchean mantle and overlying crustal rocks in the E Cathaysia basement (Liu C.z et al., 2012). Paleoproterozoic (c. 1.8 Ga), Mesoproterozoic (1.5 to 1.3 Ga), as well as Neoproterozoic (Jiangnan orogeny and Nanhua rifting), and earliest Yanshanian (c. 180 Ma) events are equally documented (Liu C.z et al., 2012).

Two conclusions may be drawn: (i) For the main part, the SCCFB basement consists in early Paleoproterozoic rocks that were involved in a strong tectonothermal event at c. 1.8 Ga, i.e., the formation of the Nuna supercontinent with the likely presence of Neoarchean reworked microcontinental blocks. In this respect, Cathaysia was likely very similar to the Yangtze craton (Zheng Y.f et al., 2013 and references therein). (ii) A central belt of younger rocks was formed during the Mesoproterozoic (ca. 1.6 to ca. 1.4 Ga). The outcropping Lanhe gneisses and granodiorite in Hainan Island are likely part of this belt. Based on the Hainan evidence, it has been proposed that prior to the worldwide Grenvillian events, Cathaysia was located to the west of the Laurentia craton and the Hainan Bayoban granite was in the continuity of the “Transcontinental granite-rhyolite province” or “Mojave province” (1500-1350 Ma) in Laurentia (“Missing Link” solution of the Rodinia supercontinent reconstruction) (Li Z.x

et al., 2002, 2007). This conclusion may be tentatively expanded to the whole of the “LTB” in Cathaysia.

3. Architecture of the Cathaysia (meta)cratonic area

At first glance, the Cathaysia Block appears to be composite, formed from three segments, separated by two major fault zones, the Zenghe-Dapu fault zone (ZDFZ) and the Changle-Nan’ao fault zone (CNFZ): the South China Caledonian fold belt (SCCFB) or E Cathaysia (Xu X.s et al., 2007), to the west of the ZDFZ, the Southeast China Coastal Volcanic Belt (SCVB) between the ZDFZ and the CNFZ, and the Metamorphic Coastal Belt (MCB) to the east of the CNFZ, both constituting the E Cathaysia block (Xu X.s et al., 2007; Figure 12). The Hainan Island is generally thought to be part of the W Cathaysia; the island is however divided into two parts by the NE-trending Baisha fault zone (Xu Y.h et al., 2014), a possible SW-wards prolongation of the ZDFZ, and thus, only the NW portion of the island (with the Mesoproterozoic Baoban and Sihuilding groups) would be part of the W Cathaysia.

3.1 The Jiangnan (Sibao) orogen and the Yangtze-Cathaysia boundary

The Jiangnan orogenic belt consists in arc (e.g., Shuangxiwu arc) and back-arc (e.g., N Jiangxi ophiolite) terranes thought to have been overthrust onto the Yangtze craton, thus welding the Yangtze with the Cathaysia, which acted as the passive margin (Charvet 2013 and references therein).

The Jiangshan-Shaoxi fault zone bordering the belt to the SE is generally considered to mark the boundary between the two terranes, in accordance with the seismological evidence for a several km jump of the Moho depth at the crossing of the fault zone (from 30-32 km to the east to 34-36 km to the west) (Wang Y.j et al., 2013 and references therein). The SW prolongation of the Jiangshan-Shaoxi fault is more problematic. Many workers point to an apparent continuity with the Chenzhou-Linwu fault, which displays the same seismological signature

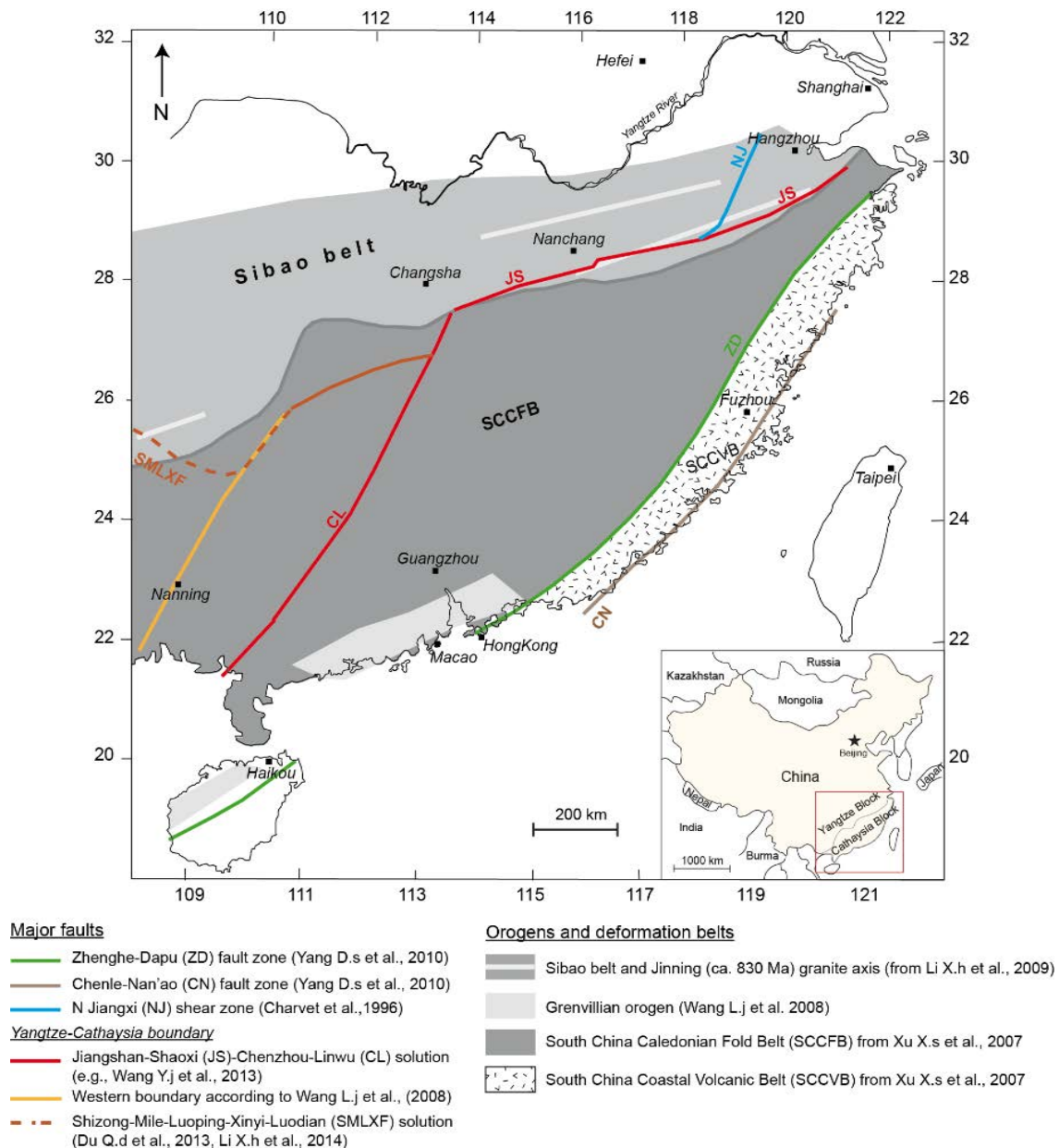


Figure 12. Architecture and major faults in the South China Craton (compiled from Yang D.s et al., 2010; Charvet et al., 1996; Wang Y.j et al., 2013; Wang L.j et al., 2008; Du Q.d et al., 2013; Li H.h et al., 2014; Li X.h et al., 2009; Xu X.s et al., 2007).

(Zhang Z.j et al., 2013), and is generally thought to represent the continuation of the Yangtze-Cathaysia boundary. Yet, the question remains open (e.g., Xia Y et al., 2014), since in the SW, the “geological” boundary, as represented by the southern border of the Jiangnan orogen, is located several 100 km to the north, raising the unsolved question of the significance of the Chenzhou-Linwu fault zone. Moreover, according to Zheng Y.f et al., (2013) (and references therein), there is a strong contrast between the Yangtze craton crust (c. 40 km) and SCLM ($\geq$

150 km) and the “Jiangnan orogeny-W Cathaysia” crust (c. 30 km) and SCLM (c. 70-80 km), lending some support to the idea that the SW prolongation of the terrane boundary is, to the north, the Anhua-Luocheng fault zone (Yang D.s et al., 2010). For some workers (Shu X.j et al., 2013 and references therein) the Chenzhou-Linwu fault would rather represent a Mesozoic lithospheric boundary (see below: Yanshanian). In support of this hypothesis, it must be noted (i) that the extent of the Kwangsi deformations is bounded to the west by the Anhua-Luocheng fault (Lin et al., 2008), as (ii) is also bounded the Phanerozoic development of granite magmatism (Wang Y.j et al., 2013). It must be added that both the Jiangshan-Shaoxi and Anhua-Luocheng faults play a major role in the Kwangsi and Indo-Sinian orogenies, being apparently reactivated, and even be envisaged that in relation with its “Caledonian” reactivation, the Jiangshan-Shaoxi fault in reality truncates the Jiangnan belt (Li X.h et al., 2009).

3.2 An E-W boundary between N and S parts of W Cathaysia?

According to Yu J.h et al., (2010) there are significant differences in the zircon age spectra from metasediments according to their location in either the Wuyishan domain or both the Nanling and Yunkai domains, which appears similar from this point of view. In all domains, there are Neoarchean (ca. 2.6-2.5 Ga) and Neoproterozoic (ca. 0.8-0.7 Ga) zircons, whereas the northern domain is characterized by Paleoproterozoic zircons (ca. 2.1-2.0 and 1.86 Ga), which are lacking in the southern domains, where to the contrary, beside Mesoarchean (ca. 3.3 Ga) zircons, there is a strong imprint of Mesoproterozoic (ca. 1.7 to 1.5 Ga) and Grenvillian (ca. 1.1 to 0.9 Ga) zircons. This is interpreted by Yu J.h et al., (2010) as recording the presence of an E-W boundary, the location of which would be marked by the E-W clustering of the earliest Yanshanian bi-modal volcanic rocks (Liu C.z et al., 2012b), gabbro-syenite complexes and A-type granites. Yu J.h et al., (2010 and references therein) further observe that inherited zircons in intrusions from both sides of the hypothetical boundary may testify for a difference in the respective basements: Mesoarchean and ca. 1.4 Ga zircons to the S (Kwangsi Tanghu granite and Guzhai granodiorite in the Nanling Ranges), and 1.86 Ga zircons in the

Mesozoic Doushui lamprophyre in the Wuyishan. It may be added that very limited evidence for the presence of E-W shear zones in the basement is preserved in the Mesoproterozoic Lanhe gneiss (Xu X.s et al., 2005). According to Yu J.h et al., (2010) the two Wuyishan and Nanling-Yunkai domains were distinct terranes, which would have been docked during the Grenvillian events, at ca. 1.1-1.0 Ga, i.e., close to the docking of Yangtze and Cathaysia. If it is so, it must be concluded that the cathaysian prolongation of the Mesoproterozoic Laurentian transcontinental granite-rhyolite province would have indeed be restricted to the Nanling-Yunkai terrane.

3.3 The significance of the Zenghe-Dapu fault zone (ZDFZ)

Owing to the fact that, from zircon data, the E Cathaysia basement apparently shares the same history as the Wuyishan domain, which is different from the Nanling-Yunkai domain, it results that the ZDFZ would not be a major terrane boundary within the Cathaysia (meta)craton (Yu J.h et al., 2010).

On the other hand, the abundance of detrital zircons with 1.0 to 0.9 Ga in the S Cathaysia metasediments (Nanling-Yunkai domain), along the Zenghe-Dapu fault, suggests a source in a Grenvillian orogen, and the Hf isotopic data indicate that they more precisely would come from a late Mesoproterozoic magmatic arc developed onto a Paleoproterozoic crust (Wang L.j et al., 2008). It is thus suggested that the ZDFZ could mark the boundary of a Grenvillian suture. In this connection, it must be noted that, in Hainan Island, to the SE of the Baisha fault zone which is likely in prolongation of the Zenghe-Dapu fault, the sediments of the Yalong Bay (in provenance of this SE domain) yield detrital zircons with Neoproterozoic ages, ranging from 984 to 728 Ma (Xu Y.h et al., 2014). Although these zircons yield HfTDM2 ages peaking at 1.6-1.3 Ga (Xu Y.h et al., 2014), reminiscent of the Baoban Complex, the ca. 1.0 Ga age would be fitting with a Grenvillian event associated to the ZDFZ.

Basaltic pillow-lavas, interpreted as ophiolite remnants, are observed along the ZDFZ and have been dated at 853 ± 4 Ma (U-Pb, Zrn: *in* Shu L.s et al., 2007). In the Jiangnan orogen, the Jinning cycle also overprints the Grenvillian Sibao event and emphasizes the similarities

between the Jiangshan-Shaoxi FZ and the Zenghe-Dapu fault. This raises the possibility that the E Cathaysia was amalgamated to the W Cathaysia basement approximatively at the same time as the latter was sutured to the Yangtze craton, and that the Zenghe-Dapu Fault Zone could be another suture in the South China (meta)craton. This would be consistent with the point of view that W and E Cathaysia were distinct terranes (Xu X.s et al., 2007), the latter with its Paleoproterozoic crust and no reworking prior to the Late Yanshanian (Cretaceous), whereas the former, with also a Paleoproterozoic crust, displays a major Mesoproterozoic crust-forming event, and was strongly reworked by the “Caledonian” and Indo-Sinian events. That the ZDFZ is a lithospheric-scale structure is demonstrated by seismological data. On the other hand, the Zenghe-Dapu fault could be only one of the large NE-trending lithospheric faults developed within the Cathaysia crust at an unknown time, and have been more strongly reworked than others.

4. The Jiangnan orogen

There is a consensus that the Yangtze and Cathaysia terrane were welded at the end of the Neoproterozoic, forming the South China Craton, and that the ~ E-W belt of folded and metamorphosed Neoproterozoic sedimentary and volcanic rocks which lay between them (the Jiangnan belt) does represent the welding terrane, formed during the Jiangnan orogeny (e.g. Charvet et al., 2013 and references therein). The precise timing of the end of the Jiangnan orogeny is however a highly controversial subject (e.g., Yao J.I et al., 2013 and references therein). There are two conflicting points of view: on one hand, some authors consider an ending of the orogeny at the youngest at c. 860 Ma, with a major development around 0.9 Ga (e.g., Li Z.x et al., 2007, Li W.x et al., 2008, Li X.h et al., 2009). On the other hand, many workers claim that the Jiangnan orogenesis was not ended before ca. 830 Ma (Wang X.I et al., 2006, Hu and Zhou 2012) or even 800 Ma (Yao J.I et al., 2013). Given the arguments for one or the other point of view (see below), it must be concluded that the Jiangnan belt is indeed

the product of two successive orogenic events, which could be named the Sibao and Jinning events (using two other names usually given to the Jiangnan orogen) (Figure 13).

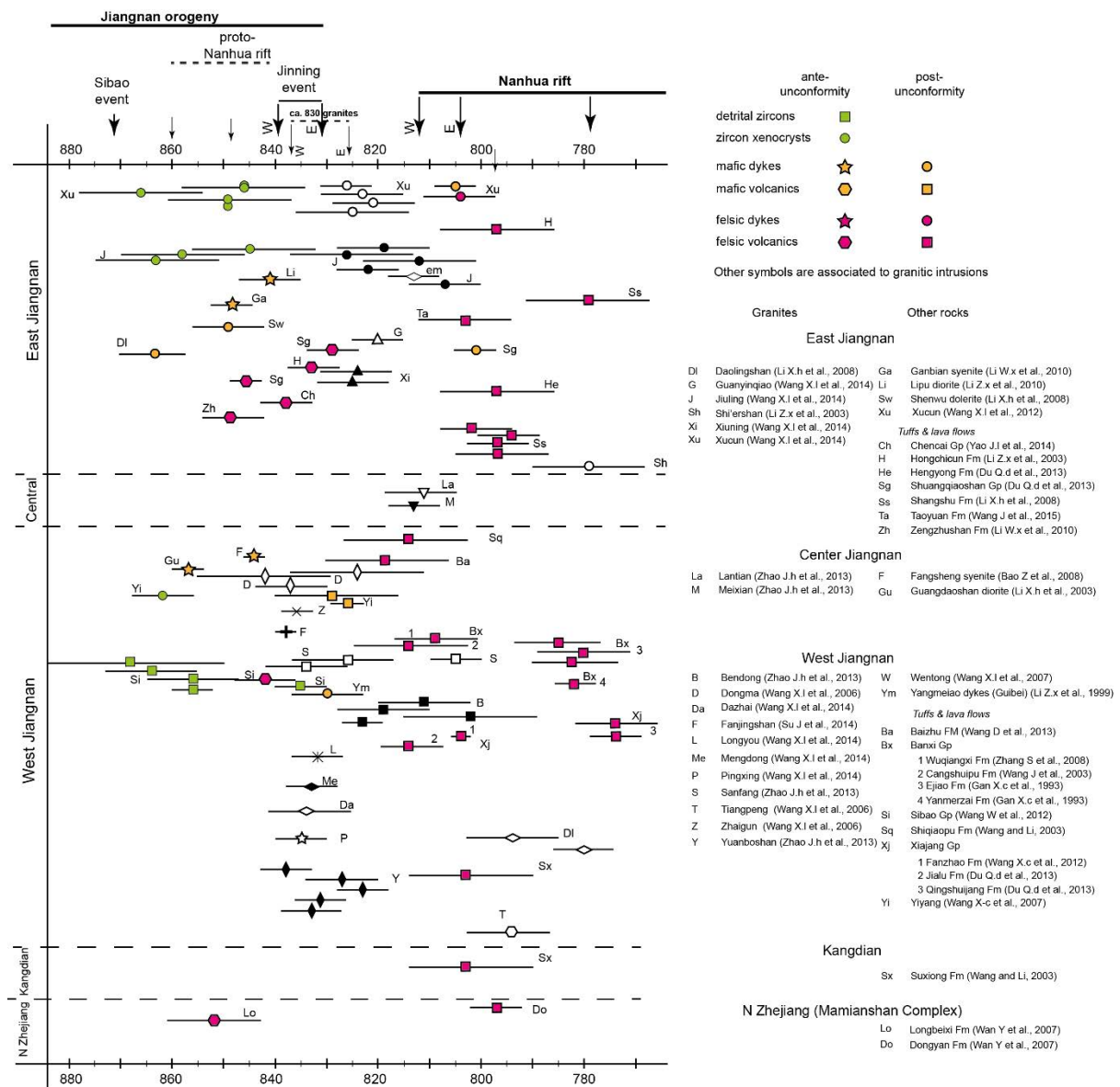


Figure 13. Compilation of dating results concerning the 880-760 Ma period in the South China Craton

When discussing the geodynamic significance of a given set of magmatic rocks, it is necessary to realize that a “subduction geochemical signature” is not *per se*, sufficient to demonstrate a supra-subduction setting. This is because it is well known that the processes that may have affected the lithospheric mantle at a given time of the regional history can be reflected in the products of mantle partial melting several 10 Myrs after the cessation of these processes. This is particularly true for the SCLM refertilization processes during subduction

(i.e. mantle enrichment in volatiles and incompatible elements). For instance, the Miocene magmas of the Apuseni Mts (Romania), although clearly produced in an intra-continental extensional setting, display typical subduction signatures, recording in fact the Tethyan subduction more than 50 Myrs earlier (Harris et al., 2013). This evidently applies to the Jinning event.

4.1. The accretionary Sibao orogen

The main argument for the Jiangnan orogeny suturing the Yangtze and Cathaysia terranes is the presence, in the easternmost segment of the belt, of ophiolitic rocks and arc terranes, the age of which are usually taken to be Neoproterozoic.

Arc and back-arc terranes in the E Jiangnan belt: The (back-arc) ophiolitic packages are dismembered within an ophiolitic melange (the Xiwan melange) elongated along the North Jianxi Shear Zone (NJSZ) (or Dexing- SZ), considered as a suture. The ophiolites were dated at ca. 1.0 to 0.9 Ga by Sm-Nd whole rock isochrones (Yao J.l et al., 2013). Adakite-like magmatic rocks are present in the melange, and one was dated at 968 ± 23 Ma (Li Z.x et al., 2007). Blue-schist rocks are also present in the Xiwan melange, and a glaucophane yielded an Ar/Ar age of 866 ± 14 Ma (in: Shu L.s et al. 2007). Obduction of the Xiwan ophiolites was associated with partial melting in the sole, yielding undeformed “obduction-type” leucogranite lenses intrusive in serpentinites and dated (U/Pb, Zrn) at 880 ± 19 Ma (Li W.x et al., 2008).

The arc terrane package (Huayu arc) is separated from the foreland (Jiuling terrane) by the NJSZ (Yao J.l et al., 2013). The Huayu arc is represented by the Shuangxiwu Group, which is dated at ca. 970 Ma (Pingshui basalt-andesites, in: Li X.h et al., 2009). The deformed and metamorphosed (in the greenschist facies) rocks of the Shuangxiwu Group are overprinted by undeformed dolerite dykes with an intra-plate signature, dated at 849 ± 7 Ma and unconformably covered by sediments of the Nanhua rift (≤ 820 Ma) (Li X.h et al., 2009).

The Tianli Schists, which were deposited after 1530 Ma on the Yangtze margin, exhibit polyphase deformation and low-grade metamorphism dated (Ar/Ar) from 1042 ± 7 Ma for the

earliest to 942 ± 8 Ma for the latest (Li Z.x et al. 2007), in line with the existence of syn-kinematic tonalites and granodiorites with a ca. 910 Ma age (Ye M.f et al., 2007). The age difference between the ≥ 0.95 Ga event(s) in the Tianli Schist and the ca. 870 (≥ 860) Ma late events in the Xiwan ophiolites was interpreted by Li Z.x et al., (2007) as recording a late opening to closing cycle of the NE Jiangxi back-arc following a first collision stage recorded by the Tianli Schists in the Jiuling terrane.

The better argument however for the existence of an old (1.0-0.9 Ga, ≥ 860 Ma) accretionary belt at the Yangtze-Cathaysia junction is the evidence for rocks with these ages beneath the Jiangnan belt. Such an evidence is provided by inherited cores in the zircons of a series of 830-800 Ma granites intrusive in the Jiangnan belt. The inherited cores yield ages in the 1200-870 Ma range (Wang X.I et al., 2006), and thus provide evidence for the partial melting of a concealed rocks of this age at depth.

4.2. The Jinning event: an inverted proto-Nanhua rift?

Three arguments are presented in favour of a more recent than ca. 860 Ma age for the Jiangnan orogeny. First, intensely folded metasediments, which are unconformably covered by the Nanhua rift sedimentary packages (≤ 820 Ma, see below), contain detrital zircons and are associated with datable volcanic layers, both yielding sedimentation ages the youngest of which are close to ca. 830 Ma (Wang W et al., 2012). Second, many of the bi-modal volcanic rocks associated with the Jiangnan metasediments displaying a typical “subduction signature”, are interpreted as recording the existence of a back-arc basin, closed towards 830 Ma (Wang et al., 2012). Third, a series of ca. 825 Ma Crd-Bt granodiorites, which overprint the Jiangnan metasediments, but are unconformably covered by the Nanhua sediments, exhibit typical S-type signatures and “generally plot in the collision-related areas of the tectonic discrimination diagrams”, which is interpreted as them being post-collisional for the just finished Jiangnan orogenesis (Wang X.I et al., 2006).

4.3 The ca.830 Ma granitoids and the age of the Jinning event

The age of the Jinning event is bracketed by the youngest ages provided by detrital zircons in Sibao Group sediments and/or volcanic intercalations, on one hand, and the age of a series of Bt-Crd granodiorites that are intrusive into these metamorphosed (low grade) and intensely folded sediments.

A compilation of available data yields two results: (i) there is no more in a given area than a few Myr between the youngest Sibao metasediment and the oldest grantoid intrusion, thus tightly constraining the age of the Jinning event, and (ii) there is a diachronism between the west and east segments of the Jiangnan belt, with a 10 Myr difference between the west (Jinning event at ca. 840 Ma) and the east (Jinning event at ca. 830 Ma), although in each area the duration of the granite event is about 10 Myr, with some overlapping between west and east.

However, Yao J.I et al., (2013) claim that the end of the Jinning event could not be older than c. 800 Ma, on the basis of the dating of detrital (magmatic) zircons in metasediments from the Huaiyu arc “basement” (considered as an equivalent of the Sibao Group), which contain zircons as young as 796 ± 10 Ma. Such a result is evidently in contradiction with the preceding data. However, as noted by Li W.x et al., (2008), the alleged “basement” phyllites and greywackes, although deformed under very low-grade conditions, are much less deformed and metamorphosed than the Tianli Schists, and as they rework Neoproterozoic magmatic zircons recording both the Sibao orogeny (970-860 Ma) and the early post-orogenic magmatism (850-830 Ma), they obviously cannot represent the Jiangnan “basement”, thus invalidating the argument of Yao J.I et al., (2013). The deformation of the cover sediments observed in the Huaiyu terrane must therefore be ascribed to a more recent episode, most likely the “Caledonian” event.

4.4 Meaning of the Jinning cycle: an inverted proto-Nanhua rift?

Other workers, on the other hand, claim that the Jiangnan orogenesis was not ended before ca. 830 Ma (Wang X.I et al., 2006, Hu and Zhou 2012) or even 800 Ma (Yao J.I et al., 2013). In the western end of the Jiangnan belt (N Guianxi), Wang X.I et al. (2006) have dated a series of late granites which yield two age groups: ca. 830 Ma granodiorites (between 835.8 ± 2.5 and 824 ± 13 Ma) and ca. 800 Ma Bt-granites (between 804 ± 5.2 and 794.2 ± 8.1 Ma), the latter, being coeval with “layered diabase” intrusions at 811.5 ± 4.8 Ma, are likely representative of the bi-modal magmatism associated with the Nanhua rifting (see below); tin mineralization is associated with the late Bt-granites. Wang X.I et al. (2006) argue that since these granites are of the S-type and “generally plot in the collision-related areas of the tectonic discrimination diagrams”, they must be related to the Jiangnan orogenesis. Yet, as seen above, post-orogenic intra-plate basaltic magmas are attested in the eastern Jiangnan belt as early as 849 ± 6 Ma (Li X.h et al., 2009). It may be noted that. In any case, according to Wang X.I et al., (2006), the ca. 830 granodiorites, which clearly overprint the low-grade metasediments of the arc series (the Sibao Group), are themselves unconformably covered by the sediments of the Nanhua rift (the Danzhou Group). The latter are not older than 745 ± 10 Ma, the age of the youngest zircon found by Yao J.I et al., (2013) in the greywackes of the Denshang Formation of the Huaiyu terrane (equivalent to the Denzhou Group: Wang X.I et al., 2006). These greywackes mainly reworked magmatic zircons with ages comprised between ca. 950 and ca. 800 Ma (Yao J.I et al., 2013), indicating that erosion of the Jiangnan belt, including the post-orogenic granites, was the main source of these sediments.

Finally, it may be concluded that the Jiangnan orogeny mainly occurred between 1.0 and 0.9 Ga, and was definitely ended at ca. 860 Ma. As noted by Ye M.f et al., (2007), this age may be compared with that of other 1.0-0.9 Ga orogenic belts such as the Eastern Ghâts Belt (India) or the Rayner Province (East Antarctica), which are known to contribute to the final welding of the Rodinia supercontinent, and thus, the Jiangnan orogeny must also be considered as part of the late Grenvillian events.

5. From the Nanhua Rift to the Kwangsian orogen

5.1. The ca. 830 Ma episode

Following the Jiangnan orogeny, post-orogenic granites were emplaced at ca. 825-830 Ma (see above) not only within the orogenic belt but also in the two surrounding cratonic areas, and were immediately exhumed to be unconformably covered by sediments of the Nanhua Rift (see below). These granites, which define a “Jiuling suite”, are clearly of crustal origin, with Hf model ages of their zircons at ca. 1.9 Ga (Zheng Y.f et al., 2007). Intra-plate dolerite dykes dated at 849 ± 7 Ma in the Shuangxiwu group of the Jiuling terrane (Li X.h et al., 2009), and ultra-mafic dykes dated at 828 ± 7 Ma in the Huaiyu terrane (in: Wang X.l et al. 2006), both unconformably covered by sediments of the Nanhua system, suggest that this post-orogenic magmatism was bi-modal and could therefore testify for a first rifting stage in the SCmC (Li X.h et al., 2009).

However, along the JSFZ, the ca. 830 Ma granites are overprinted by late sinistral ductile shearing (Yao M.f et al., 2013 and references therein), for instance a 825 ± 3 Ma Crd-granite, the shearing of which was sealed by the pre-Nanhua unconformity (ca. 0.75 Ga) (Charvet, 2013). This “late Jiangnanian” reactivation of the Jiangshan-Shaoxi fault zone, more than 40 Myr after the end of the orogeny, is very similar to the “late-Pan-African” reactivation of the Pan-African megashear zones in the Touareg Shield. It may thus be speculated that, just as in the Touareg Shield, linear delamination along the reactivated JSSZ could have been at the origin of the ca. 830 Ma magmatism in the Jiangnan orogen.

5.2. The failed Nanhua Rift

Starting very soon after the ca. 830 Ma episode, a major rifting event is attested in the SCmC by an unconformity, siliciclastic sedimentation and a bi-modal magmatism, giving way in the western Yangtze craton to the N-S Kangdian Rift, and in W Cathaysia and the adjacent Jiangnan belt to the NE-trending Nanhua Rift, itself subdivided in several sub-rifts, and

extending from the Anhua-Luocheng fault to the Wuchi-Yunkai domain (e.g., Wang Y.j et al., 2011).

In the Guangfeng sub-rift, the volcano-sedimentary Taoyuan Formation which unconformably covers the Tianli Schists is dated at 827 ± 14 Ma (Li W.x et al., 2008), whereas in the N Zhejiang sub-rift, the Shuangxiwu Group is overprinted by the Daolishan “granite-diorite” complex, dated at 794 ± 9 Ma and likely feeding the bi-modal volcanism (basalt-rhyolite) of the unconformable Shangsuo Formation, with similar 797 ± 11 to 792 ± 5 Ma ages (Li X.h et al., 2008). In the Mayuan Complex, the Nanshan Formation is the oldest Neoproterozoic metasedimentary formation found together with Paleoproterozoic basement rocks and is dated at 807 ± 12 Ma (Wan Y.s et al., 2007). The Mamianshan bi-modal metavolcanic rocks are dated at 816 ± 9 Ma (Li W.x et al., 2005). These data show that the rifting was initiated as early as ca. 820 Ma, with coeval sedimentation and bi-modal magmatism in the 820-800 Ma range. The Jiangshan-Shaoxi fault zone is thought to have been one of the main rift faults at that time. Along the ZDFZ, there are manifestations of a bi-modal volcanism, with tholeiitic basalts dated (U-Pb Zrn) at 795 ± 7 Ma, and rhyolites dated at 818 ± 9 Ma (in: Shu L.s et al., 2007), and it is therefore likely that this fault, too, was active in the rifting process.

The importance of the Cryogenian sedimentation in the W Cathaysia Block has been underestimated for a long time. Recent dating in both the Wuyishan (Mayuan Group) and the Yunkaishan domains demonstrates however that most of the metasediments in these areas contain very young zircons of late Neoproterozoic (≤ 0.8 Ga) to early Paleozoic ages (Wan Y.s et al., 2007, 2011). Cryogenian sedimentation and bi-modal magmatism were continued until ca. 750 Ma (e.g., Li W.x et al., 2008) or 730 Ma (e.g., Xu X.b et al., 2011), with a second generation of granitic intrusions at ca. 760-750 Ma defining the “Chenjiang suite” characterized by a significant juvenile component (Zheng Y.f et al., 2007). Most of the amphibolite layers or lenses found in the metasedimentary rocks of the Cathaysia basement (Mayuan, Badu, Yunkai and Zhoutan Complexes) were found by Li L.m et al., (2011) to yield very consistent late Mesoproterozoic Hf model ages at ca. 1.2-1.1 Ga, possibly pointing to a distensive event of

this age in W Cathaysia. However, these amphibolites are more likely of Neoproterozoic age (800-750 Ma) and would be part of the bi-modal magmatic activity related to the Nanhua rift (Wang Y.j et al., 2012).

Following the Cryogenian rift stage, thermal subsidence lead to a thick succession of up to 7-8 km of Ediacarian (Sinian) to middle Ordovician siliciclastic deposits (Charvet et al., 2010 and references therein) that occupy the whole W Cathaysia and may be unconformable onto the Cryogenian formations and granites (Zheng Y.f et al., 2007). However, from the middle Ordovician to the Silurian, there was a northwestward (present coordinates) migration of the depocenters onto the Yangtze Block (in: Li Z.x et al., 2010), with formation of a foreland basin testifying for the beginning of the Kwangsian orogenesis.

6. The “South China Caledonian Fold belt”: Kwangsian orogeny

Everywhere in E Cathaysia, there is a strong unconformity below the middle Devonian, testifying for a major “Caledonian” tectono-metamorphic event in the Middle Paleozoic.

The Kwangsian orogen is a double verging NE-trending belt, interpreted as resulting from the inversion of the Nanhua Rift (Charvet et al., 2010; Charvet, 2013) or (less convincingly) from an intracontinental subduction process (Faure et al. 2009). The Jiangshan-Shaoxi fault was reactivated as a thrust fault onto the Yangtze Block (e.g., Shu L.s et al., 2007, Li Z.x et al., 2010), as was also reactivated the ZDFZ (Wang Y.j et al., 2013). From the metamorphic point of view, it is highly dissymmetrical, with mainly low- to very-low grade Ediacarian (Sinian) and Cambrian-Ordovician metasediments to the west, and a high-grade metamorphic core (the Wuyi-Yunkai belt) running along the ZDFZ to the east (e.g., Yao W.h et al., 2012), and comprising most of the (reworked) basement outcrops. This was interpreted by Faure M et al., (2009) as the result of a decollement between the Neoproterozoic-Ordovician cover and the reworked older Precambrian basement, although, according to Lin W et al., (2008), there is a continuum between the schistosity of the low-grade and high-grade rocks. Moreover, it has

been recently demonstrated that many of the metasediments and metagranites in the Wuyi-Yunkai domain, which were classically considered as being Paleoproterozoic, are in fact metamorphosed Ediacarian (Sinian) and Cambrian siliciclastics sediments (Wan Y.s et al., 2007, 2010). Finally, in the Yunkai domain, high-grade rocks and migmatites appear as the core of metamorphic domes more or less rimmed by low-grade Sinian to Ordovician rocks (Wan Y.s et al., 2011).

In the Wuyi-Yunkai belt, the metamorphic grade is up to lower amphibolite (migmatites, including partially melted amphibolites) and granulite facies (in metasediments of the Yunkai domain). Metamorphic ages span a 468-428 Ma range (U/Pb zircon), whereas Ar/Ar dating (Ms, Bt, Hrn) on the mylonites of the main shear zones display a 438-406 Ma range (Wang Y.j et al., 2012, and references therein). The Kwangsian metamorphic P-T-t path was studied with some details by Wang Y.j et al., (2012) in the amphibolites of the Yunkai domain. They describe a three-stage metamorphic evolution: (i) prograde M1 (as relicts in garnet cores), with $\text{Pl-Qtz} \pm \text{Hrn} \pm \text{Ep}$, at 600°C and 6 kb, (ii) peak amphibolite facies M2, with $\text{Grt-Qtz-Hrn-Pl} \pm \text{Bt} \pm \text{Ep}$, at 720°C and 10.3 kb, and (iii) retrograde M3, with $\text{Pl-Hrn-Qtz} \pm \text{Bt}$ (Grt unstable) at 680°C and 5.3 kb. The P-T-t path is one of sub-isothermal decompression. The peak metamorphism is dated at c. 450 Ma and the retrograde path is dated at c. 435-430°C. In the Wushiyan area, migmatites (with a paragneiss mesosome) from the Mayuan Complex were studied by Liu R et al., (2010). They describe banded migmatites with biotite melanosomes and syn-kinematic leucosomes, clearly resulting from water present partial melting; migmatization was however evidently a two-stage process, since in some places garnet occurs in place of biotite (dehydration melting). When garnet is absent, the HREE-rich zircons from the mesosome yield a 453 ± 3 Ma age, easily interpreted as the age of the peak metamorphism (= migmatite 1). When garnet is present, however, zircons from the mesosome, which being HREE-poor were equilibrated with the garnet, yield a distinctly younger 442 ± 8 Ma age, consistent with dating in leucosomes (438 ± 5 Ma). There is thus a ca. 10 Ma gap between migmatite 1 and 2, and comparison with the previous data suggest that dehydration melting occurred along the

exhumation path. Describing the migmatite domes in the Yunkai area, Wan et al., (2010) state that “the magmatic component decreases gradually from the center to the margin” where are exposed the banded syn-kinematic migmatites, thus confirming that an important melt generation process (likely migmatite 2) overprinted the earlier migmatites. According to Lin W et al., (2008) this is in the Yunkai domain the result of “a post-orogenic syn-migmatite extensional event” following the “top-to-NW ductile shearing” associated with the peak amphibolite facies. In the Chencai Complex, Li Z.x et al., (2010) describe a similar evolution, with a four-stage metamorphic process: (i) prograde M1 (relicts in garnet cores), with Chl-Ms-Bt-Grt, (ii) peak M2 (regional schistosity), with Ms-Bt-Ky-Grt, (iii) static M3, with Sil (at the expense of garnet), and (iv) retrograde and post-kinematic M4, with Chl. The P-T-t path is also one of sub-isothermal decompression, but with evidence of thermal increase at low pressure, implying heat input in the orogen. A garnet micaschist yields a peak metamorphism age of 447 ± 7 Ma, while a Sil migmatite (paragneiss protolith) was dated at 433 ± 3 Ma (Li Z.x et al., 2010). *migmatization* (425 Ma in the Nanling Ranges: Xu X.s et al., 2005). In the Wuyi domain, the peak metamorphism of a Neoproterozoic amphibolite (Hrn-Pl-Qtz) was dated at 443 ± 6 Ma, consistent with the 452 ± 8 Ma age of an overprinting undeformed granite vein (Li Z.x et al., 2010), i.e., a c. 447 Ma age for the peak. In several places, the amphibolites suffered migmatization, owing to Hrn-dehydration melting (Li L.m et al., 2011). As hornblende destruction releases zirconium, some zircons in the amphibolite may yield the migmatization age. Metamorphic zircons yield ages of 446 ± 5 , 435 ± 2 and 434 ± 4 Ma (Li L.m et al., 2011), the former being clearly the amphibolite facies age, and the two others evidently recording the hornblende breakdown. Summarizing, it appears that the P-T-t path in the Wuyi-Yunkai high-grade domain was characterized by a syntectonic prograde stage implying both significant crustal thickening and heat input (the highest conditions recorded in the granulites being 750-880°C and 11-10 kb, in: Li L.m et al., 2011), at ca. 450 Ma, followed by a quasi-isothermal decompression path allowing water-present partial melting in the paragneisses at the onset of the exhumation and, owing to a new heat input, a second post-kinematic melting stage involving biotite and hornblende dehydration melting, at ca. 440-435 Ma. Evidence of heat input

in the Kwangshian orogen is provided by the observation of mafic intrusions coeval with the tectono-metamorphic evolution. At the beginning of the evolution, the Wugongshan appinites were emplaced along the Jiangshan-Shiaoxi fault at 473 ± 3 and 452 ± 2 Ma; they were derived from a metasomatised SCLM (Zhong Y.f et al., 2014). High-Mg basalts (the Chayuanhua suite) were recently described (Yao W.h et al., 2012); they are late-orogenic, being unconformably deposited onto Cambro-Ordovician sediments, but tilted under the, themselves unconformable, middle Devonian conglomerates. They were dated at ca. 435 Ma (Yao W.h et al., 2012), i.e., were coeval with the second (dehydration melting) migmatite event. They are interpreted as issued from a basaltic underplating resulting from the delamination of the eclogitised lower crust, following the crustal thickening episode (Yao W.h et al., 2012; Li Z.x et al., 2013; Xia Y et al., 2014) and this may be easily correlated with the late migmatisation event.

Kwangshian granites are abundant, and subdivide into “gneissoid” granites, for the most part located in the high-grade Wuyi-Yunkai domain, and “massive” granites, mostly found to the west of the Heynan-Guangfeng fault (Wang Y.j et al., 2011), i.e., within the low-grade domain. The latter cover 20 000 km² (Li L.m et al., 2011), while the former are likely to be numerous, having been by the past more or less confused with Paleoproterozoic granites (Wang Y.j et al., 2011). To the east of the Jiangshan-Shaoxi fault massive granites were emplaced from 454 ± 2 to 382 ± 2 Ma (Zhang F.f et al., 2012, Li Z.x et al., 2013), with however most of them around 430 Ma, while to the west of the fault, they are younger, spanning only the 429 ± 11 to 400 ± 4 Ma, with a peak at ca. 420 Ma (Zhang F.f et al., 2012). The “gneissoid” granites (a confuse notion, including magmatic foliation and subsolidus plastic deformation: Wang Y.j et al., 2011) span a similar age interval, and they were in the same way mainly emplaced between 435 and 430 Ma (Wang Y.j et al., 2011). Massive and “gneissoid” granites both contain paragneiss or amphibolite enclaves (Wang Y.j et al., 2011; Zhang F.f et al., 2012), in good agreement with their dating as mainly post-orogenic. The “gneissoid” granites are not, therefore, really syn-orogenic, since the main prograde deformation was at c. 450 Ma; they are

syn-kinematic of a shearing episode, at ca. 830 Ma (Xu X.b et al., 2011), and it may be suspected that this event was correlated with the extensional event – in other terms, although the word is not used for the Kwangsi orogen by the Chinese workers, the migmatitic domes and their related “gneissic” granites could well have been metamorphic core complexes. By the way, Wan Y.s et al., (2010 and references therein) interpret the whole Kwangsi orogen as “extension-detachment” related (Yao W.h et al., 2012). In any case, it is evident that the main episode of granite magmatism in the Kwangsi orogen was post-orogenic and coeval with intense migmatization and mafic magmatism.

Most Kwangsi granites, either massive or “gneissoid”, are of the “S”-type (Ms-, Tur- and Grt-granites) and derive of partial melting of the Cathaysian crust, with negligible juvenile components (Zhang F.f et al., 2012, Xia Y et al., 2014). A few monzogranite and granodiorite intrusions, only found in the core of the high-grade Wuyi-Yunkai domain, display a significant juvenile component, and were formed at a higher temperature (810-850°C) than the other Kwangsi granites (700-830°C) (Xia Y et al., 2014). These granites are among the youngest in the orogen, being dated at ca. 410 Ma, and they reworked the earlier granites, since they contain inherited zircon cores at 436 ± 5 Ma (Xia Y et al., 2014). They point to a permanence/renewal of the mafic underplating at ca. 410 Ma and span a 440-382 (± 2) Ma interval (Li Z.x et al., 2013 and references therein), with however a marked late-orogenic “peak” at 440-415 Ma (Yao W.h et al., 2012, and references therein). They consequently post-date the peak metamorphism (460-445 Ma: Yao W.h et al., 2012 and references therein) but are still coeval with 446 ± 5 to 423 ± 2 Ma in several amphibolites: Li L.m et al., 2011). The granites are mainly of the “S”-type and result of partial melting of the Paleoproterozoic crust, with only a very minor juvenile component, mainly represented by scarce coeval mafic enclaves, whereas a few intrusions, only found in the core of the high-grade Wuyi-Yunkai belt, display a significant juvenile contribution (Xia Y et al., 2014).

The origin of the Kwangsi orogeny remains unclear. Most workers call for “far-field stress related to collision-subduction systems beyond the southern margin of the South China Block” (Wang Y.j et al., 2012 and references therein), i.e., a Paleotethysian control.

7. The Indosinian events

A strong unconformity separates the Late Paleozoic to Middle Triassic shallow-platform sediments (2.5 to 3 km) from the overlying Mesozoic deposits including the late Triassic conglomerates, testifying for an Indo-Sinian tectonic event.

In the Cathaysia Block the Indo-Sinian events are observed in the same area as the “Caledonian” deformations that they overprint; they are however more expanded, being also observed in the Yangtze Block, where they overprint the Jiangnan belt. The early Indo-Sinian events include compressive tectonic deformations (fold-and-thrust belts), metamorphism and granite magmatism, the latter spanning a 265-205 Ma interval (Mao et al. 2013 and references therein) and covering 14,300 km² (Sun W.d et al., 2012). The great NE-SW faults are (re)activated during the Indo-Sinian episode.

There are in fact two Indo-Sinian magmatic episodes: a first one (245-230 Ma) with I-type granites, is associated with subduction and formation of the Song-Ma suture, to the SW of the SCC; a second one (218-210 Ma) with “S”-type Ms-, Crd- and Tur-granites, is pervasive in Cathaysia (Shu L.s et al., 2008 and references therein). The latter are associated with a first cycle of Nb-Ta mineralization in the Nanling Ranges (Mao et al., 2013), with several RMG (e.g., Limu granites), organized in three E-W trending belts about 30-40 km wide according to Shu L.s et al. (2007). This is however more evident for the Early Yanshanian granites. It may be recalled that the Mesoproterozoic Lanhe gneiss, in the Nanling Ranges, display E-W trending ductile shearing (Xu X.s et al., 2005).

The Indo-Sinian deformation is partitioned, with several NE-trending fold-and-thrust belts separated by zones of weaker deformations with only “Jura style” folding (Shu L.s et al.,

2007); most prominent are the Xuefengshan and Jiulingshan fold and thrust belts, within the Jiangnan orogen, and the Nanling fold and thrust belt, close to the ZDFZ, all with NW to NNW vergencies.

Following the compressive events, distensive conditions are realized from 200 to 180 Ma (e.g., Zaw et al., 2007), with the formation of several metamorphic core complexes (Faure et al., 1996, Shu L.s et al., 2007). According to Shu L.s et al., (2007), the three E-W belts of granitic intrusions testify for three linear zones of crustal thinning in the Nanling Ranges. It is not clear if the high-grade metamorphism recorded in several areas (and significantly in the former Wuyi-Yunkai belt) is related with the distensive events, although it is likely. In the Badu Complex, Xia Y et al., (2012) report ages of ca. 230 Ma and consider that granulite facies was attained, with the coeval intrusion of a quartz monzonite (226 ± 1.4 Ma).

The Indo-Sinian intracontinental event in the SCC is likely the result ("far field effects") of the penecontemporaneous collisions of (i) the SCC and the North China Craton (Qinling-Dabie-Sulu orogenic belt, with the ca. 220 Ma UHP metamorphism), and (ii) the SCC and the Indo-Sinian Block (Song-Ma suture, about 245-230 Ma) (e.g., Wang D.z et al., 2001, Shu L.s et al., 2008).

According to Li Z.x and Li X.h (2007) and Li Z.x et al., (2013), the Indo-Sinian crustal thickening was associated with the flat subduction of the Paleo-Pacific (Izanagi) plate, in the same way as the Laramian Cordilleran orogeny was related to the Farallon plate subduction.

8. The Yanshanian events

From the Early Jurassic to the late Cretaceous, the Tethysian tectonic was progressively replaced by the Paleo-Pacific regime (J_1 period) with the initiation of extensional basin and range tectonics in response to flat subduction of the Paleo-Pacific plate under the eastern Asian continental margin along the Japan Trench (Wang H.z and Mo X.x, 1995; Wang D.z and Zhou X.m, 2002; Li Z.x and Li X.h, 2007). Evidences of this regime are mainly observed in the

coastal areas of the SCB with the uplift of the Wuyi range (Shu L.s et al., 2009).

The Basin and Range structure are mainly composed of Precambrian basement and paleozoic meta-sedimentary rocks. Two depositional regimes coexist at the early $J_{1/2}$ period (200-165 Ma): the Tethysian regime composed of E-W trending carbonates and flyschs and the paleo-pacific regime that consists of NE-trending volcanoclastic rocks and red beds. In the Nanling belt, large E-W trending S-type granitoid zones are emplaced (Zhou J.c et al., 2006) and are considered as issued from the Tethysian regime or the post-collision Tethysian belt (Shu L.s et al., 2009).

The Paleo-Pacific plate subduction induced the remobilization of intracontinental faults previously formed during the Tethysian regime, and magmatism. The appearance of A-type granite at ca. 190 Ma was interpreted as the beginning of the slab break-off which evolved into large-scale anorogenic magmatism (Li Z.x and Li X.h, 2007). Igneous rocks, mainly observed in the eastern Nanling Range, are interpreted as evidences of the transition from the Tethysian compressional regime to the Paleo-Pacific extension regime (Wang D.z and Shu L.s, 2012). This change from to Basin and Range settings was interpreted as originated from the lithospheric rebound following the delamination of the flat slab (Li Z.x and Li X.h, 2007).

During the J_2 and J_3 periods (165-150 Ma), the Nanling-Wuyishan belt experienced large-scale granitic magmatism from 180 to 145 Ma (Zhou J.c et al., 2006) that controlled the formation of granite ranges and fault-depression basins. The first stage (180-160 Ma) is interpreted as caused from underplating of basaltic magmas that provided the necessary heat input for anatexis of Proterozoic sedimentary rocks (Wang D.z and Zhou X.m, 2002). The second stage (160-145 Ma) yields NE-trending batholith mainly exposed in the eastern Nanling Range and are constrained by the Paleo-Pacific plate subduction regime (Wang D.z and Shu L.s, 2012). From the 190 to 150 Ma, the magmatism progressively propagated from the center of the orogen to the broad inland region where fractionated I-type granites are associated with A-type granites and rift-type syenite during the slab rollback (Li Z.x and Li X.h, 2007)

At the K_1 period (150-110 Ma), all the SCB is affected by the influence of the Paleo-

Pacific plate subduction that gave rise to intracontinental deformation metamorphism inducing a wide intracontinental fold-and-thrust belt (Lin W et al., 2008; Zhang Y.q et al., 2008; Xu X.b et al., 2009). The low-angle subduction reaches the Chenzhou-Linwu (CL) fault and the Yangtze River at ca. 140 Ma (Ling et al., 2009) and creates a back-arc extensional environment in the whole SCB (Wang D.z and Zhou X.m, 2002; Shu L.s and Zhou x.m, 2002; Zhou X.m and Li W.x, 2000; Zhou J.c et al., 2006). This regime induced partial melting of the mantle of the upper slab, crustal thinning and underplating of basaltic rocks associated to upwelling of mixed crust-mantle magmas.

At ca. 136Ma, the thickened SCB lithosphere reached a limiting crustal thickness and started to collapse (Li J.h et al., 2013). From 136 to 118 Ma, the SCB is under an extensional regime followed by tectonic inversion between 117-108 Ma and these alternating settings will repeat at ca. 107 Ma (Li J.h, 2014). These variations are explained by the variation of the dip angle of the subducting slab and rate of convergence across the Paleo-Pacific subduction boundary (Li J.h, 2014). At ca. 100 Ma, a short-term uplift occurred in the coastal areas (i.e. uplift of pre-Cretaceous granites) in response to the possible collision between the eastern asia continental margin and the Philippine plate (Charvet J et al., 1985, 1999; Maruyama S et al., 1997; Shu L.s et al., 2004, 2009).

The K₂ period (110-70 Ma) starts at the end of this uplift period with the migration of the subduction zone of the Pacific plate eastward toward the high-angle Japan and Maliana trenches (Zhou X.m and Li W.x, 2000; Shu L.s and Zhou X.m, 2002). This extension generated multiple NE-NNE striking extensional basins and produced A-type granites and bimodal volcanism (Shu L.s et al., 2009). From this period, the subduction-type magmatism produced by melting of the subducting slab migrated from the SCB and thus, granitic magmatism ceased at ca. 90 Ma (Wang D.z and Zhou X.m, 2002). The extensional settings were followed by transpressional regime at ca. 86 Ma and caused a second tectonic inversion of the K₂ basins as expressed by the folding and strike-slip faulting observed in the SCB and the exhumation of the Jiangnan belt (Tian M et al., 2010; Shi W et al., 2012; Tang S.I et al., 2013).

9. Magmatism during the Jurassic-Cretaceous period

9.1 Introduction to the diagrams

Two types of diagrams are used in the following. These diagrams aim to combine interpretations of bulk-rock and mineralogical analysis for an integrated study. Both diagrams are constructed from oxides analyses turned into molar fractions and then used to build vectors that could project analyses in a given mineralogical reference.

The first diagram is named A-B diagram and was constructed by Debon and Le Fort (1983) (Figure 14A). This diagram is binary and opposes the aluminous character to the ferro-magnesian charge by defining two vectors: $A = Al - (Na+K+2Ca)$ and $B = Fe+Ti+Mg$ and discriminates the metaluminous domain (A negative) from the peraluminous domain (A positive). Here, the metaluminous domain includes all subaluminous, metaluminous and peralkaline rocks. The diagram can be divided in different sectors by lines rooted at the origin. As quartz and feldspars are located at the origin, the location of the rock in the diagram is representative of its characteristic minerals. The main granite lineages can be discriminated as the identification of the three RMG classes defined by Linnen and Cuney (2005). However, this type of representation is highly sensitive to hydrothermal alteration.

In order to deconvolute this possible inconvenience, we combined our A-B results with a second diagram called (QBF)³ by de La Roche et al. (1964) (Figure 14B). This ternary diagram defines three vectors:

- $Q3 = Si+7Al/2-5Ca-13(Na+K)/2,$
- $B3 = -7Al/2+7Ca+7(K+Na)/2+8(Fe+Ti+Mg)/3,$
- $F3 = Al+3Ca+4(K+Na)-5(Fe+Ti+Mg)/3.$

These vectors are then normalized to the sum of the three vectors in order to create the three poles Q³, B³ and F³. The (QBF)³ representation tends to disperse the alteration trends parallel to the differentiation trends in the A-B diagram (i.e. greisenization trend).

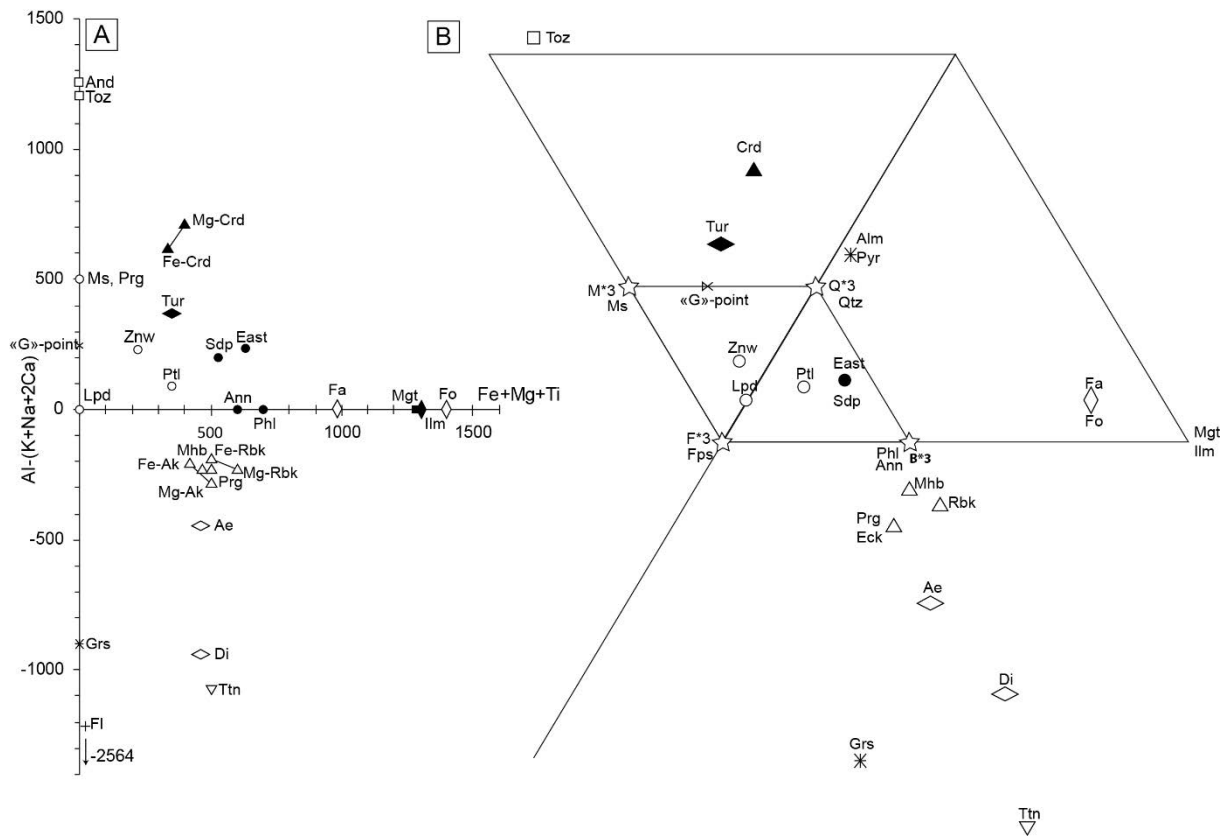


Figure 14. Theoretical diagrams used to combine bulk-rock and mineralogical analysis. (A) A-B binary diagram constructed by Debon and Lefort (1983), (B) (QBF)*3 ternary diagram constructed by de La Roche et al., 1964.

9.2 The $J_{1/2}$ period (200-165Ma)

Whole-rock analysis of rocks dated between 200 and 165 Ma are various and described mafic compositions (basalts and gabbros), intermediate compositions (granodiorite and syenite) and felsic compositions (granite and rhyolite) (Figure 15, Appendix A).

The mafic melts evolution is define by two distinct trends associated with basalt and gabbro analysis respectively (Figure 15). The basalt trend is mainly defined by the Changpu formation which associated with many basaltic magmas in the same area. Their geochemical characteristics describe an asthenospheric mantle origin, fractionation crystallization and variable degrees of contamination (Cen T et al., 2016). The grabbroic trend is defined by the Xialan and Xianli grabbros. The Xianli grabbros are associated to the Wengong granite and are described as derived from the melting of a depleted asthenospheric mantle source and also indicate fractional crystallization of the parental magma combined with variable degrees

of crustal contamination (Zhu W.g et al., 2010). The Xialan gabbro are also interpreted as derived from the asthenospheric mantle and subsequently modified by interaction with the overlying SCLM and contamination with the upper crust (Bai Z.j et al., 2015).

The felsic melts also display two groups of melts defining two sources of granites, each associated to a distinct mafic trend (Figure 15). The granitic pole associated to rhyolite compositions is mainly defined by the Waitun granites that are interpreted of crustal origin, derived from partial melting of a mixed source of meta-igneous and metasedimentary material, triggered by underplating of basaltic magmas (Wang G.c et al., 2016). The A-B and (QBF)*3 diagrams are in accordance with this interpretation. The other granitic pole is located at the centre of the A-B diagram and is depleted in the B*3 end-member and is mainly defined by the Wengong granite. This granite is described as a peraluminous A2-type granite formed from partial melting of the lower crust and mixed with mantle-derived basaltic magmas (Zhu W.g et al., 2010).

The intermediate compositions of granodiorite are aligned in trends between the felsic and mafic end-members (Figure 15). The granodiorite shows interaction between granitic and basaltic magmas. This interaction is in the continuity of the two end-members and is likely to define a mixing trend typical of I-type suites. On the other hand, syenites are the ultimate end-member of the gabbroic magmas but do not correlate to any felsic magma (Figure 15). In that case, this trend is not likely to represent mixing and is more likely to represent an A2-type suite associated with the A2-type Wengong intrusion.

9.3 The J_3 period (165-150Ma)

Whole-rock analysis of basalt, diorite, gabbro, MME, syenite lamprophyre and granite described heterogeneous compositions among the 165-150 Ma period (Figure 16, Appendix A).

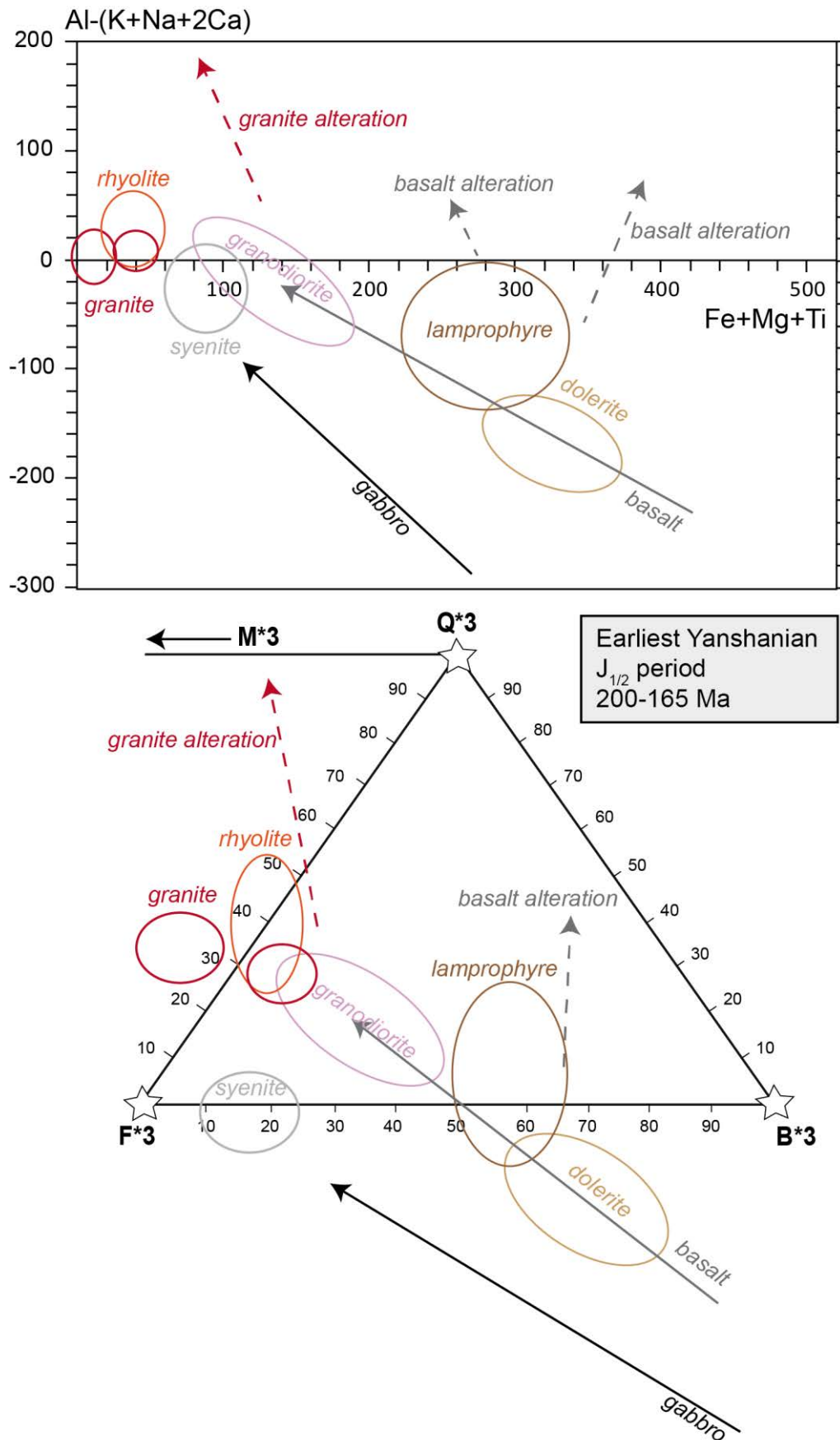


Figure 15. Schematic representation of the whole-rock analysis compilation dated between 200 and 165 Ma. See details in Appendix B1.

The mafic melts describe one trend composed of basalts, diorites and gabbros (Figure 16). The basaltic trend is defined by the Daoxian basalts which are described as alkaline and tholeiitic series. These basalts were formed from partial melting of shallow lithospheric mantle in response to thermal perturbations associated with rifting and continental arc settings (Jiang Y.h et al., 2009). The gabbros and diorite are described as the result of the mixing of mantle-derived juvenile basaltic magma and a magma issued from the dehydration partial melting of lower crust basaltic rocks (Xu X.s et al., 2007).

The felsic melts described by granite analysis show three trends converging toward one pole which is represented by numerous granite as, for example, the Mufushan complex, the Jiufeng complex and the Guposhan complex (Figure 16). These magmas are characterized as originated from a crustal-derived metasedimentary source or A-type granites reworked by basaltic mantle-derived magmas (Wang L.x et al., 2014; Huang H.q et al., 2015; Wang Z et al., 2014).

The Qitianling and Guposhan complex from the three intermediate magmatic trends (Figure 16). These trends are composed of granite and MME. These MME, aligned with their respective granitic associated rocks, show a partial mixing with the granites. This interpretation was also exposed by Wang Z. et al. (2014) and is interpreted as triggered by underplating of mafic magma from upwelling asthenosphere and/or metasomatised lithospheric mantle in an extensive regime. The three trends are bounded by the two mafic trends observed in the $J_{1/2}$ period, one of which is observable in the J_3 period.

One sample of syenite only was found in the literature (Shiling syenite, Xu X.s et al., 2007) and seems disconnected from all trends. More data need to be compiled before interpretation.

9.4 The K1 period (150-110Ma)

The whole-rock analysis associated to the 150-110 Ma in the literature are associated to granites, rhyolites, syenites, diabase, basalts and gabbros (Figure 17, Appendix A).

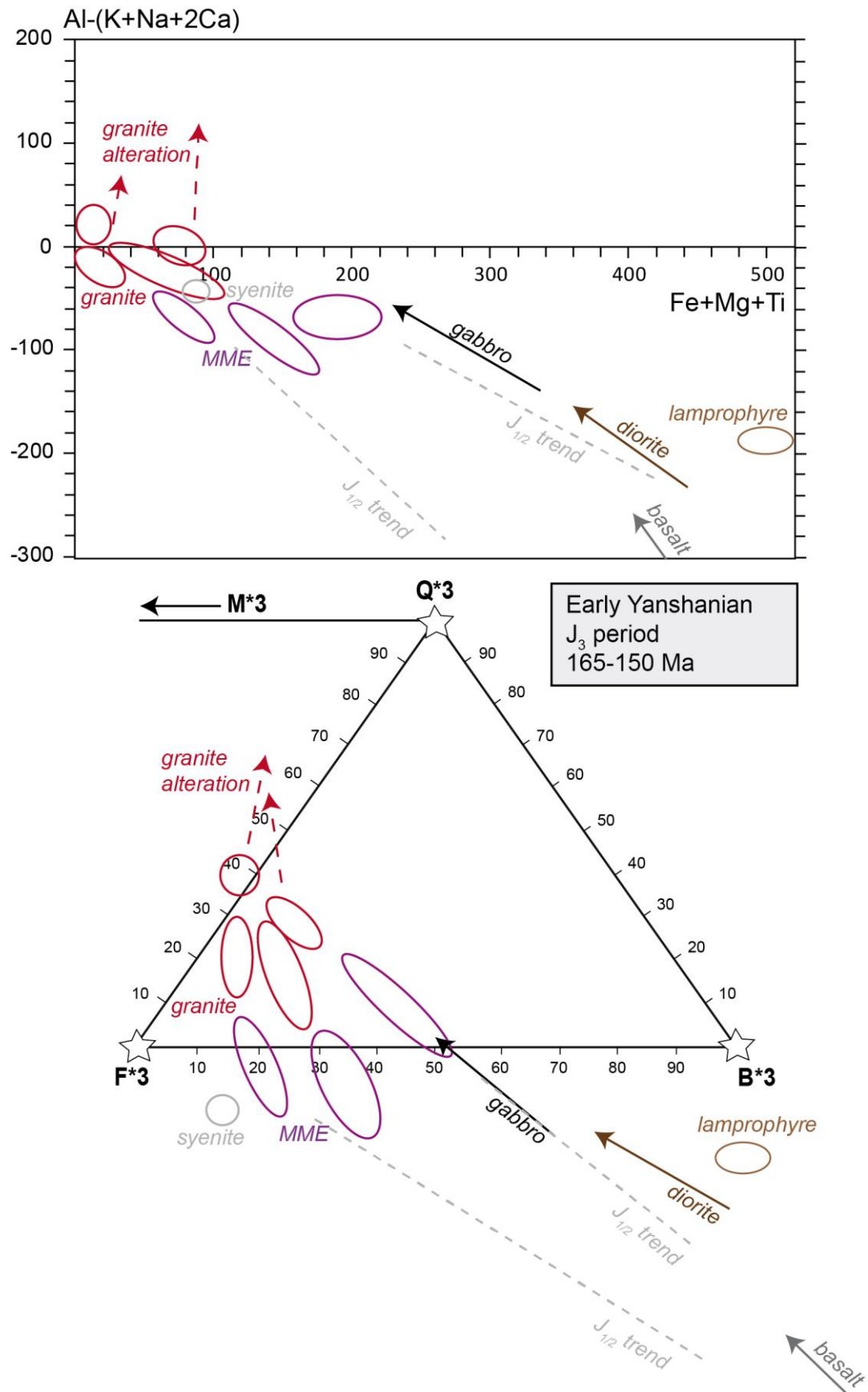


Figure 16. Schematic representation of the whole-rock analysis compilation dated between 165-150 Ma. See details in Appendix B2.

The mafic melts define one trend composed of gabbro and basalts. This trend is different from all mafic trends observed in the $J_{1/2}$ and J_3 periods. The basaltic trend is mainly characterized by the Ji'an-Taihe and Xinjiang Jinua-Qinzhou alkali basalts and tholeiites (Figure 17). These basalts are interpreted as originated from an enriched mantle and mixed crustal materials associated the mixing of some components of the subducting paleo-Pacific plate (Yu X.q et al., 2006). The gabbros are mostly characterised by the Daiqianshan-Pingtan complex (Figure 17). They are associated to dehydration melting of amphibolites during a thickening of the lithosphere to convert the pre-existing lower-crust basic rocks into amphibolites (Chen C.h et al., 2004).

Within the felsic melts, two main groups can be defined. The first one is characterized by the Suzhou complex, the Xiangshan rhyodacite and Baijuhuajian granite (Figure 17). On one hand, the Suzhou and Baijuhuajian granite are defined as A-Type intrusions issued from the remelting of ancient igneous protoliths triggered by input of depleted mantle-derived magmas (Gao Y.y et al., 2015; Wang J et al., 2009). On the other hand, the Xiangshan rhyodacite are interpreted as largely derived from Proterozoic metamorphic rocks, without significant amount of mantle-derived magmas (Yang S.y et al., 2010). The second group is defined by the Taoshan granitic complex (Figure 17). This complex is interpreted as derived from partial melting of metasedimentary rocks and defined as S-type (Zhao Z.f et al., 2015).

Intermediate compositions are defined by syenites from the Caiyuan-Hongong complex and the Tieshan complex located in adjacent regions (Figure 17). Both complexes are interpreted as derived from an enriched mantle metasomatized by subducted sedimentary material with a significant contribution of depleted asthenospheric mantle (He Z.y and Xu X.s, 2012; Deng Z et al., 2014). In the diagrams, the syenite are likely to represent cumulates with a fractionation trend toward the felsic magmas (Figure 17).

9.5 The K2 period (110-70Ma)

Whole-rock analysis of rocks dated between 110 and 70 Ma are described by gabbro, basalt, diorite, MME, syenite, granite and rhyolite (Figure 18, Appendix A).

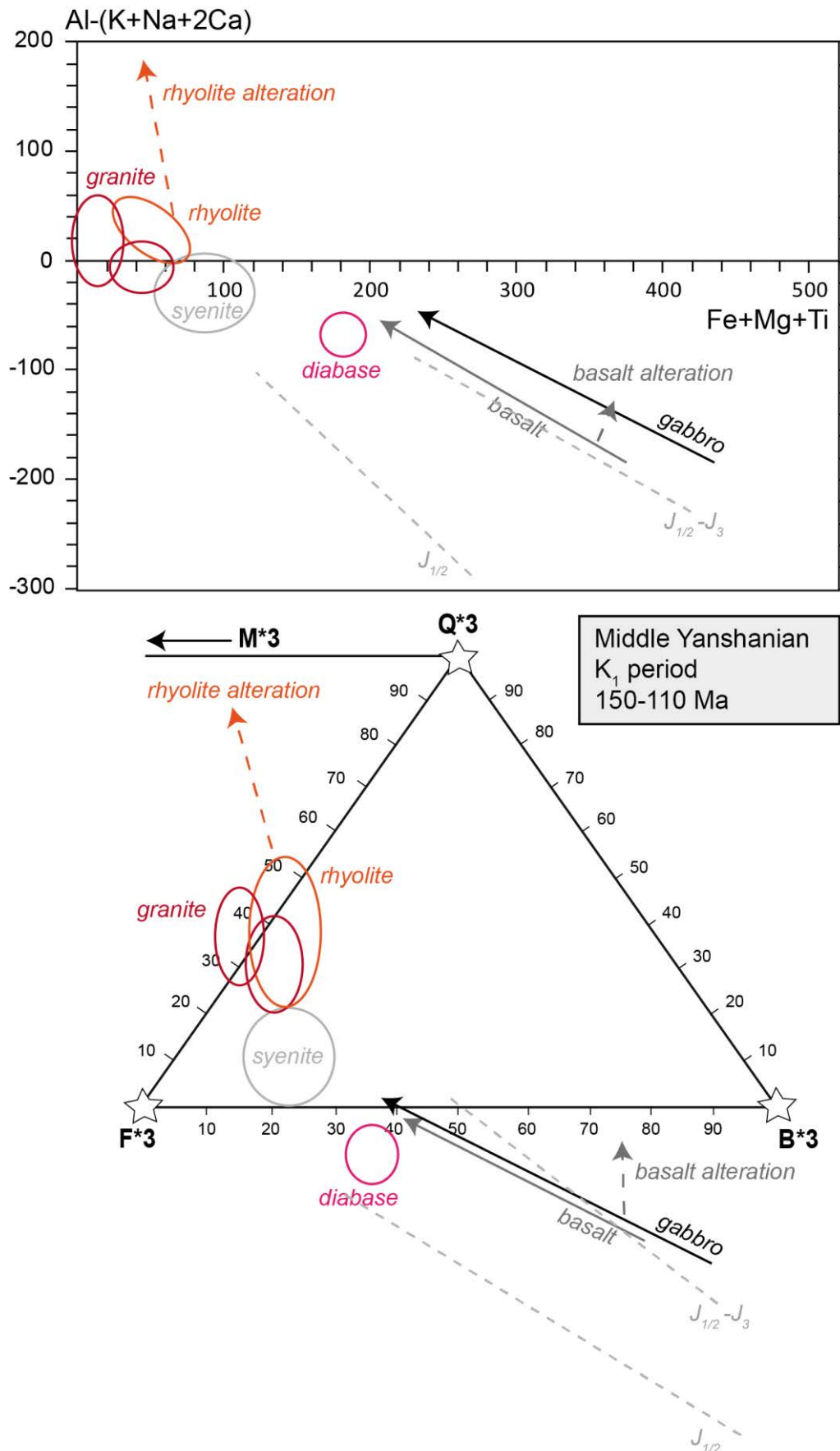


Figure 17. Schematic representation of the whole-rock analysis compilation dated between 150-110 Ma. See details in Appendix B3.

The mafic melts define two trends characterized by basalts and gabbros that were not observed in the $J_{1/2}$, J_3 and K_1 periods. The first trend is described by the Jiasha gabbros (Figure 18). These gabbros are interpreted as originated from parental magmas formed through partial melting of mantle sources that contained ancient crustal components or was previously enriched and underwent crystal fractionation and accumulation processes (Cheng Y.b et al., 2012). The second trend is defined by both basaltic and gabbroic rocks from the Pingtan-Dongcai-Donghueshan-Taohuashan-Quinglanshan-Daiquianshan grabbroic complex and Hengyang basalts (Figure 18). The gabbros are interpreted as derived from an arc-related source mantle modified by previous subduction and contaminated by crustal material during intrusion (Wang Z.h et al., 2002). The Hengyang basaltic compositions are explained by the mixing of asthenospheric melts with an increasing amount of slab dewatering-related melts that sunk to the mantle transition zone (Meng L et al., 2012).

The felsic melts are divided in two groups: a cluster associated to rhyolite and granite and a granitic trend associated with the MME (Figure 18). The cluster is characterized by the Taohuadao, Putuoshan complexes and Yandangshan rhyolites. The granitic complex are described of A-type and I-type affinity. Both granitic complexes and rhyolite are interpreted as originated from the mixing of mantle and crustal-derived magmas generated by the dehydration melting of basaltic rocks (Zhao J.l et al., 2016; He Z. et al., 2009). The granitic trend associated with MME indicates partial mixing with the granites. This trend is characterized by the Longchahe granite and Matou monzonite (Figure 18). They are interpreted as originated from the mixing of two components between peraluminous crustal melts and alkaline mafic magma (Cheng Y.b et al., 2012; Liu L et al., 2014).

The intermediate compositions are mainly composed of syenites from Yandangshan (Figure 18). As exposed in the K_1 period, these syenites are likely to represent cumulates with a fractional trend toward granitic melts. These syenite are interpreted as derived from high-pressure fractional crystallization from a basaltic magma likely originated by melting of mantle (He Z et al., 2009).

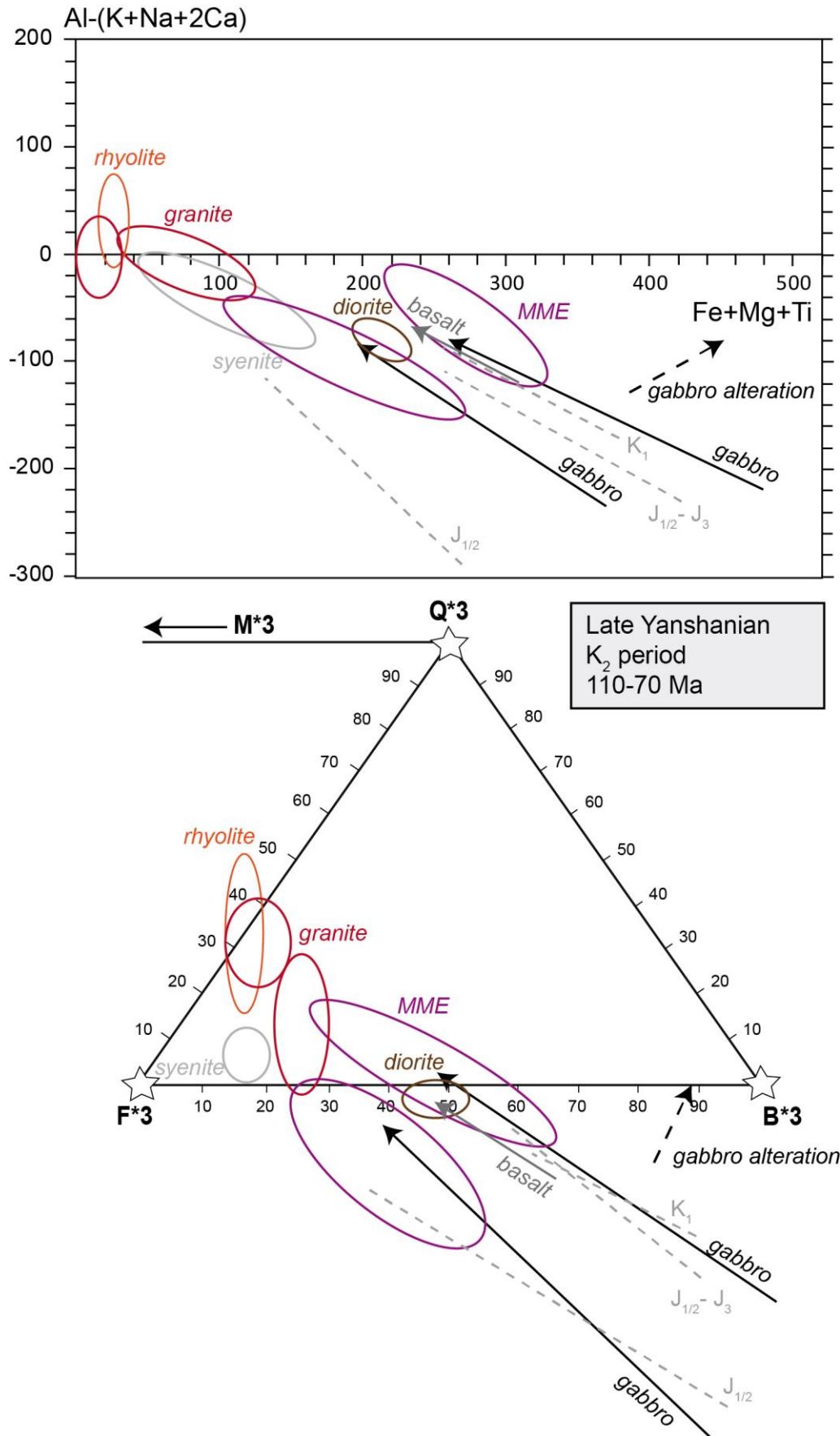
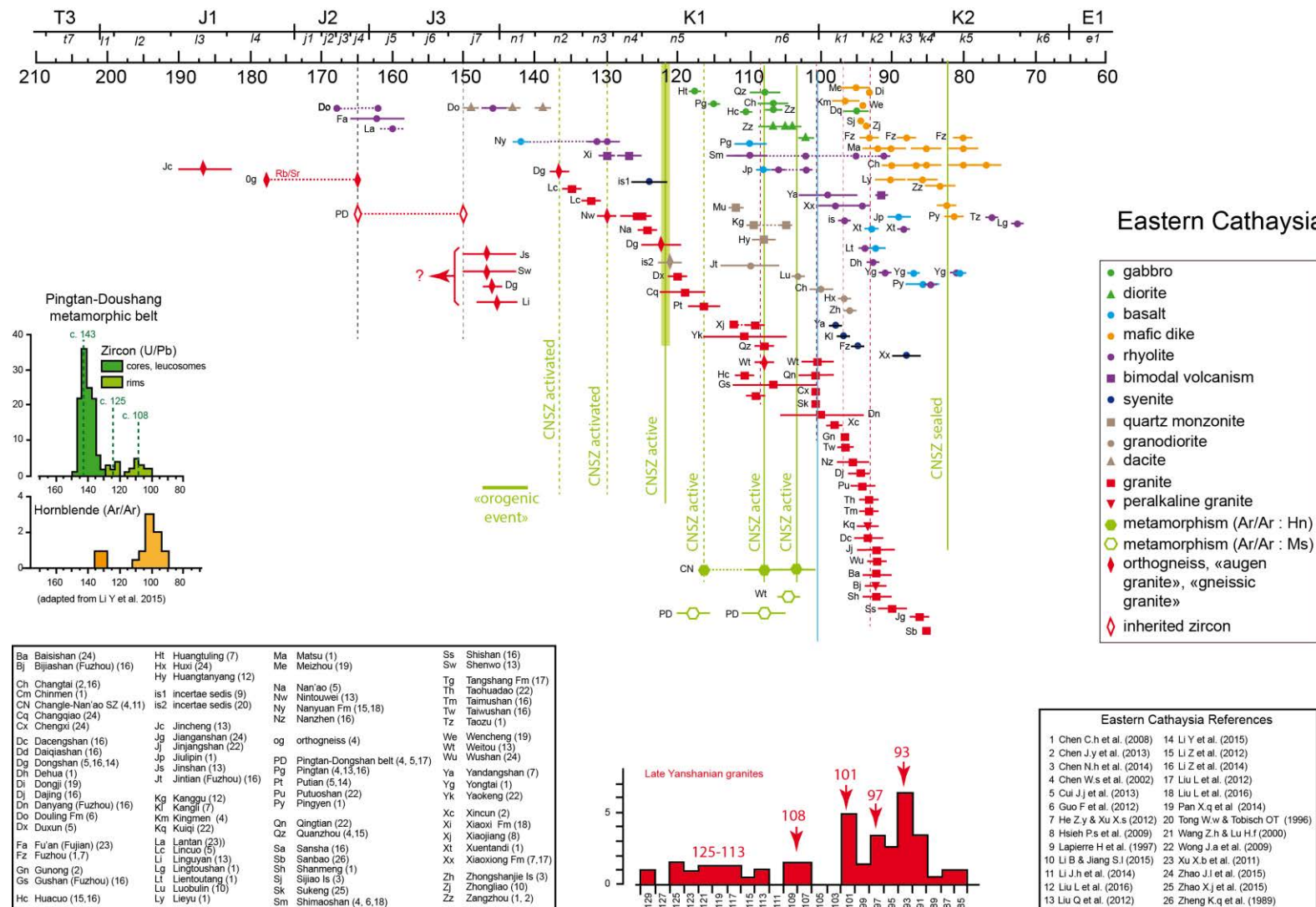


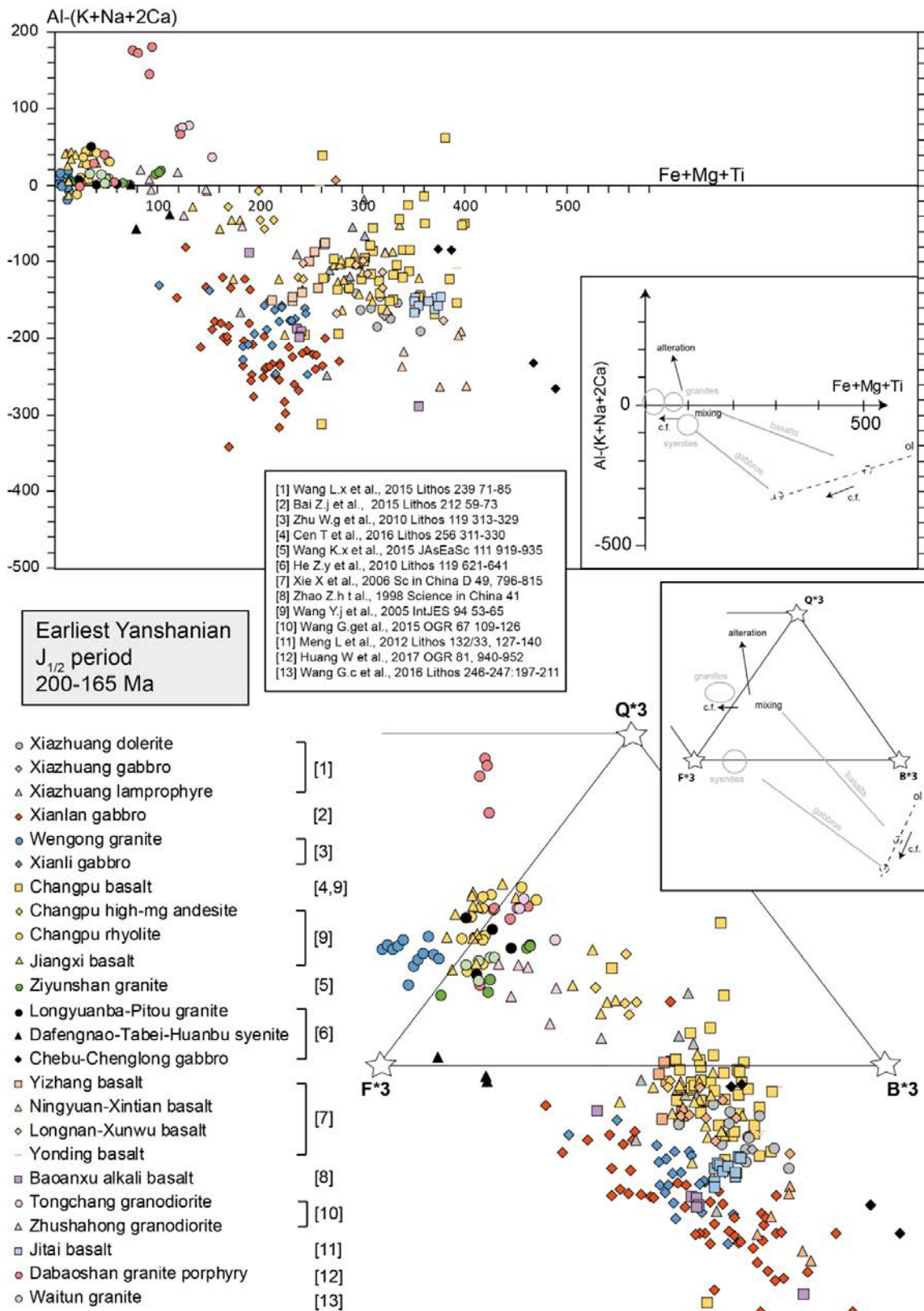
Figure 18. Schematic representation of the whole-rock analysis compilation dated between 110-70 Ma. See details in Appendix B4 and B5.



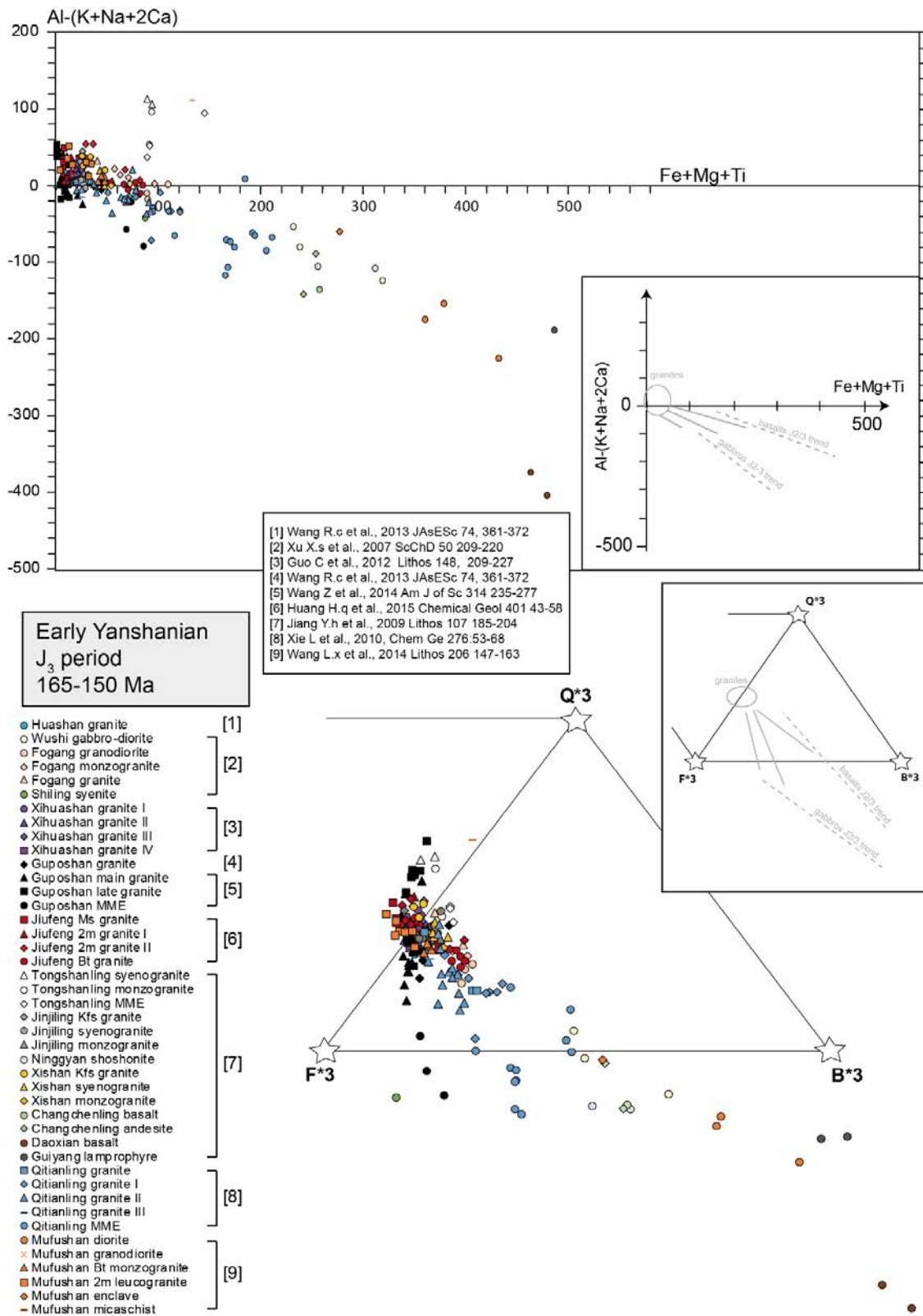
Appendix A2 - Compilation of dating analyses of rocks formed between 210 and 60 Ma in the Eastern Cathaysia Block



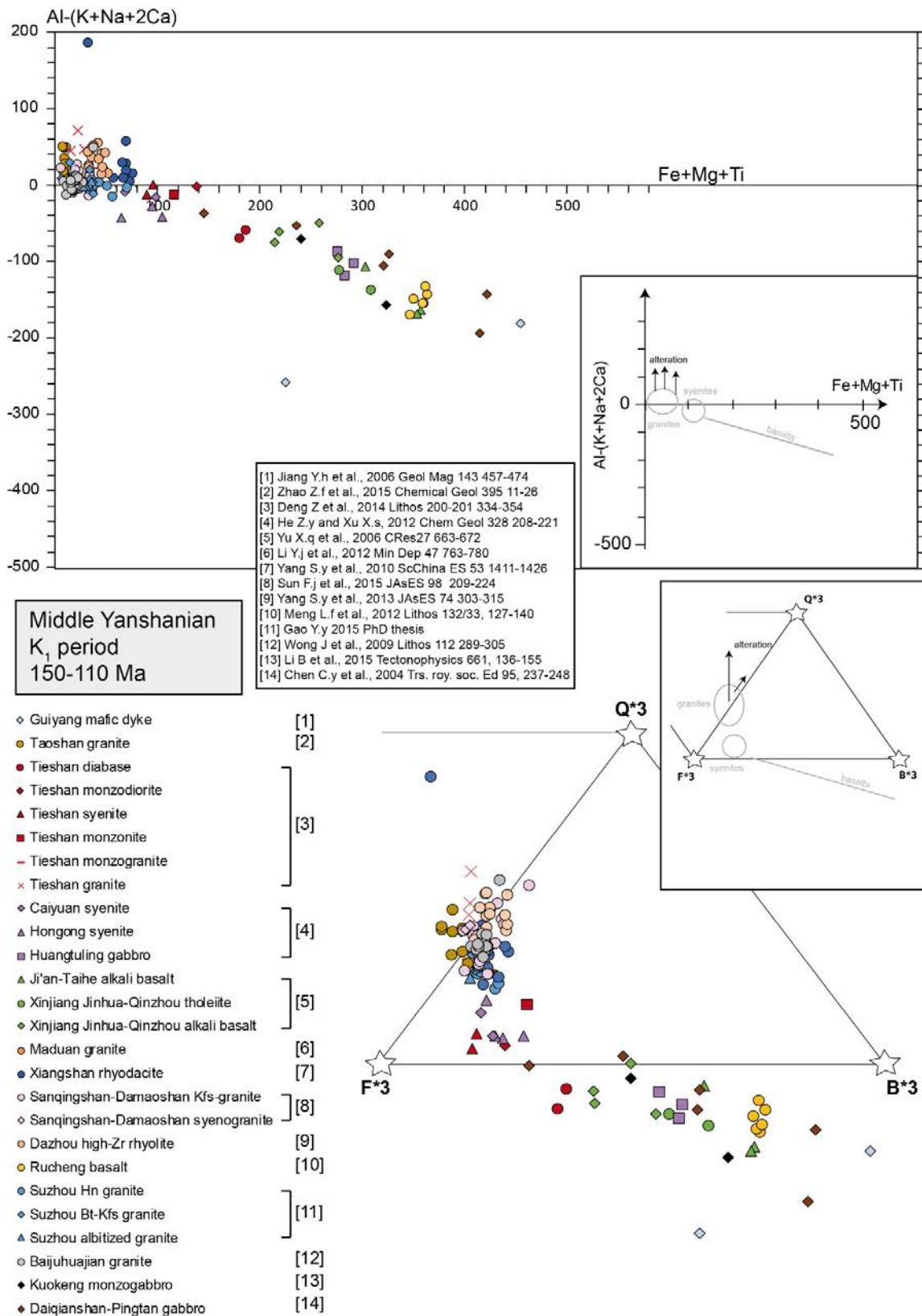
Appendix B1 – Compilation of whole-rock analysis from the J1/2 period in the Cathaysia and plotted in the A-B and (QBF)*3 diagrams (Debon et Lefort, 1983; de La Roche et al., 1964)



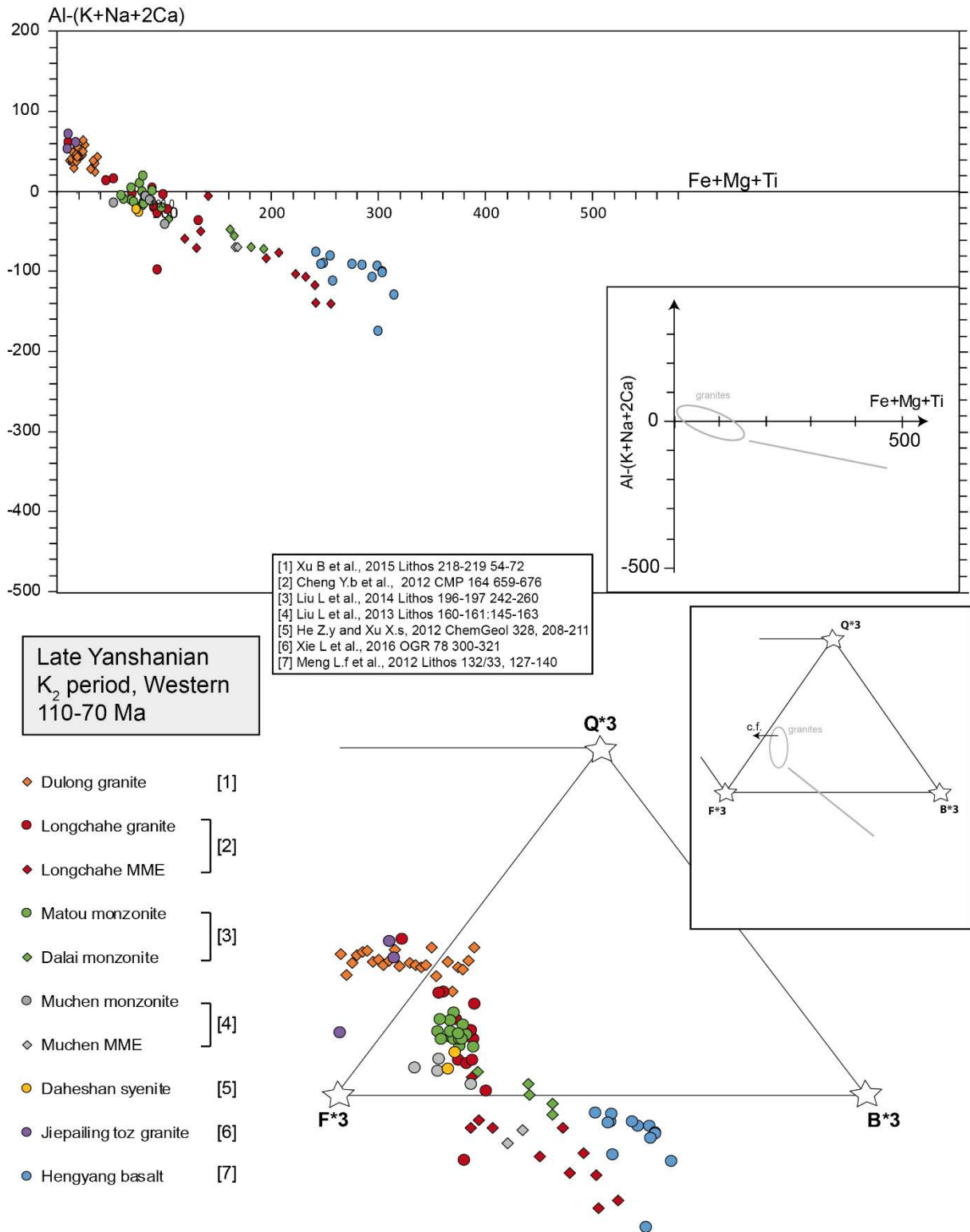
Appendix B2 – Compilation of whole-rock analysis from the J3 period in the Cathaysia and plotted in the A-B and (QBF)*3 diagrams (Debon et Lefort, 1983; de La Roche et al., 1964)



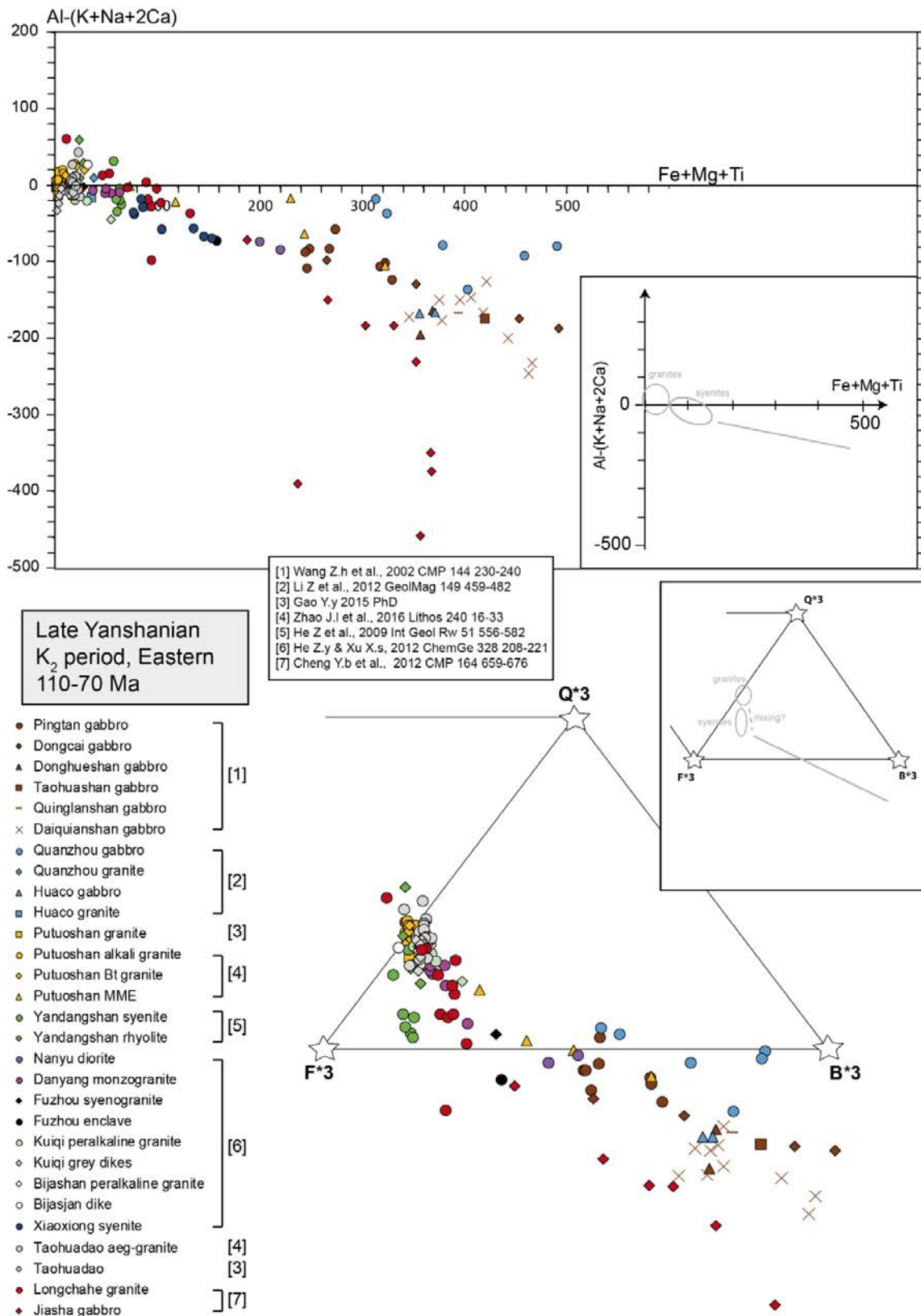
Appendix B3 – Compilation of whole-rock analysis from the K1 period in the Cathaysia and plotted in the A-B and (QBF)*3 diagrams (Debon et Lefort, 1983; de La Roche et al., 1964)



Appendix B4 – Compilation of whole-rock analysis from the K2 period in the Western Cathaysia and plotted in the A-B and (QBF)*3 diagrams (Debon et Lefort, 1983; de La Roche et al., 1964)



Appendix B5 – Compilation of whole-rock analysis from the K2 period in the Eastern Cathaysia and plotted in the A-B and (QBF)*3 diagrams (Debon et Lefort, 1983; de La Roche et al., 1964)



Appendix C – Whole rock data compiled from the literature and used in Appendix B1 to B5, organized by period of time.

	SiO ₂	TiO ₂	Al ₂ O ₃	FeO	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	Si	Ti	Al	Fe	Mn	Mg	Ca	Na	K	P	A	B	Q*3	B*3	F*3	
Earliest Yanshanian (200-165 Ma)																											
Xiazhuang (Wang L.x et al., 2015 Lithos 239 71-85)																											
Dolerite																											
09GD74	48.6	2.2	14.8	9.0	2.9	0.2	6.4	10.7	2.7	0.3	0.2	808.8	27.5	290.3	127.6	2.8	158.8	190.8	87.1	6.4	2.8	-184.8	313.9	-29.5	87.4	42.0	
09GD46	46.4	2.3	13.7	11.8	3.2	0.2	6.5	10.5	2.3	0.5	0.2	772.2	28.8	268.7	166.5	2.8	161.3	187.2	74.2	10.6	2.8	-190.6	356.6	-31.4	96.9	34.5	
09GD22	49.7	1.9	15.2	9.2	2.6	0.2	5.8	10.0	2.9	0.5	0.3	827.1	23.8	298.2	130.4	2.8	143.9	178.3	93.6	10.6	4.2	-162.7	298.1	-24.1	80.0	44.1	
09GD31	48.7	2.2	14.6	10.1	2.3	0.2	6.6	9.5	2.8	0.5	0.3	810.5	27.5	286.4	142.9	2.8	163.8	169.4	90.4	10.6	4.2	-153.4	334.2	-21.6	83.9	37.7	
09GD27	49.1	2.0	14.8	9.8	2.0	0.2	6.5	10.1	2.5	0.9	0.3	817.1	25.0	290.3	138.7	2.8	161.3	180.1	80.7	19.1	4.2	-169.7	325.0	-25.5	85.3	40.2	
09GD37	48.7	3.5	12.2	12.3	4.0	0.3	4.4	8.8	2.7	0.6	0.6	810.5	43.8	239.3	174.7	4.2	109.2	156.9	87.1	12.7	8.5	-174.4	327.7	-25.3	90.8	34.5	
09GD42	50.1	3.1	12.4	12.0	3.5	0.3	4.0	8.1	3.1	0.7	0.8	833.7	38.8	243.2	170.5	4.2	99.3	144.4	100.0	14.9	11.3	-160.5	308.6	-22.0	84.2	37.8	
09GD28	49.8	3.5	12.5	11.9	3.8	0.3	4.2	8.4	2.5	0.5	0.6	828.8	43.8	245.2	169.1	4.2	104.2	149.8	80.7	10.6	8.5	-145.7	317.2	-15.6	83.1	32.5	
09GD02	50.1	2.2	14.3	9.4	3.7	0.2	5.0	8.6	2.5	0.8	0.4	833.7	27.5	280.5	133.2	2.8	124.1	153.4	80.7	17.0	5.6	-123.9	284.8	-12.1	72.3	39.8	
09GD70	50.4	3.5	12.5	9.9	5.4	0.2	4.1	7.7	2.9	0.6	0.5	838.7	43.8	245.2	140.1	2.8	101.7	137.3	93.6	12.7	7.0	-135.7	285.6	-14.2	76.7	37.6	
09GD66	50.7	3.0	13.7	9.5	4.4	0.2	3.7	6.4	4.9	0.2	0.7	843.7	37.5	268.7	134.5	2.8	91.8	114.1	158.1	4.2	9.9	-121.9	263.9	-18.0	68.4	49.7	
09GD67	49.7	3.3	13.1	10.5	4.0	0.2	4.2	8.0	2.9	0.9	0.6	827.1	41.3	257.0	148.4	2.8	104.2	142.7	93.6	19.1	8.5	-141.0	294.0	-17.7	78.2	39.5	
Gabbro																											
09GD50	48.6	2.3	14.0	10.5	2.9	0.2	5.8	9.7	2.8	0.4	0.3	808.8	28.8	274.6	148.4	2.8	143.9	173.0	90.4	8.5	4.2	-170.2	321.1	-25.6	86.6	39.0	
Lamprophyre																											
09GD08	54.2	0.9	13.9	5.3	1.3	0.3	7.1	10.5	3.1	2.2	0.3	902.0	11.3	272.7	77.4	4.2	176.2	187.2	100.0	46.7	4.2	-248.6	264.9	-44.1	88.9	55.3	
09GD65	52.3	3.6	14.9	9.6	3.1	0.2	3.5	8.0	2.0	2.5	0.4	870.4	45.1	292.3	135.9	2.8	86.8	142.7	64.5	53.1	5.6	-110.7	267.8	-9.2	65.1	44.0	
XW0512	55.4	0.8	15.9	5.0	2.8	0.2	6.2	7.3	2.5	2.9	0.4	922.0	10.0	311.9	72.0	2.8	153.8	130.2	80.7	61.6	5.6	-90.7	235.9	-4.7	54.3	50.4	
XW0513	55.7	0.8	15.1	5.0	3.0	0.2	8.4	6.5	2.1	2.4	0.4	926.9	10.0	296.2	72.0	2.8	208.4	115.9	67.8	51.0	5.6	-54.3	290.4	8.5	55.2	36.3	
XW0514	56.0	0.9	15.1	5.0	3.1	0.2	8.8	6.4	1.0	2.6	0.4	931.9	11.3	296.2	72.0	2.8	218.4	114.1	32.3	55.2	5.6	-19.5	301.6	21.5	50.4	28.1	
XW0515	55.1	0.8	15.3	5.3	2.0	0.2	8.7	7.4	1.2	3.0	0.5	917.0	10.0	300.1	76.2	2.8	215.9	132.0	38.7	63.7	7.0	-66.2	302.1	6.5	59.2	34.3	
XW0516	53.9	0.8	15.3	4.8	2.8	0.2	8.4	8.6	2.3	1.8	0.5	897.0	10.0	300.1	69.2	2.8	208.4	153.4	74.2	38.2	7.0	-119.0	287.7	-9.3	67.6	41.7	
XW0517	55.3	0.8	15.5	4.6	3.9	0.4	4.1	9.6	2.0	3.0	0.4	920.3	10.0	304.0	68.9	5.6	101.7	171.2	64.5	63.7	5.6	-166.6	180.7	-22.9	62.5	60.4	
Xialan gabbros (Bai Z.j et al., 2015 Lithos 212 59-73)																											
XL323-01	37.4	2.9	10.4		24.3	0.2	9.6	11.3	0.9	0.1	0.0	622.4	35.8	203.0	2.6	3.0	239.0	201.5	29.0	1.5	0.3	-230.5	277.4	-50.9	115.9	35.0	
XL323-03	48.4	0.8	15.8		9.7	0.1	8.4	13.7	1.6	0.1	0.0	806.0	9.9	309.5	1.8	2.0	207.9	245.0	52.3	3.0	0.4	-235.7	219.6	-41.3	86.3	55.0	
XL323-04	45.0	1.6	18.8		11.0	0.2	6.8	12.3	1.9	0.2	0.0	749.0	19.5	368.2	2.0	2.3	169.0	219.3	62.6	3.2	0.3	-136.3	190.5	-22.8	61.8	61.0	
XL323-05	48.9	0.6	19.9		6.8	0.1	5.9	13.9	2.1	0.3	0.1	813.3	8.0	390.7	1.4	1.6	146.7	247.7	67.4	5.7	0.7	-177.8	156.0	-31.2	61.8	69.4	
XL323-06	46.3	1.2	15.6		11.0	0.2	8.0	14.0	1.6	0.2	0.0	770.3	14.6	305.0	1.9	2.1	197.8	249.1	52.0	3.4	0.4	-248.5	214.3	-47.9	90.4	57.5	
XL323-08	36.4	4.9	12.4		27.4	0.2	5.9	9.7	1.5	0.2	0.0	604.9	61.3	243.4	2.4	2.7	145.2	172.1	46.8	3.4	0.3	-150.9	208.9	-32.7	84.8	47.9	
XL323-10	38.8	3.9	13.6		22.6	0.2	5.7	11.3	2.1	0.3	0.0	645.7	48.6	266.8	2.1	2.4	142.2	201.3	67.1	5.3	0.1	-208.3	192.9	-51.0	90.2	60.8	
XL323-11	40.0	4.1	11.3		23.4	0.2	6.7	11.0	1.6	0.4	0.0	665.2	51.6	221.5	2.6	3.0	165.0	196.1	52.0	9.3	0.1	-232.1	219.2	-53.1	102.5	50.6	
XL323-12	46.6	0.9	16.3		10.2	0.2	9.4	13.5	1.6	0.1	0.0	775.0	11.5	319.5	1.9	2.1	232.3	240.5	52.0	2.1	0.1	-215.6	245.7	-38.1	86.2	51.9	
XL323-13	46.5	1.5	20.5		9.7	0.1	5.1	13.1	2.1	0.2	0.0	773.3	18.9	402.7	1.5	1.7	126.8	232.9	66.2	4.2	0.4	-133.5	147.2	-22.8	52.9	69.9	
XL323-14	43.6	2.5	18.1		14.1	0.2	5.6	11.8	2.0	0.1	0.0	725.7	31.5	355.2	1.9	2.1	138.2	210.9	64.9	2.8	0.6	-134.3	171.6	-24.1	60.6	63.5	
XL323-15	47.7	1.6	17.5		9.9	0.1	5.9	13.0	2.5	0.2	0.0	793.3	20.4	343.5	1.6	1.8	146.2	231.1	80.3	4.5	0.4	-203.5	168.2	-39.2	71.6	67.6	
XL323-17	42.3	3.1	13.3		17.6	0.2	6.2	12.2	2.8	0.5	0.1	703.3	38.8	261.1	2.0	2.3	153.1	218.1	90.7	10.4	0.7	-276.2	193.9	-67.9	100.4	67.4	
XL323-18	39.2	4.2	10.5		24.1	0.2	7.3	12.1	1.3	0.1	0.1	652.0	52.9	205.6	2.5	2.8	181.9	215.0	41.3	2.5	0.7	-268.4	237.3	-62.7	116.1	46.6	
XL323-19	36.9	2.5	15.8		15.6	0.2	5.5	18.1	0.1	0.0	0.0	614.2	31.3	309.1	1.9	2.1	136.5	322.9	4.5	0.2	0.6	-341.5	169.6	-87.3	116.0	71.4	
XL323-20	39.6	4.1	11.6		22.3	0.2	6.1	11.8	1.6	0.2	0.0	658.3	50.8	226.8	2.4	2.7	150.4	210.9	50.7	5.1	0.4	-250.9	203.6	-59.7	104.8	54.8	
XL323-21	31.5	6.1	9.1		32.2	0.2	6.2	11.6	0.7	0.0	0.0	524.0	76.6	178.9	2.8	3.1	153.6	207.4	22.9	0.8	0.3	-259.6	232.9	-74.6	131.1	43.5	
XL323-22	41.3	3.5	11.4		20.7	0.2	7.1	12.8	1.5	0.2	0.0	686.5	44.1	222.8	2.4	2.7	177.2	227.4	47.1	3.6	0.3	-282.6	223.6	-64.5	112.4	52.1	
XL323-23	41.4	3.6	12.8		20.2	0.2	6.4	12.2	1.7	0.2	0.0	688.5	44.8	250.7	2.3	2.5	158.1	216.8	53.9	4.0	0.4	-240.9	205.1	-53.7	98.0	55.7	
XL323-24	39.3	4.2	11.3		22.8	0.2	6.6	11.5	1.9	0.2	0.0	653.5	53.1	220.9	2.4	2.7	163.0	204.4	62.3	4.7	0.3	-254.8	218.5	-62.1	108.1	54.1	
XL323-25	38.8	4.6	12.1		23.9	0.2	6.1	11.7	1.6	0.1	0.0	645.2	58.1	237.9	2.5	2.8	151.4	208.6	51.6	2.5	0.4	-233.5	211.9	-55.4	101.8	53.6	
XL323-26	44.2	2.8	13.7		16.1	0.2	5.9	12.0	2.2	0.2	0.1	734.7	35.2	268.3	2.0	2.3	145.9	213.6	72.3	5.1	0.7	-236.3	183.1	-50.9	89.0	61.8	
XL323-27	42.4	3.7	12.7		19.3	0.2	6.4	11.9	1.9	0.2	0.0	706.1	45.9	249.3	2.4	2.7	157.6	211.5	59.7	4.2	0.4	-237.6	205.9	-51.5	96.1	55.4	
XL323-28	44.0	3.1	11.8		16.8	0.2	7.4	13.1	1.7	0.3	0.0	732.4	39.3	230.5	2.5	2.8	183.1	233.6	56.1	5.5	0.6	-298.4	224.9	-65.0	110.9	54.1	
XL323-29	43.6	3.2	12.0		16.8	0.2	7.1	12.9	2.7	0.2	0.0	725.1	39.4	234.6	2.5	2.8	177.2	230.2	86.5	4.7							

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XL323-35	42.0	5.7	12.5	17.7	0.2	7.3	11.3	1.9	0.2	0.0	699.1	71.1	244.4	2.6	3.0	181.4	200.8	61.6	3.4	0.4	-222.2	255.1	-46.1	99.6	46.6
XL323-36	43.3	5.1	13.8	15.4	0.2	6.6	12.1	1.9	0.1	0.0	720.3	64.1	271.1	2.4	2.7	164.8	215.4	62.3	2.8	0.3	-224.8	231.2	-46.1	93.4	52.7
XL323-37	40.9	6.3	12.1	19.7	0.2	7.4	10.7	1.6	0.1	0.0	681.1	78.2	236.4	2.8	3.1	182.6	191.3	51.6	2.3	0.3	-200.3	263.6	-39.6	98.4	41.1
XL323-38	41.8	4.5	15.9	17.4	0.2	8.7	6.6	2.0	0.2	0.0	695.8	56.7	310.9	2.5	2.8	214.9	117.7	65.5	3.2	0.4	6.8	274.1	19.0	48.2	32.8
XL323-39	46.8	3.4	17.2	10.5	0.2	4.7	11.3	3.4	0.3	0.0	778.2	42.1	337.4	1.9	2.1	117.6	201.1	108.7	6.4	0.3	-180.0	161.5	-37.6	66.6	71.0
XL323-40	44.0	3.0	13.6	16.0	0.2	6.1	12.2	2.3	0.2	0.0	732.1	37.9	266.4	2.1	2.4	151.6	218.3	73.9	3.6	0.6	-247.6	191.7	-54.1	92.7	61.4
XL323-41	42.8	3.3	13.6	18.0	0.2	6.7	12.5	1.8	0.2	0.0	712.9	41.8	266.6	2.4	2.7	165.3	222.0	56.5	3.2	0.6	-237.1	209.4	-50.3	94.4	55.9
XL323-43	44.8	2.9	16.3	14.5	0.2	5.9	9.9	2.6	0.3	0.1	745.0	36.0	319.7	2.0	2.3	147.1	176.4	82.9	7.0	0.7	-122.9	185.2	-20.3	60.9	59.3
XL323-45	49.4	1.2	20.8	7.6	0.1	4.1	12.8	2.9	0.2	0.0	821.4	15.0	407.6	1.4	1.6	102.5	228.6	92.6	5.1	0.4	-147.3	118.9	-26.5	49.7	76.8
XL323-46	41.6	3.8	12.5	19.9	0.2	6.9	11.3	2.3	0.1	0.1	691.8	46.9	245.2	2.8	3.1	170.0	202.0	73.9	2.5	0.7	-235.3	219.7	-53.3	98.2	55.1
XL323-47	46.4	2.1	20.1	11.8	0.1	4.0	10.5	3.0	0.3	0.1	771.7	25.9	395.1	1.5	1.7	99.5	186.9	96.5	6.4	1.1	-81.5	126.9	-12.4	39.4	73.0
XL323-48	46.8	1.8	15.7	11.5	0.2	6.7	13.9	1.9	0.3	0.0	778.0	22.5	308.0	2.0	2.3	166.5	248.4	60.7	6.2	0.6	-255.6	191.0	-51.1	88.2	62.9
XL323-49	43.5	2.4	18.3	14.8	0.2	5.3	11.3	2.2	0.2	0.1	724.1	30.3	358.4	1.9	2.1	131.8	202.2	69.4	4.5	0.8	-119.9	163.9	-21.1	56.3	64.8
XL323-51	45.9	2.7	16.0	14.1	0.2	5.2	12.0	2.4	0.3	0.1	764.4	34.2	314.0	2.1	2.4	128.3	214.3	77.4	6.6	0.7	-198.7	164.6	-39.7	73.6	66.1
XL323-52	51.7	0.8	18.3	5.9	0.1	5.3	13.0	3.0	0.5	0.0	859.5	9.5	359.4	1.5	1.7	131.3	231.6	97.5	10.6	0.3	-212.0	142.3	-39.4	65.9	73.5
XL323-53	45.1	2.9	15.9	15.0	0.2	5.2	12.0	2.3	0.4	0.1	750.9	36.7	311.9	2.0	2.3	129.3	213.3	75.2	7.4	0.8	-197.3	168.0	-40.2	74.6	65.7
XL323-54	45.4	2.7	17.0	13.1	0.2	4.8	12.2	2.4	0.4	0.1	754.7	33.2	333.3	1.9	2.1	117.9	216.8	78.7	9.1	1.0	-188.3	152.9	-38.9	69.0	69.9
XL323-55	43.4	3.5	14.9	17.4	0.2	5.8	11.3	2.1	0.2	0.0	722.4	43.9	292.7	2.4	2.7	142.9	201.1	67.1	4.0	0.6	-180.8	189.2	-35.6	77.0	58.6
XL323-56	49.5	0.8	18.2	7.6	0.1	6.4	12.8	2.4	0.3	0.1	823.9	9.5	356.8	1.6	1.8	158.6	228.1	78.4	6.2	0.7	-183.9	169.7	-31.8	65.9	65.9
XL323-57	46.4	1.8	15.5	12.9	0.2	7.2	12.1	2.1	0.2	0.1	771.3	22.7	304.2	2.3	2.5	178.9	215.4	66.8	4.7	1.1	-198.0	203.8	-36.2	79.0	57.2
XL323-58	43.5	3.2	14.1	17.7	0.2	5.7	11.2	2.2	0.5	0.1	723.2	40.6	276.0	2.4	2.7	140.9	198.8	71.0	10.4	1.0	-203.0	183.9	-43.0	82.1	60.9

Xialan and Wengong complex (Zhu W.g et al., 2010 Lithos 119 313-329)

Xialan gabbro

CQ0701	48.4	2.1	13.7	13.2	0.2	6.2	10.6	3.1	0.9	0.2	806.1	25.8	269.3	2.5	2.8	154.8	189.0	101.0	18.3	2.7	-228.0	183.1	-46.4	82.1	64.3
CQ0702	50.9	2.3	14.4	15.9	0.3	2.8	7.5	4.0	0.8	0.9	846.6	29.2	282.7	4.1	4.7	68.2	134.1	127.8	17.8	13.2	-131.1	101.5	-21.0	48.3	72.7
CQ0703	47.6	2.1	14.0	14.1	0.2	6.2	10.7	2.7	0.9	0.4	792.3	26.3	275.4	2.9	3.2	154.1	190.1	87.8	18.3	5.4	-210.8	183.3	-41.6	79.7	62.3
CQ0704	46.0	1.2	16.5	12.3	0.2	8.2	11.4	2.1	0.3	0.1	764.9	14.6	324.0	2.3	2.5	204.0	203.5	68.4	5.7	1.8	-157.0	220.9	-26.1	71.3	54.4
CQ0705	43.7	3.9	13.1	17.3	0.2	6.6	11.8	2.5	0.3	0.1	726.7	48.6	257.4	2.5	2.8	164.3	209.5	79.1	5.7	1.8	-246.5	215.3	-54.2	96.2	58.0
CQ0706	43.9	3.4	13.9	17.8	0.2	6.7	10.9	2.7	0.3	0.1	730.4	42.8	273.4	2.6	3.0	166.7	195.1	87.1	5.5	1.7	-209.4	212.2	-44.6	86.4	58.2
CQ0707	43.2	5.3	14.3	17.5	0.2	6.5	10.4	2.5	0.3	0.1	718.1	66.6	279.9	2.5	2.8	160.3	185.6	80.0	5.5	1.5	-176.9	229.4	-35.3	82.1	53.2
CQ0708	43.0	5.1	14.8	16.5	0.2	6.8	10.7	2.5	0.2	0.1	714.9	63.6	289.3	2.3	2.5	168.7	189.9	79.1	5.1	1.4	-174.6	234.6	-34.9	81.7	53.2
CQ0709	52.7	1.5	14.8	9.9	0.2	5.2	8.8	3.1	0.7	0.2	877.2	18.5	290.3	1.9	2.1	130.0	156.0	101.3	14.6	2.5	-137.7	150.4	-16.7	55.5	61.1
LGXL0701	46.9	1.2	16.4	10.3	0.1	9.1	11.3	2.3	0.4	0.1	780.3	15.3	321.5	1.8	2.0	226.3	201.1	72.6	7.4	1.7	-160.8	243.3	-26.1	74.5	51.6
LGXL0702	46.5	1.2	16.5	10.1	0.1	8.6	11.5	2.4	0.6	0.1	774.2	15.5	322.9	1.8	2.0	213.6	204.5	78.1	13.0	1.7	-177.2	230.9	-32.5	76.1	56.4
LGXL0703	49.3	1.4	16.1	10.4	0.1	7.4	10.5	2.5	0.7	0.1	820.4	17.5	315.8	1.8	2.0	184.4	187.9	81.0	15.7	1.5	-156.8	203.6	-24.3	67.2	57.1
LGXL0704	46.8	1.1	16.9	9.8	0.1	9.2	12.1	2.0	0.2	0.1	778.2	13.5	332.1	1.6	1.8	229.3	214.9	65.5	4.9	1.3	-168.0	244.4	-27.5	75.6	51.9
LGXL0705	49.4	1.7	15.3	11.3	0.2	6.7	11.3	2.8	0.7	0.2	821.4	21.5	300.5	2.0	2.3	165.3	201.5	91.3	13.8	2.3	-207.6	188.8	-38.6	76.1	62.5
LGXL0706	46.9	1.4	17.2	10.5	0.1	7.9	11.4	2.7	0.3	0.1	779.7	17.6	336.4	1.8	2.0	194.8	202.9	86.8	6.6	1.8	-162.8	214.2	-29.3	70.2	59.1
LGXL0707	46.6	1.2	16.6	11.0	0.2	8.2	10.9	2.7	0.4	0.1	775.0	15.3	325.4	2.0	2.3	204.2	194.7	86.2	9.3	1.8	-159.5	221.5	-28.5	71.3	57.2
LGXL0708	48.6	1.4	16.0	10.1	0.2	7.1	11.4	2.8	0.6	0.2	809.0	17.0	314.6	1.9	2.1	175.9	202.6	90.7	13.6	2.1	-194.8	194.8	-36.3	73.9	62.4
LGXL0709	46.8	1.5	16.3	10.8	0.2	8.0	11.3	2.6	0.5	0.1	779.5	19.1	319.7	1.9	2.1	197.3	201.1	83.9	11.5	1.8	-177.9	218.3	-32.9	74.6	58.3
LGXL0710	48.9	1.1	14.4	9.2	0.1	9.3	12.6	2.2	0.5	0.1	813.1	13.8	281.9	1.8	2.0	231.0	225.4	69.4	9.6	1.1	-247.8	246.5	-45.1	92.6	52.4
LGXL0711	47.5	1.4	15.7	10.5	0.1	7.5	11.0	2.7	0.9	0.1	790.3	18.0	308.6	1.8	2.0	186.8	195.3	88.4	18.7	1.8	-189.1	206.6	-36.3	75.4	60.8
LGXL-01	47.0	1.2	15.6	9.5	0.1	9.0	11.5	2.4	0.7	0.1	781.7	15.3	306.8	1.6	1.8	223.8	205.4	76.5	14.9	1.5	-195.4	240.7	-36.1	81.5	54.6

Wengong granite

WG0701	76.8	0.1	12.2	1.3	0.0	0.1	0.4	3.8	4.7	0.0	1278	1.3	238.7	0.1	0.1	2.5	7.7	121.7	98.7	0.3	3.0	3.9	35.0	0.0	65.0
WG0702	77.5	0.1	12.4	1.5	0.0	0.1	0.3	4.0	4.5	0.0	1290	1.3	242.3	0.0	0.0	2.5	4.6	129.4	94.9	0.3	8.7	3.7	36.2	-1.2	65.0
WG0703	75.6	0.1	12.0	1.3	0.0	0.1	0.4	3.9	4.4	0.0	1257	1.1	234.4	0.1	0.1	3.0	7.3	124.2	93.4	0.3	2.1	4.2	34.7	0.2	65.1
WG0704	73.6	0.4	12.8	2.7	0.1	0.2	0.7	3.8	4.2	0.1	1225	5.3	250.1	0.8	0.8	5.5	12.8	122.9	88.1	0.8	13.4	11.5	35.9	-0.9	65.1
WG0705	74.3	0.4	12.5	3.1	0.1	0.3	0.3	3.9	4.3	0.1	1236	5.3	245.4	0.8	0.8	6.7	5.0	124.2	91.7	0.8	19.5	12.7	37.7	-2.0	64.3
WG0706	73.3	0.4	12.6	3.4	0.1	0.2	0.8	4.5	4.2	0.1	1220	5.1	246.4	1.1	1.3	6.0	15.0	146.5	88.7	0.8	-18.8	12.2	24.2	5.7	70.1
WG0707	73.5	0.4	12.6	4.1	0.1	0.3	1.2	3.9	4.1	0.1	1223	5.4	247.2	1.6	1.8	6.5	20.7	126.5	87.0	0.8	-7.8	13.5	29.9	3.7	66.4
WG0708	73.1	0.4	12.3	3.2	0.1	0.2	0.9	3.8	4.2	0.1	1217	5.1	242.1	1.0	1.1	5.7	15.9	123.6	89.0	0.8	-2.2	11.8	31.8	2.3	65.9
WG0709	73.1	0.4	12.4	3.5	0.1	0.4	0.7	4.1	3.8	0.1	1217	5.5	243.8	0.8	0.8	10.2	12.7	132.0	80.7	0.8	5.8	16.4	33.7	1.4	64.9
WG0710	76.5	0.2	12.1	1.8	0.0	0.1	0.1	3.8	4.5	0.0	1273	2.0	236.6	0.1	0.1	2.7	1.2	123.9	94.5	0.3	15.7	4.9	38.6	-2.4	63.8
WG0711	75.5	0.2	12.3	1.9	0.0	0.2	0.6	4.0	4.3	0.0	1256	2.1	241.5	0.4	0.4	4.2	10.7	129.4	90.2	0.3	0.4	6.7	33.3	0.9	65.8
WG0712	75.2	0.2	12.3	2.0	0.0	0.2	0.5	3.9	4.5	0.0	1251	2.1	241.1	0.4	0.4	3.7	9.6	127.1	96.2	0.3	-1.5	6.2	32.2	1.3	66.6

Chapter 1

20GN20	45.4	2.2	17.1	8.4	3.3	0.2	8.3	10.7	2.0	2.0	0.6	756.0	27.7	334.4	157.8	2.7	206.7	190.4	65.8	41.8	7.7	-154.1	392.2	-27.4	89.0	38.3
20GN22	48.6	1.7	17.5	9.1	2.2	0.1	7.5	8.4	2.0	1.4	0.7	808.5	21.4	343.3	153.8	1.8	185.4	149.8	63.6	30.6	9.3	-50.4	360.5	2.8	64.8	32.4
20GN27	54.2	2.0	14.1	6.0	3.9	0.2	5.2	7.7	3.4	1.3	0.4	902.1	25.4	275.8	132.7	2.7	128.8	136.9	108.7	27.8	6.1	-134.7	286.9	-14.5	71.1	43.4
20GN28	53.0	2.5	13.4	5.6	4.5	0.2	4.5	9.5	2.6	1.7	0.5	881.3	31.0	263.4	134.6	3.1	111.2	169.6	82.3	35.9	6.8	-193.9	276.8	-28.7	82.9	45.8
20GH76	50.4	1.4	16.1	6.4	3.4	0.2	6.9	11.0	1.9	1.2	0.2	837.9	17.5	316.6	132.1	2.5	170.0	195.8	61.0	25.7	2.5	-161.7	319.6	-21.6	80.7	40.9
20GN77	50.4	1.5	16.5	6.2	3.5	0.2	6.7	10.7	1.8	0.5	0.2	838.7	19.1	323.1	129.7	2.5	167.2	189.9	58.4	9.8	2.8	-124.9	316.1	-10.5	73.7	36.8
20GN36	53.1	1.6	15.1	8.1	1.6	0.3	5.8	7.5	2.8	1.9	0.2	883.8	20.3	297.0	132.6	4.2	143.7	134.1	90.7	39.7	2.1	-101.6	296.5	-7.5	65.8	41.7
20GN37	53.5	1.7	15.0	7.7	2.1	0.3	5.7	7.5	2.8	1.9	0.2	889.7	20.7	294.0	133.1	4.2	140.9	133.2	91.3	39.5	2.1	-103.2	294.7	-7.5	65.8	41.6
20GN39	53.5	1.6	14.7	7.9	1.7	0.3	6.0	8.1	2.8	1.5	0.2	890.7	19.8	288.0	130.9	3.7	148.4	143.7	90.4	32.7	2.1	-122.5	299.0	-11.2	70.3	40.9
20GN40	53.4	1.6	15.3	7.9	1.5	0.3	5.9	7.7	2.7	1.8	0.2	888.5	19.9	299.3	128.5	4.1	145.2	137.8	88.1	37.2	2.1	-101.6	293.5	-6.8	65.3	41.5
20GN41	52.8	1.6	15.2	7.8	2.1	0.3	6.0	7.4	2.8	1.9	0.2	878.8	20.2	297.8	134.1	4.1	147.9	132.1	88.7	40.3	2.1	-95.6	302.1	-6.2	65.5	40.6
20GN42	50.0	1.4	16.7	8.0	1.6	0.2	7.7	8.6	2.7	1.0	0.2	832.4	17.4	327.6	132.0	3.2	189.8	152.5	87.4	20.2	2.1	-85.0	339.2	-5.3	68.3	36.9
20GN43	50.8	1.6	15.7	7.9	1.9	0.3	6.6	9.4	2.8	1.0	0.2	844.9	19.8	308.0	133.4	3.8	163.5	168.3	90.4	22.1	2.3	-141.1	316.7	-18.5	76.5	42.0
20GN44	51.4	1.3	15.7	8.2	1.6	0.2	7.9	8.1	2.6	1.0	0.2	855.7	16.5	307.4	133.2	3.0	196.3	144.1	83.6	20.8	2.1	-85.2	346.0	-2.5	69.5	33.0
20GN45	50.1	1.6	15.5	8.7	1.9	0.3	6.7	9.8	2.7	0.8	0.2	832.9	20.0	303.1	144.7	3.5	165.0	174.9	86.5	16.6	2.4	-149.8	329.8	-20.1	80.5	39.6
Group 2 (high-mg andesite)																										
20GN74	59.1	0.8	13.4	4.4	2.9	0.1	5.2	6.9	2.6	2.5	0.4	982.9	10.3	261.9	97.3	2.0	128.5	123.0	83.3	52.0	4.9	-119.5	236.1	-5.0	60.2	44.8
20GN70	59.4	0.8	13.2	5.1	2.0	0.1	5.4	6.4	3.0	2.6	0.4	988.5	10.0	258.1	95.8	1.8	135.0	113.6	97.1	55.0	5.2	-121.2	240.8	-6.8	60.8	46.0
20GN25	62.7	1.2	13.5	4.5	2.3	0.1	3.7	4.9	2.6	2.3	0.3	1044	14.6	264.0	91.9	1.6	92.3	88.1	84.2	48.0	3.7	-44.4	198.8	18.3	39.7	42.0
20GN26	61.7	1.2	13.5	5.0	2.7	0.1	3.8	5.2	2.1	2.5	0.3	1027	14.4	263.8	103.6	1.4	94.0	92.7	68.1	53.7	3.5	-43.4	212.0	18.9	41.8	39.4
20GN80	63.1	1.0	13.1	4.1	3.6	0.1	3.6	4.8	2.7	2.6	0.4	1050	12.6	257.0	102.7	1.7	88.1	84.7	88.4	54.4	5.5	-55.2	203.5	14.9	42.3	42.8
20GN82	63.1	1.3	13.0	3.4	4.2	0.2	3.3	4.4	2.5	1.1	0.4	1051	16.6	255.2	100.1	2.1	80.9	78.6	80.3	23.1	4.9	-5.6	197.7	33.4	32.4	34.1
20GN65	65.1	1.2	13.1	3.8	2.9	0.1	2.6	3.7	3.7	1.6	0.3	1083	14.4	257.7	89.5	1.7	64.5	66.7	118.4	32.9	4.5	-27.0	168.4	23.2	31.5	45.3
Group 3 (rhyolite)																										
20GN83	76.6	0.2	11.7	0.4	1.9	0.0	0.2	0.3	1.7	5.8	0.0	1274	2.1	229.9	28.3	0.1	4.2	5.0	54.9	122.1	0.3	43.0	34.6	51.4	-3.4	52.0
20GN84	74.0	0.2	13.1	0.3	1.8	0.0	0.2	0.5	1.6	7.0	0.0	1232	2.1	257.6	25.9	0.3	4.0	8.0	50.0	148.4	0.3	43.1	32.0	44.6	-3.8	59.2
20GN85	76.5	0.1	11.6	0.3	1.7	0.0	0.2	0.5	1.8	5.7	0.0	1273	1.0	226.8	24.8	0.4	5.2	9.5	58.7	121.7	0.3	27.5	31.0	47.0	-0.8	53.7
20GN86	76.9	0.2	11.9	0.6	1.2	0.0	0.2	0.1	1.7	6.1	0.0	1280	2.0	234.2	23.3	0.1	4.5	1.8	56.1	129.7	0.1	44.8	29.8	50.6	-4.5	53.9
20GN90	77.1	0.2	11.7	0.2	0.9	0.0	0.3	0.4	2.4	6.1	0.0	1283	2.8	230.3	13.9	0.4	7.7	6.4	77.4	130.1	0.3	9.9	24.3	38.9	1.7	59.3
20GN91	72.4	0.4	14.1	0.3	1.7	0.0	0.4	0.1	1.9	7.9	0.1	1205	4.9	275.6	24.1	0.4	10.9	2.5	60.3	167.9	0.7	42.3	39.9	37.9	-2.4	64.5
20GN92	73.2	0.3	13.2	0.2	1.6	0.0	0.2	0.1	2.5	7.9	0.0	1219	4.3	259.3	22.0	0.4	6.0	2.5	79.7	167.3	0.6	7.3	32.3	28.3	3.4	68.3
20GN93	76.9	0.3	12.0	0.1	1.1	0.0	0.4	0.3	1.9	6.1	0.0	1279	3.1	236.0	14.7	0.6	9.9	4.5	60.7	130.4	0.3	36.0	27.7	47.4	-3.0	55.6
20GN95	76.6	0.2	11.8	0.2	0.7	0.1	0.3	0.4	3.1	6.0	0.0	1274	2.4	230.5	11.7	0.7	6.9	7.0	101.0	127.2	0.4	-11.6	21.0	30.4	5.5	64.1
20GN34	76.9	0.4	11.1	0.8	2.1	0.0	0.4	1.2	1.9	3.9	0.1	1280	5.3	217.5	36.2	0.4	10.2	21.2	61.6	81.7	1.0	31.7	51.7	53.6	1.6	44.8
20GN35	77.0	0.4	10.5	0.8	2.2	0.0	0.4	1.7	1.8	3.6	0.1	1281	5.4	205.2	38.4	0.6	8.9	30.5	58.7	76.2	1.3	9.2	52.7	49.7	6.3	43.9
20GN08	72.1	0.3	13.5	1.8	1.1	0.0	0.5	0.7	2.3	6.3	0.1	1200	4.0	264.4	38.0	0.4	11.4	11.8	75.5	134.2	1.3	31.2	53.4	37.8	1.9	60.3
Changpu basalts (Cen T. et al., 2016 Lithos 256 311-330)																										
08GN05-1	54.5	2.0	16.0	10.1		0.2	5.0	9.5	1.8	0.8	0.3	907.0	24.5	313.5	139.7	2.3	123.1	170.1	56.5	17.8	3.7	-101.1	287.3	-0.5	64.0	36.5
08GN05-2	58.1	1.9	16.8	9.9		0.1	4.1	4.1	3.7	1.3	0.2	967.5	23.2	329.1	137.2	1.7	100.5	72.6	118.4	26.5	2.7	39.0	260.8	29.3	31.6	39.1
08GN05-3	54.0	1.9	15.9	9.7		0.2	4.7	10.3	1.6	1.5	0.3	898.8	23.8	312.3	134.7	2.3	117.4	183.5	51.0	31.2	3.5	-136.9	275.8	-10.8	69.2	41.6
08GN05-4	52.3	2.2	17.1	11.2		0.2	5.1	7.6	3.1	1.1	0.3	869.7	26.9	335.4	156.1	2.4	127.0	134.6	99.7	22.7	3.8	-56.3	310.0	1.6	58.1	40.3
08GN05-5	54.8	1.9	16.1	10.1		0.2	4.3	8.9	2.0	1.5	0.3	911.1	24.0	315.8	140.9	2.1	106.9	157.8	64.2	31.8	3.9	-95.9	271.9	-1.7	60.5	41.1
08GN14-1	54.4	2.8	14.6	12.3		0.2	3.9	7.2	3.2	1.2	0.3	906.0	34.9	285.4	170.5	2.7	96.3	128.7	102.3	25.3	4.2	-99.6	301.7	-4.7	66.1	38.7
08GN14-2	57.1	2.6	13.5	10.3		0.1	2.7	12.2	0.6	0.4	0.4	950.7	32.9	265.4	142.9	1.8	68.0	217.2	18.1	8.9	5.8	-196.0	243.8	-14.7	78.4	36.2
08GN14-3	54.3	2.8	14.0	12.7		0.2	3.9	7.6	3.0	1.1	0.4	903.6	35.3	275.4	176.4	2.7	97.0	134.8	97.8	23.4	5.1	-115.3	308.7	-7.7	70.4	37.3
08GN14-4	55.2	2.9	13.9	12.7		0.2	3.9	6.1	2.8	2.1	0.3	919.1	35.9	273.0	176.2	2.7	95.5	109.0	90.4	43.5	4.4	-78.7	307.7	1.3	63.0	35.7
08GN14-6	50.9	2.4	15.5	13.1		0.2	4.6	11.8	1.0	0.2	0.4	846.2	29.8	304.4	181.8	2.3	114.9	209.7	32.3	4.9	5.8	-152.1	326.5	-12.8	81.6	31.1
08GN32-7	51.3	1.9	14.7	11.7																						

Chapter 1

Jiangxi basalts and Ziyunshan granite (Wang K.x et al., 2015 JAsEaSc 111 919-935)

Basalt																										
20GN47	51.5	1.3	16.2	7.5	1.6	0.2	6.8	7.9	2.9	1.3	0.3	856.7	16.3	317.8	105.8	2.1	169.5	141.2	93.3	28.5	3.8	-86.4	291.6	-5.4	62.5	42.9
20GN16	55.5	1.6	14.5	5.2	3.5	0.2	3.2	7.4	2.6	2.7	0.3	924.3	19.5	284.0	74.0	2.4	79.7	131.6	85.2	56.7	3.8	-121.0	173.2	-11.4	53.5	57.9
20GN18	51.2	2.0	14.5	8.5	3.9	0.1	6.2	8.0	2.2	1.9	0.6	852.2	24.9	284.4	119.5	1.6	154.1	143.4	70.0	39.9	7.9	-112.2	298.5	-9.3	70.4	38.9
20GN2	47.0	1.8	15.4	6.8	4.2	0.2	7.5	9.7	2.3	1.1	0.2	782.5	22.0	301.1	96.8	2.5	187.1	173.1	75.5	22.5	3.1	-143.2	305.9	-21.6	79.3	42.3
20GN20	45.4	2.2	17.1	8.4	3.3	0.2	8.3	10.7	2.0	0.4	0.6	756.0	27.7	334.4	118.8	2.7	206.7	190.4	65.8	7.4	7.7	-119.7	353.2	-15.4	79.7	35.7
20GN22	48.6	1.7	17.5	9.1	2.2	0.1	7.5	8.4	2.0	1.4	0.7	808.5	21.4	343.3	127.8	1.8	185.4	149.8	63.6	30.6	9.3	-50.4	334.6	2.9	61.8	35.3
20GN27	54.2	2.0	14.1	6.0	3.9	0.2	5.2	7.7	3.4	1.3	0.4	902.1	25.4	275.8	85.8	2.7	128.8	136.9	108.7	27.8	6.1	-134.7	240.0	-14.9	65.7	49.2
20GN28	53.0	2.5	13.4	5.6	4.5	0.2	4.5	9.5	2.6	1.7	0.5	881.3	31.0	263.4	80.6	3.1	111.2	169.6	82.3	35.9	6.8	-193.9	222.8	-29.7	76.9	52.8
20GN76	50.4	1.4	16.1	6.4	3.4	0.2	6.9	11.0	1.9	1.2	0.2	837.9	17.5	316.6	91.6	2.5	170.0	195.8	61.0	25.7	2.5	-161.7	279.1	-22.1	76.3	45.8
20GN77	50.4	1.5	16.5	6.2	3.6	0.2	6.7	10.7	1.8	0.5	0.2	838.7	19.1	323.1	88.0	2.8	167.2	189.9	58.4	9.8	2.5	-124.9	274.4	-10.8	69.0	41.8
20GN36	53.1	1.6	15.1	8.1	1.6	0.2	5.8	7.5	2.8	1.9	0.3	883.7	20.3	297.0	114.2	2.1	143.7	134.1	90.7	39.7	4.2	-101.6	278.1	-7.6	63.7	43.9
20GN37	53.5	1.7	15.0	7.7	2.1	0.2	5.7	7.5	2.8	1.9	0.3	889.7	20.7	294.0	109.3	2.1	140.9	133.2	91.3	39.5	4.2	-103.2	270.9	-7.6	63.0	44.5
20GN39	53.5	1.6	14.7	7.9	1.7	0.2	6.0	8.1	2.8	1.5	0.3	890.7	19.8	288.0	112.0	2.1	148.4	143.7	90.4	32.7	3.7	-122.5	280.1	-11.3	68.1	43.1
20GN40	53.4	1.6	15.3	7.9	1.5	0.2	5.9	7.7	2.7	1.8	0.3	888.5	19.9	299.3	111.7	2.1	145.2	137.8	88.1	37.2	4.1	-101.6	276.7	-6.9	63.3	43.6
20GN41	52.8	1.6	15.2	7.8	2.1	0.2	6.0	7.4	2.8	1.9	0.3	878.8	20.2	297.8	110.3	2.1	147.9	132.1	88.7	40.3	4.1	-95.6	278.3	-6.2	62.7	43.5
20GN42	50.0	1.4	16.7	8.0	1.6	0.2	7.7	8.6	2.7	1.0	0.2	832.4	17.4	327.6	113.5	2.1	189.8	152.5	87.4	20.2	3.2	-85.0	320.7	-5.3	66.2	39.1
20GN43	50.8	1.6	15.7	7.9	1.9	0.2	6.6	9.4	2.8	1.0	0.3	844.9	19.8	308.0	112.1	2.3	163.5	168.3	90.4	22.1	3.8	-141.1	295.4	-18.7	74.1	44.6
20GN44	51.4	1.3	15.7	8.2	1.6	0.2	7.9	8.1	2.6	1.0	0.2	855.7	16.5	307.4	115.6	2.1	196.3	144.1	83.6	20.8	3.0	-85.2	328.4	-2.5	67.5	35.1
20GN45	50.1	1.6	15.5	8.7	1.9	0.2	6.7	9.8	2.7	0.8	0.3	832.9	20.0	303.1	123.0	2.4	165.0	174.9	86.5	16.6	3.5	-149.8	308.1	-20.4	78.2	42.2
20GN74	59.1	82.0	13.4	4.4	2.9	0.1	5.2	6.9	2.6	2.5	0.5	982.9	1026	261.9	62.6	2.0	128.5	123.0	83.3	52.0	7.0	-119.5	1217	-3.2	134.7	-31.5
20GN70	59.4	0.8	13.2	5.1	2.0	0.1	5.4	6.4	3.0	2.6	0.4	988.5	10.0	258.1	72.1	1.8	135.0	113.6	97.1	55.0	5.2	-121.2	217.1	-6.9	58.0	48.9
20GN25	62.7	1.2	13.5	4.5	2.3	0.1	3.7	4.9	2.6	2.3	0.3	1044	14.6	264.0	64.3	1.6	92.3	88.1	84.2	48.0	3.7	-44.4	171.3	18.6	36.0	45.4
20GN26	61.7	1.2	13.5	5.0	2.7	0.1	3.8	5.2	2.1	2.5	0.3	1027	14.4	263.8	71.2	1.4	94.0	92.7	68.1	53.7	3.5	-43.4	179.6	19.2	37.5	43.3
20GN80	63.1	1.0	13.1	4.1	3.6	0.1	3.6	4.8	2.7	2.6	0.4	1050	12.6	257.0	58.9	1.7	88.1	84.7	88.4	54.4	5.5	-55.2	159.6	15.3	36.5	48.2
20GN82	63.1	1.3	13.0	3.4	4.2	0.2	3.3	4.4	2.5	1.1	0.4	1051	16.7	255.2	49.6	2.1	80.9	78.6	80.3	23.1	4.9	-5.6	147.1	34.5	25.2	40.4
20GN65	65.1	1.2	13.1	3.8	2.9	0.1	2.6	3.7	3.7	1.6	0.3	1083	14.4	257.7	54.3	1.7	64.5	66.7	118.4	32.9	4.5	-27.0	133.2	23.7	26.6	49.7
20GN83	76.6	0.2	11.7	0.4	1.9	0.0	0.2	0.3	1.7	5.8	0.0	1274	2.1	229.9	5.0	0.1	4.2	5.0	54.9	122.1	0.3	43.0	11.3	52.1	-7.1	55.0
20GN84	74.0	0.2	13.1	0.3	1.8	0.0	0.2	0.5	1.6	7.0	0.0	1232	2.1	257.6	3.7	0.3	4.0	8.0	50.0	148.4	0.3	43.1	9.8	45.2	-7.3	62.1
20GN85	76.5	0.2	11.6	0.3	1.7	0.0	0.2	0.5	1.8	5.7	0.0	1273	2.3	226.8	4.5	0.4	5.2	9.5	58.7	121.7	0.3	27.5	12.0	47.6	-3.8	56.2
20GN86	76.9	0.2	11.9	0.6	1.2	0.0	0.2	0.1	1.7	6.1	0.0	1280	2.0	234.2	8.9	0.1	4.5	1.8	56.1	129.7	0.1	44.8	15.4	51.0	-6.7	55.7
20GN90	77.1	0.2	11.7	0.2	0.9	0.0	0.3	0.4	2.4	6.1	0.0	1283	2.8	230.3	2.7	0.4	7.7	6.4	77.4	130.1	0.3	9.9	13.2	39.2	0.0	60.8
20GN91	72.4	0.4	14.1	0.3	1.7	0.0	0.4	0.1	1.9	7.9	0.1	1205	4.9	275.6	3.9	0.4	10.9	2.5	60.3	167.9	0.7	42.3	19.6	38.3	-5.5	67.2
20GN92	73.2	0.3	13.2	0.2	1.6	0.0	0.2	0.1	2.5	7.9	0.0	1219	4.3	259.3	2.9	0.4	6.0	2.5	79.7	167.3	0.6	7.3	13.1	28.6	0.5	70.8
20GN93	76.9	0.3	12.0	0.1	1.1	0.0	0.4	0.3	1.9	6.1	0.0	1279	3.1	236.0	1.9	0.6	9.9	4.5	60.7	130.4	0.3	36.0	14.9	47.7	-5.0	57.3
20GN95	76.6	0.2	11.8	0.2	0.7	0.1	0.3	0.4	3.1	6.0	0.0	1274	2.4	230.5	3.4	0.7	6.9	7.0	101.0	127.2	0.4	-11.6	12.7	30.5	4.3	65.2
20GN34	76.9	0.4	11.1	0.8	2.1	0.0	0.4	1.2	1.9	3.9	0.1	1280	5.3	217.5	10.8	0.4	10.2	21.2	61.6	81.7	1.0	31.7	26.2	54.4	-2.4	48.0
20GN35	77.0	0.4	10.5	0.8	2.2	0.0	0.4	1.7	1.8	3.6	0.1	1281	5.4	205.2	11.3	0.6	8.9	30.5	58.7	76.2	1.3	9.2	25.7	50.5	2.2	47.3
20GN8	72.1	0.3	13.5	1.8	1.1	0.0	0.5	0.7	2.3	6.3	0.1	1200	4.0	264.4	24.7	0.4	11.4	11.8	75.5	134.2	1.3	31.2	40.1	38.1	-0.1	62.1
Granite																										
09XM-4	66.6	0.4	16.1	2.3	0.3	0.1	1.4	3.1	3.6	4.2	0.1	1108	5.4	316.4	33.3	0.8	34.0	55.5	115.5	89.2	1.5	0.8	72.7	21.9	10.9	67.2
09XM-5	68.5	0.3	15.4	2.2	0.2	0.1	1.3	2.8	3.5	4.1	0.1	1140	4.3	302.3	30.5	0.7	31.0	49.6	112.6	87.3	1.3	3.3	65.8	25.8	9.3	64.9
10-ZYS-5	69.9	0.1	16.1	0.5	1.0	0.1	0.4	1.8	4.3	5.1	0.1	1163	1.8	316.0	7.9	1.8	10.4	31.7	139.7	107.9	1.4	4.9	20.1	21.1	2.0	76.9
01XM01*	68.3	0.6	14.7	3.2	0.6	0.1	2.0	3.3	2.4	3.6	0.2	1136	7.1	288.3	45.4	1.0	50.6	58.1	76.8	76.0	2.3	19.3	103.1	36.2	11.9	51.9
01XM04*	68.3	0.6	14.7	3.0	0.8	0.1	2.0	3.4	2.2	3.9	0.2	1137	7.1	288.7	41.8	0.8	48.9	60.1	71.6	82.0	2.3	15.0	97.8	35.0	12.0	53.0
01XM12*	68.7	0.6	14.8	3.2	0.6	0.1	2.0	3.5	2.3	3.6	0.2	1144	7.3	291.1	44.5	0.8	49.6	61.5	73.9	77.3	2.3	16.9	101.4	35.8	12.1	52.1

Granite-syenite-gabbro complex (He Z.y et al., 2010 Lithos 119 621-641)

Lonyuanba Bt Granite	72.5	0.3	13.0	2.6	0.1	0.8	2.2	2.3	4.8	0.2	1206	4.1	254.6	36.2	0.7	20.1	38.3	74.5	101.1	2.1	2.3	60.4	35.2	8.8	56.0
2m granite	74.2	0.3	13.7	1.6	0.0	0.4	0.3	2.8	5.6	0.2	1235	3.3	269.5	22.8	0.4	9.4	5.3	89.1	119.1	2.1	50.6	35.4	44.3	-4.7	60.4
Pitou granite à amp granite	72.7	0.2	13.4	2.7	0.1	0.0	0.8	3.3	6.1	0.0	1210	2.6	263.6	37.6	0.8	0.2	13.9	105.5	129.5	0.4	0.8	40.5	27.2	6.0	66.8
	77.3	0.1	11.2	1.5	0.0	0.0	0.6	2.6	5.1	0.0	1287	1.8	219.9	21.1	0.4	0.0	10.2	84.5	108.5	0.3	6.5	22.8	41.0	2.2	56.8
Dafengnao syenite	64.1	0.4	15.2	5.0	0.1	0.1	1.9	4.9	6.1	0.0	1067	4.6	298.4	70.9	1.7	3.5	33.5	159.4	128.7	0.6	-56.8	79.0	-3.6	23.2	80.4
Tabel syenite	62.4	0.3	18.1	4.5	0.1	0.3	0.8	6.4	5.5	0.2	1038	3.6	354.5	63.5	1.4	6.9	14.3	207.8	117.2	2.3	0.9	74.1	2.1	10.8	87.1
Huanbu syenite	61.3	0.6	16.8	6.2	0.2	0.6	1.8	5.5	6.0	0.1	1020	7.4	329.9	88.6	3.1	15.6	32.5	175.9	127.4	1.7	-38.2	111.7	-4.9	24.0	80.9
Chebu gabbro DN01	48.7	1.7	16.4	11.7	0.2	8.2	8.4	2.7	1.0	0.3	810.8	20.7	321.1	163.8	2.1	202.5	148.9	87.8	20.4	4.5	-84.8	386.9	-6.1	74.8	31.3
DN02	47.8	1.6	17.0	11.7	0.1	7.6	8.8	2.7	0.7	0.3	794.6	20.5	332.5	164.4	2.0	189.1	156.2	88.4	14.9	3.9	-83.2	374.0	-6.7	73.2	33.6
Chenglong gabbro LN01	43.6	4.3	11.7	18.3	0.2	7.2	11.3	2.5	0.6	0.4	724.9	53.9	228.7	256.5	2.5	177.9	201.0	79.1	13.4	5.5	-265.6	488.3	-50.9	128.6	22.9
LN02	44.9	4.1	12.1	17.7	0.2	6.8	10.4	2.5	0.9	0.4	747.7	50.7	237.9	247.8	2.5	168.0	185.1	81.0	18.7	6.2	-231.9	466.5	-42.2	118.4	23.3

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2-2	44.5	2.6	13.9	6.6	4.5	0.2	7.8	11.5	3.3	0.9	0.5	740.7	32.2	272.5	148.9	2.4	194.5	205.2	106.8	18.9	7.0	-263.7	375.6	-56.4	111.9	44.5
4-1	43.4	2.8	13.5	4.4	7.8	0.2	8.5	10.5	3.9	1.2	0.6	722.7	34.5	264.4	157.5	3.0	209.7	187.8	124.9	25.9	8.3	-261.9	401.7	-59.1	115.1	44.1
7-4	45.9	3.0	14.6	4.1	6.5	0.1	6.6	10.5	3.7	1.3	0.7	764.4	37.5	286.0	138.8	1.6	162.5	187.8	119.7	28.0	10.0	-237.3	338.9	-51.3	100.5	50.8
7-5	47.9	2.5	15.3	6.5	3.9	0.1	6.2	8.2	3.9	2.2	0.6	797.3	31.0	299.1	138.8	1.8	153.6	145.9	124.6	46.3	8.3	-163.4	323.4	-33.3	82.6	50.7
XPA-1	49.6	1.8	16.2		10.8	0.2	6.6	6.6	3.8	2.2	0.8	826.1	22.4	316.8	135.5	2.7	164.3	118.2	122.9	45.9	10.7	-88.5	322.2	-12.9	66.7	46.2
PA-01	44.8	2.7	14.8		12.7	0.2	8.2	10.2	3.2	0.7	0.7	745.7	33.5	289.7	158.4	2.7	204.2	181.5	102.9	14.0	9.3	-190.3	396.2	-36.7	99.6	37.1
XTB-2	44.6	2.7	14.8		12.5	0.2	8.2	10.3	3.3	0.7	0.7	742.2	33.4	289.5	156.9	2.7	203.0	183.3	104.9	14.6	9.3	-196.6	393.3	-38.8	100.5	38.3
Yizhang																										
20YZH-2	51.4	1.9	17.2	6.1	3.3	0.1	4.0	10.1	2.2	0.3	0.3	855.6	23.2	338.2	126.5	2.0	98.0	180.6	71.0	5.9	3.9	-100.0	247.7	-5.1	59.5	45.6
20YZH-4	50.3	2.1	17.1	7.1	2.9	0.1	3.8	11.0	2.2	0.3	0.3	836.2	25.9	335.6	135.8	1.8	95.3	195.8	72.0	6.6	4.5	-134.5	257.0	-15.4	67.9	47.5
20YZH-5	51.2	2.0	17.2	7.0	2.8	0.1	4.2	9.7	2.0	0.4	0.3	851.9	25.2	338.2	132.6	1.6	104.5	172.6	63.6	7.6	4.2	-78.3	262.3	1.1	57.4	41.5
20YZH-7	52.2	2.0	17.1	6.7	3.3	0.1	4.2	8.9	2.5	0.6	0.3	869.4	24.8	334.8	134.9	1.8	103.7	159.1	79.1	13.2	4.2	-75.5	263.4	0.6	56.2	43.2
20YZH-8	52.0	2.0	17.3	6.7	3.0	0.1	3.9	9.4	2.5	0.5	0.3	864.5	24.5	339.9	130.9	1.8	97.8	167.6	80.7	11.0	4.1	-87.0	253.2	-2.9	57.1	45.9
20YZH-9	53.5	2.0	16.3	6.3	2.2	0.1	2.9	10.8	2.1	0.9	0.3	890.5	24.7	318.9	114.5	1.8	72.5	192.4	66.8	18.0	4.2	-150.7	211.6	-16.3	64.3	52.0
20YZH-10	51.9	2.0	16.4	6.6	2.3	0.2	3.4	11.2	1.8	0.7	0.3	864.0	25.5	322.3	121.1	2.3	85.4	199.9	58.7	14.9	4.4	-151.1	232.0	-16.9	67.8	49.0
20YZH-11	52.2	2.1	16.0	7.1	2.2	0.1	3.2	10.9	1.7	0.7	0.3	868.0	25.8	314.0	126.6	1.7	78.9	194.9	55.5	14.0	4.6	-145.3	231.3	-14.2	67.1	47.2
20YZH-13	49.9	2.0	17.0	7.4	2.6	0.1	3.2	11.3	1.8	0.6	0.3	829.9	25.5	334.1	134.9	1.7	80.4	202.0	58.7	11.7	4.5	-140.4	240.8	-16.5	67.6	48.9
Longnan-Xunwu																										
9717-1	45.6	3.1	13.9		9.6	0.5	3.4	8.6	0.3	2.7	0.8	758.9	38.4	272.7	120.2	7.1	83.1	154.1	8.7	57.5	11.0	-101.7	241.8	-7.0	67.0	40.0
N24-2	49.7	3.3	14.6		14.0	0.2	3.4	5.7	4.7	1.4	0.5	827.3	40.7	286.0	175.7	2.7	83.9	101.6	150.7	30.4	6.3	-98.3	300.3	-15.5	67.5	48.0
9711-2	48.7	2.4	14.7		11.3	0.2	6.0	9.2	2.6	0.5	0.5	811.0	30.0	288.0	141.6	2.3	147.6	163.5	85.2	10.0	7.0	-134.2	319.3	-16.2	78.8	37.4
N49	47.6	1.5	15.6		11.2	0.2	5.9	8.5	3.2	0.7	0.2	792.5	19.0	305.6	140.3	2.3	145.7	151.4	102.6	15.5	3.0	-115.3	305.0	-16.0	72.7	43.3
64009-1	47.5	1.5	15.5		11.0	0.2	5.5	7.7	4.0	0.3	0.2	789.8	19.3	304.4	137.4	2.1	137.0	136.6	128.4	6.8	3.0	-104.0	293.6	-15.2	69.1	46.1
64010-2	51.4	2.2	14.3		11.3	0.2	4.2	9.6	2.8	0.7	0.3	855.1	27.5	281.1	141.9	2.4	105.2	171.7	90.0	15.1	3.9	-167.5	274.6	-23.1	78.1	45.0
64011	50.7	2.2	14.3		13.6	0.3	4.9	7.8	3.1	0.8	0.3	843.1	27.3	280.1	170.8	3.7	121.1	138.2	100.0	17.4	3.9	-113.7	319.2	-10.8	73.6	37.2
N44-2	47.4	1.7	14.2		11.6	0.2	8.5	10.3	1.4	2.0	0.3	788.5	21.8	278.7	144.9	2.5	211.9	184.0	44.2	42.9	3.8	-176.4	378.6	-26.7	94.8	31.9
Yongding																										
YD-1-2	52.2	1.5	15.9		10.5	0.5	4.4	4.9	1.7	4.0	0.3	869.4	19.0	311.9	131.8	6.8	107.9	87.6	54.2	83.9	4.4	-1.3	258.7	16.5	41.7	41.8
YD-1	49.0	3.1	15.3	6.1	7.0	0.1	3.9	6.6	3.6	1.6	0.6	815.8	38.8	300.7	172.1	1.4	97.8	118.4	116.8	34.2	7.7	-87.1	308.7	-10.5	66.6	44.0
PK1-(1)	49.7	2.3	14.3		13.0	0.2	6.6	9.3	2.7	0.8	0.3	827.1	28.3	280.1	163.2	2.5	164.0	165.7	86.5	16.1	4.5	-153.8	355.5	-20.2	85.9	34.4
PKb3	49.4	2.5	14.0	8.4	4.9	0.3	7.4	8.3	2.6	0.2	0.3	822.6	31.7	275.2	178.0	3.7	182.6	147.6	85.2	3.2	4.2	-108.5	392.3	-6.8	82.6	24.2
PKb11	50.2	2.3	14.3	7.1	4.6	0.2	6.6	8.9	2.7	1.2	0.3	835.2	28.4	280.1	155.5	2.4	164.8	158.5	87.8	25.1	4.1	-149.8	348.7	-19.8	83.8	36.1
PKb11	50.2	2.3	14.1	6.9	5.4	0.2	6.5	9.1	2.7	1.1	0.3	835.1	28.3	276.2	162.9	2.3	162.0	161.4	85.8	24.2	4.2	-156.6	353.2	-21.1	85.8	35.2
Baoanxu alkali basalts (Zhao Z.h et al., 1998 Sc in China 41)																										
XTB-1	41.1	3.1	13.6	6.8	5.6	0.2	8.8	11.9	3.5	0.9	1.0	683.5	38.3	266.0	97.7	3.2	218.9	211.8	111.3	19.3	13.5	-288.3	354.8	-69.3	118.7	50.6
XTB-2	44.6	2.7	14.8		12.5	0.2	8.2	10.3	3.3	0.7	0.7	742.2	33.4	289.5	2.4	2.7	203.0	183.3	104.9	14.6	9.3	-196.6	238.8	-42.7	84.2	58.4
XTB-3	44.8	2.7	14.9		12.5	0.2	8.0	10.1	3.2	0.7	0.7	745.5	33.5	291.5	2.5	2.8	199.5	180.3	102.3	15.7	9.3	-187.1	235.6	-39.7	81.7	58.0
XPA-1	49.6	1.8	16.2		10.8	0.2	6.6	6.6	3.8	2.2	0.8	826.1	22.4	316.8	2.4	2.7	164.3	118.2	122.9	45.9	10.7	-88.5	189.1	-14.0	50.3	63.7
PA-01	44.8	2.7	14.8		12.7	0.2	8.2	10.2	3.2	0.7	0.7	745.7	33.5	289.7	2.4	2.7	204.2	181.5	102.9	14.0	9.3	-190.3	240.1	-40.3	83.0	57.3
PA-02	45.1	2.7	14.5		12.6	0.2	8.2	10.2	3.2	0.8	0.7	750.0	33.7	285.0	2.5	2.8	202.7	182.1	102.9	16.1	9.4	-198.2	238.9	-42.2	84.5	57.7
PA-03	45.0	2.7	14.6		12.7	0.2	8.2	10.2	3.3	0.8	0.7	748.4	33.5	286.6	2.5	2.8	202.2	181.7	104.9	17.0	9.3	-198.7	238.3	-42.9	84.4	58.5
Dexing complex (Wang G.q et al., 2015 OGR 67 109-126)																										
Tongchang granodiorite																										
DX-60	64.4	0.5	15.0		6.7	0.0	2.5	3.2	3.4	1.8	0.3	1071	6.0	295.0	83.9	0.4	63.0	56.2	108.4	37.6	3.7	36.7	152.9	37.7	16.2	46.1
DX-132	64.1	0.5	15.7		4.5	0.0	2.4	2.5	2.8	2.7	0.2	1067	5.6	307.8	56.2	0.3	59.3	43.9	90.4	56.3	3.2	73.4	121.2	47.2	3.9	48.9
DX-133	64.3	0.5	16.0		4.5	0.0	2.8	2.3	3.2	2.4	0.2	1070	6.3	313.3	56.4	0.3	68.5	41.5	101.6	50.3	3.2	78.2	131.1	47.1	4.4	48.5
DX-134	65.4	0.5	15.8		4.0	0.0	2.7	2.9	2.2	2.9	0.2	1088	6.1	310.5	50.3	0.4	67.7	51.0	72.0	60.5	3.0	76.0	124.2	50.1	3.8	46.1
Zhushahong granodiorite																										
DX-260	58.8	0.6	14.3		5.5	0.0	4.3	4.9	2.7	3.4	0.5	978.9	8.0	279.9	69.3	0.6	105.5	87.2	86.5	73.0	6.6	-54.0	182.7	8.1	40.1	51.8
DX-261	65.0	0.4	14.9		2.8	0.0	2.2	2.7	3.0	5.0	0.2	1082	5.1	292.9	35.2	0.3	53.6	48.7	95.5	106.8	2.5	-6.8	93.9	20.6	16.0	63.5
DX-265	61.7	0.5	14.9		4.4	0.0	2.6	4.4	3.4	3.1	0.2	1026	6.4	291.3	55.6	0.4	63.5	78.5	108.7	65.2	3.4	-39.6	125.5	12.3	27.9	59.8
DX-287	68.5	0.4	14.0		2.8	0.0	2.1	2.3	2.8	4.6	0.2	1139	5.1	274.8	35.4	0.4	51.6	40.1	89.7	97.7	2.4	7.2	92.2	30.1	12.7	57.2
DX-290	64.8	0.5	15.4		4.5	0.0	2.3	3.1	3.5	2.8	0.2	1078	5.9	301.3	56.5	0.6	57.3	55.5	114.2	58.8	2.8	17.3	119.7	29.5	15.0	55.6
DX-297	63.6	0.5	14.7		5.3	0.1	3.0	3.3	3.3	3.3	0.2	1059	6.5	287.8	66.8	0.7	74.2	58.5	104.9	70.5	3.1	-4.6	147.5	23.1	23.7	53.2
DX-301	67.2	0.4	15.6		3.2	0.0	1.6	2.7	3.7																	

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07SC45-1	46.8	3.0	15.7	11.9	0.2	7.4	8.5	3.6	2.4	0.7	778.8	36.9	308.0	149.0	2.1	184.4	150.9	116.2	50.1	9.7	-160.0	370.3	-32.8	87.2	45.6
07SC45-2	46.7	3.0	15.7	11.8	0.2	7.5	8.4	3.8	2.3	0.7	777.2	36.9	308.0	147.8	2.1	185.4	150.1	121.3	48.6	10.0	-162.3	370.1	-33.8	87.6	46.3
07SC45-3	47.0	2.9	15.8	11.8	0.2	7.6	8.6	3.1	2.4	0.7	782.2	36.5	309.9	147.8	2.3	187.6	152.8	100.4	50.5	9.9	-146.6	371.9	-27.7	85.1	42.5
07SC45-6	46.8	2.9	15.8	11.7	0.2	7.8	8.5	3.1	2.6	0.7	778.8	36.5	309.9	146.5	2.1	193.5	151.0	99.1	54.8	9.9	-146.0	376.6	-28.0	85.6	42.4
07SC45-7	47.0	2.9	15.9	11.7	0.2	7.3	8.3	3.7	2.2	0.7	782.2	36.5	311.9	146.5	2.1	180.9	147.8	120.7	46.7	10.4	-151.2	364.0	-30.7	84.6	46.1
07SC45-9	46.8	3.0	15.8	11.9	0.2	7.6	8.4	4.1	1.7	0.7	778.8	36.9	309.9	149.0	2.1	187.6	149.1	132.9	36.5	9.7	-157.7	373.6	-32.5	86.9	45.6
Dabaoshan granite porphyry (Huang W. et al., 2017 OGR 81 940-952)																									
5803-21	72.1	0.4	14.0	4.1	0.0	0.8	0.0	0.0	4.6	0.0	1200	4.9	273.8	51.5	0.3	18.9	0.7	0.1	96.6	0.3	175.7	75.2	92.5	-25.2	32.6
5803-82	68.4	0.4	14.6	5.7	0.0	0.8	0.1	0.0	4.8	0.1	1139	4.6	286.2	71.3	0.1	18.6	2.0	0.3	101.3	1.1	180.7	94.5	90.1	-23.4	33.3
5804-566	76.3	0.2	10.8	0.6	0.0	0.6	1.7	0.1	7.1	0.1	1270	2.8	211.3	7.5	0.4	13.6	29.4	3.5	150.7	1.1	-1.9	23.9	43.9	4.2	51.9
5810-566	65.1	0.4	15.3	1.8	0.0	1.2	2.8	0.2	9.1	0.2	1083	4.5	300.7	22.3	0.4	30.5	49.4	4.8	192.6	2.3	4.5	57.3	24.1	8.1	67.7
5804-100	70.9	0.4	14.6	4.2	0.1	1.0	0.1	0.3	4.8	0.0	1180	4.6	287.2	52.1	1.0	23.8	1.1	10.0	102.1	0.3	172.9	80.6	87.1	-23.5	36.4
5002-101	69.5	0.3	14.4	1.2	0.1	0.8	2.1	0.3	8.0	0.1	1156	3.3	281.9	15.3	1.7	18.9	37.6	8.4	169.4	1.7	28.8	37.4	38.4	-0.1	61.7
5805-297	64.4	0.5	14.5	4.1	0.1	2.6	2.0	0.6	6.0	0.2	1072	6.0	284.0	51.5	1.0	64.8	35.7	19.4	127.0	3.0	66.4	122.2	47.8	5.6	46.5
5805-434	71.3	0.3	13.3	2.0	0.2	0.8	1.8	0.4	6.7	0.1	1187	3.3	260.3	25.2	2.1	19.4	32.8	13.6	141.8	1.8	39.3	47.8	47.1	-0.6	53.5
5810-342	68.4	0.4	14.6	5.3	0.1	0.8	0.4	0.8	4.8	0.2	1138	4.6	285.4	66.6	0.7	20.3	7.1	24.8	101.3	2.3	145.0	91.6	76.0	-16.0	40.0
Waitun granite (Wang G.c et al., 2016 Lithos 246-247 197-211)																									
WT-2	72.2	0.3	14.3	2.7	0.1	0.5	1.2	5.4	2.9	0.1	1201	3.6	280.9	33.7	0.8	12.9	21.2	175.5	61.1	1.1	1.8	50.2	25.4	7.1	67.5
WT-3	72.6	0.3	13.9	2.5	0.0	0.6	1.9	3.9	3.5	0.1	1208	4.1	273.4	31.6	0.6	13.6	33.2	124.6	74.9	1.1	7.6	49.3	32.3	6.0	61.7
WT-4	71.9	0.3	14.4	2.2	0.1	0.6	1.7	3.7	4.3	0.1	1196	3.5	282.3	27.9	1.1	14.1	30.0	118.7	90.2	1.1	13.4	45.6	31.5	4.2	64.2
WT-5	73.3	0.2	14.1	1.7	0.0	0.4	0.7	4.1	4.9	0.1	1221	2.8	276.0	21.8	0.6	9.9	12.1	132.6	104.5	1.0	14.7	34.5	30.1	2.3	67.6
WT-6	73.1	0.3	13.5	2.5	0.0	0.6	1.9	3.8	3.5	0.1	1217	3.6	265.6	31.3	0.6	13.6	33.9	121.0	74.7	1.1	2.1	48.6	32.3	6.9	60.7

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	SiO ₂	TiO ₂	Al ₂ O ₃	FeO	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	Si	Ti	Al	Fe	Mn	Mg	Ca	Na	K	P	A	B	Q*3	B*3	F*3	
Early Yanshanian (165-150 Ma)																											
Huashan granite (Wang R.c et al., 2013 JAsEsc 74 361-372)																											
Hn-Bt-gr HS01	73.8	0.2	13.0	1.6	0.7	0.1	0.1	1.1	3.1	5.7	0.0	1228	2.9	255.0	23.0	0.7	2.5	19.6	98.4	120.0	0.6	-2.6	28.4	30.0	4.8	65.2	
Bt-gr HS05	72.6	0.3	13.8	1.6	0.7	0.1	0.2	1.0	3.4	5.6	0.1	1208	3.3	270.3	23.4	0.7	5.0	17.3	108.7	119.1	1.1	7.9	31.6	29.5	3.2	67.3	
f,g, Bt-gr HS11	76.9	0.1	12.4	0.5	0.5	0.0	0.1	0.4	4.5	3.1	0.0	1280	1.3	243.4	7.0	0.6	1.2	7.1	145.2	65.8	0.3	18.1	9.5	39.8	-2.2	62.4	
Wushi Fogang complex (Xu X.s et al., 2007 ScChD 50 209-220)																											
Wushi																											
Hn-gabbro FG32	49.0	2.3	16.3	8.7	3.9	0.2	4.9	10.3	1.8	0.9	0.6	814.8	28.7	320.1	169.1	2.5	120.6	183.0	58.4	18.3	8.5	-122.4	318.4	-12.2	74.6	37.7	
diorite FG31-2	54.7	1.1	16.4		9.3	0.2	4.3	7.6	2.5	2.2	0.3	910.5	14.3	321.5	116.7	2.5	107.2	136.1	81.6	46.9	3.5	-79.2	238.2	-1.4	52.6	48.8	
Ws-H	54.8	1.1	17.1	6.5	1.6	0.2	4.3	7.7	2.4	1.7	0.3	911.5	13.4	336.2	110.4	2.7	107.7	137.7	76.5	36.5	4.5	-52.1	231.5	6.6	46.2	47.1	
Fogang																											
granodiorite																											
FG33-2	68.3	0.6	14.3	3.1	1.3	0.1	0.9	2.7	3.1	4.3	0.2	1136	7.3	279.5	59.2	1.0	22.3	48.9	100.0	90.7	2.3	-8.9	88.8	25.0	15.4	59.7	
FG22-1	66.7	0.7	15.1	3.6	1.3	0.1	1.4	3.1	2.6	4.6	0.2	1110	8.1	295.4	65.6	1.1	35.7	55.6	83.3	97.7	3.0	3.2	109.4	26.7	16.0	57.3	
FG34-2	67.5	0.6	14.6		4.6	0.1	1.0	2.9	3.5	4.1	0.2	1123	7.9	287.2	57.6	1.6	24.3	52.1	112.0	87.5	2.3	-16.4	89.8	20.7	17.0	62.3	
monzogranite																											
FG9-1	68.4	0.6	14.6	3.8	0.9	0.1	0.9	2.8	3.0	4.0	0.2	1138	8.0	286.2	64.6	1.0	23.3	50.5	95.2	85.6	2.5	4.5	95.9	29.2	13.7	57.1	
FG15	69.2	0.4	14.8	2.2	1.1	0.1	0.9	2.4	3.5	3.8	0.1	1151	5.3	289.5	44.5	1.0	21.1	42.4	111.6	80.9	1.4	12.1	70.8	30.4	8.4	61.2	
FG2h	69.5	0.3	15.4	2.4	0.5	0.1	0.6	2.0	3.5	4.6	0.1	1157	4.3	302.5	38.8	0.7	14.1	34.8	112.3	96.8	1.5	23.8	57.2	30.9	3.9	65.2	
FG11-3h	70.3	0.4	14.6	2.1	0.9	0.1	0.8	2.3	3.2	4.0	0.1	1171	4.5	286.2	39.1	0.8	18.6	41.0	103.3	85.1	1.3	15.8	62.3	33.1	6.3	60.6	
FG17-2h	70.9	0.5	13.9	2.9	0.5	0.1	0.8	2.1	3.2	4.4	0.2	1180	6.0	273.2	46.9	1.0	19.1	37.6	101.6	92.4	2.3	4.0	72.1	30.5	10.1	59.3	
granite																											
FG-zh24	73.2	0.3	13.3	1.8	0.7	0.1	0.5	1.4	3.0	4.8	0.1	1218	3.3	261.3	33.4	0.8	11.2	25.7	95.5	102.1	1.0	12.3	47.8	35.2	4.8	60.0	
FG24-5	71.0	0.5	13.7	2.4	1.1	0.1	0.9	2.6	3.2	3.4	0.2	1181	6.4	268.7	47.2	0.8	21.6	46.0	102.6	72.4	2.4	1.7	75.2	32.6	11.1	56.2	
FG27	71.9	0.3	13.5	2.3	0.9	0.1	0.3	1.4	2.7	5.5	0.1	1196	3.9	263.8	42.2	0.8	8.4	25.7	87.4	117.2	1.3	7.8	54.5	32.0	6.8	61.3	
FG16	72.7	0.3	13.8	1.6	0.6	0.0	0.3	0.8	2.9	5.5	0.2	1210	3.1	271.1	29.5	0.4	7.9	13.6	92.3	117.4	2.1	34.3	40.6	38.6	-0.7	62.1	
Kfs-granite																											
FG5-1	76.9	0.2	11.9	1.0	0.5	0.0	0.1	0.8	2.7	4.9	0.0	1279	2.0	233.8	20.3	0.4	3.2	14.1	87.4	103.4	0.6	14.8	25.6	41.9	0.9	57.2	
FG29-1	77.2	0.2	12.2	0.8	0.1	0.1	0.1	0.4	3.7	4.5	0.0	1285	2.0	239.7	12.7	1.0	2.0	7.7	120.0	95.5	0.1	8.8	16.7	37.1	0.8	62.2	
Shiling																											
sodalite syenite FG3-2	59.6	0.3	18.4	3.2	2.2	0.2	0.5	1.7	6.7	5.9	0.1	991.0	4.1	360.5	71.4	2.4	11.7	30.1	216.2	125.5	1.3	-41.4	87.2	-13.2	20.8	92.4	
Xihuashan complex (Guo C et al., 2012 Lithos 148 209-227)																											
first phase (I)																											
XHS-3	76.0	0.1	12.8	0.8	0.5	0.1	0.2	0.6	3.0	5.0	0.1	1264	1.6	251.5	17.5	1.0	5.5	11.4	97.8	105.1	0.7	25.8	24.6	41.2	-1.4	60.2	
XHS-4	75.4	0.1	13.0	0.9	0.5	0.1	0.2	0.5	2.9	5.3	0.0	1255	1.5	254.0	19.6	1.0	5.0	8.4	92.0	111.9	0.6	33.4	26.0	42.5	-2.7	60.2	
XHS-7	75.8	0.1	12.8	0.9	0.3	0.1	0.2	1.0	3.4	4.6	0.0	1262	1.0	251.7	15.3	1.0	4.0	17.8	109.4	96.8	0.3	9.8	20.3	36.5	1.1	62.3	
XHS-8	76.5	0.1	12.7	0.8	0.3	0.1	0.2	0.9	3.4	4.7	0.0	1273	0.9	249.7	14.6	1.0	4.0	16.0	108.1	98.9	0.4	10.6	19.4	37.2	0.8	62.0	
XHS-9	74.5	0.1	13.3	0.9	0.3	0.1	0.2	1.0	3.4	4.9	0.0	1240	1.1	259.9	16.1	1.0	4.2	17.1	110.4	103.2	0.6	12.1	21.5	34.7	0.8	64.5	
XHS-10	74.7	0.1	13.3	0.9	0.2	0.1	0.2	1.1	3.4	5.0	0.0	1243	1.3	261.1	15.6	1.0	4.7	19.4	108.4	105.1	0.4	8.7	21.5	33.8	1.5	64.7	
XHS-11	77.0	0.1	12.1	0.0	0.3	0.1	0.2	1.0	3.2	4.5	0.0	1282	1.3	237.3	4.3	1.1	5.0	17.5	102.6	95.5	0.4	4.2	10.5	38.2	0.8	61.0	
XHS-25	74.6	0.1	13.4	1.1	0.3	0.1	0.3	1.4	2.9	5.2	0.0	1242	1.8	263.2	19.8	0.8	7.2	24.1	92.3	109.3	0.6	13.5	28.8	36.1	1.7	62.2	
XHS-30	75.5	0.1	12.7	0.9	0.4	0.1	0.2	1.0	3.2	4.8	0.0	1257	1.1	248.9	16.9	1.0	3.7	18.4	104.2	102.1	0.4	5.8	21.7	35.5	2.1	62.4	
XHS-31	76.1	0.1	12.6	0.8	0.3	0.1	0.1	0.9	3.2	4.9	0.0	1266	1.0	247.5	14.8	1.0	3.2	16.4	103.9	103.6	0.3	7.2	19.1	36.2	1.5	62.4	
XHS-32	76.5	0.1	12.4	0.9	0.4	0.1	0.2	1.0	3.1	4.8	0.0	1272	1.1	243.4	17.2	1.1	4.0	16.9	100.7	102.8	0.4	6.1	22.3	36.9	2.2	60.9	
XHS-33	75.2	0.1	13.1	0.9	0.3	0.1	0.2	1.0	3.2	5.2	0.0	1251	1.1	256.2	16.4	1.0	3.7	18.0	102.9	110.2	0.4	7.0	21.2	34.1	1.8	64.1	
XHS-34	76.8	0.1	12.0	0.9	0.3	0.1	0.2	0.9	3.1	4.7	0.0	1278	1.1	235.2	17.3	1.1	3.7	15.7	99.7	99.6	0.4	4.5	22.2	38.0	2.5	59.6	
XHS-35	74.8	0.1	13.3	0.8	0.3	0.1	0.1	1.0	3.4	5.1	0.0	1244	1.1	260.1	14.6	1.0	3.5	18.5	110.7	107.6	0.4	4.7	19.2	32.3	2.0	65.7	
second phase (II)																											
X09-3	75.8	0.0	13.1	0.5	0.3	0.1	0.1	0.5	4.0	4.4	0.0	1261	0.3	256.4	10.8	1.7	1.2	8.2	128.8	93.4	0.1	17.8	12.3	36.4	-1.7	65.3	
X09-4	76.3	0.0	13.0	0.6	0.1	0.1	0.1	0.5	4.1	4.5	0.0	1270	0.1	255.4	9.4	1.7	1.5	9.1	133.6	95.1	0.1	8.5	11.0	33.6	0.0	66.5	
X09-5	76.0	0.0	12.9	0.6	0.2	0.1	0.1	0.5	3.9	4.6	0.0	1265	0.3	253.8	9.7	1.6	1.7	9.6	126.5	98.3	0.1	9.8	11.6	34.3	-0.2	65.9	
XHS-1	76.8	0.0	12.7	0.6	0.1	0.1	0.0	0.6	3.8	4.7	0.0	1278	0.1	248.3	9.9	1.6	1.0	10.7	122.6	99.6	0.1	4.7	11.0	34.3	0.7	65.0	
XHS-2	76.0	0.0	13.1	0.6	0.2	0.1	0.0	0.6	4.0	4.7	0.0	1265	0.1	256.2	10.7	1.6	1.0	9.8	129.7	98.9	0.1	7.9	11.9	33.2	0.2	66.6	
XHS-12	75.7	0.0	13.3	0.4	0.2	0.1	0.0	0.5	4.0	4.7	0.0	1259	0.1	260.1	8.2	0.8	1.0	9.6	128.8	100.0	0.1	12.1	9.3	33.7	-1.0	67.3	
XHS-13	75.5	0.0	13.0	0.5	0.2	0.1	0.1	0.6	4.1	4.6	0.0	1257	0.3	255.2	9.7	1.1	1.2	10.7	132.6	97.2	0.1	3.9	11.2	31.7	0.9	67.4	
XHS-14	75.7	0.0	12.9	0.6	0.1	0.1	0.1	0.6	4.1	4.4	0.0	1260	0.3	253.6	9.4	1.4	1.2	11.4	133.3	92.6	0.1	5.0	10.9	32.7	0.7	66.6	
XHS-21	76.1	0.0	13.0	0.6	0.2	0.1	0.1	0.7	4.1	4.4	0.0	1266	0.3	254.0	10.7	1.3	1.5	11.6	133.6	92.4	0.1	4.9	12.4	32.9	0.9	66.2	
XHS-22	76.2	0.0	12.9	0.5	0.2	0.1	0.1	0.7	4.0	4.4	0.0	1268	0.3	253.2	10.3	1.1	1.5	12.7	130.4	92.4	0.1	5.2	12.0	33.5	0.8	65.7	
XHS-23	75.8	0.0	13.0	0.6	0.2	0.1	0.0	0.6	4.0	4.7	0.0	1262	0.1	255.4	10.4	1.1	1.0	10.5	129.7	98.9	0.1	5.7	11.5	32.5	0.6	66.9	
XHS-24	76.2	0.0	13.1	0.5	0.3	0.1	0.0	0.6	4.1	4.4	0.0	1268	0.3	257.2	10.3	1.0	1.0	10.3	133.3	93.6	0.1	9.6	11.6	33.8	-0.2	66.3	
third phase (III)																											

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XHS-15	75.3	0.1	13.0	1.1	0.4	0.1	0.1	0.7	3.5	4.5	0.0	1254	1.0	254.2	20.2	1.4	3.0	12.5	112.6	95.8	0.4	20.9	24.1	38.6	-0.5	61.9	
XHS-16	75.6	0.1	12.7	0.5	0.8	0.1	0.1	0.6	3.5	4.5	0.0	1258	0.9	249.9	16.7	1.4	3.2	10.5	111.3	96.0	0.4	21.6	20.8	39.5	-1.1	61.6	
XHS-17	75.0	0.1	13.4	1.5		0.1	0.1	0.8	3.6	4.6	0.0	1248	1.1	263.4	20.8	1.4	3.2	13.4	115.5	98.5	0.4	22.7	25.2	37.3	-0.7	63.4	
XHS-18	75.8	0.1	13.1	0.8	0.3	0.1	0.1	0.6	3.5	4.8	0.0	1262	1.0	256.2	15.0	1.4	3.2	10.2	113.3	101.5	0.4	21.1	19.2	38.1	-1.3	63.2	
XHS-19	75.9	0.1	13.1	0.7	0.3	0.1	0.1	0.7	3.7	4.5	0.0	1263	0.8	256.8	12.5	1.4	2.7	12.5	120.4	96.4	0.3	15.1	16.0	36.3	-0.6	64.3	
XHS-20	75.1	0.1	12.9	0.9	0.3	0.1	0.2	0.7	3.6	4.6	0.0	1250	1.0	253.8	16.7	1.3	3.7	12.3	116.2	96.6	0.4	16.4	21.4	36.8	0.0	63.2	
XHS-26	78.1	0.0	12.1	0.5	0.3	0.1	0.1	0.4	3.7	4.2	0.0	1299	0.5	237.0	9.8	1.3	2.0	7.8	119.7	89.8	0.3	11.7	12.3	39.4	-0.5	61.1	
XHS-27	76.0	0.1	13.0	0.7	0.3	0.1	0.1	0.6	3.5	4.9	0.0	1265	0.9	255.0	13.6	1.0	3.2	10.7	114.2	103.4	0.4	16.0	17.7	36.6	-0.5	63.9	
XHS-28	76.3	0.0	12.7	0.5	0.2	0.1	0.1	0.5	3.9	4.7	0.0	1269	0.4	248.3	9.9	1.3	1.5	9.3	125.2	99.8	0.1	4.8	11.7	33.6	0.8	65.6	
XHS-29	76.3	0.0	12.8	0.6	0.2	0.1	0.1	0.6	4.0	4.5	0.0	1270	0.4	251.9	10.2	1.3	1.2	9.8	130.0	95.1	0.1	7.1	11.8	33.9	0.4	65.7	
fourth phase (IV)																											
XHS-36	74.9	0.1	13.4	0.5	0.6	0.0	0.2	0.2	3.2	5.7	0.0	1246	1.5	262.3	14.6	0.4	4.0	4.1	102.9	120.4	0.4	30.7	20.1	38.5	-3.1	64.6	
XHS-37	74.9	0.1	13.5	0.5	0.8	0.1	0.2	0.2	3.3	5.4	0.0	1246	1.6	264.2	16.9	0.7	4.2	3.7	105.2	114.4	0.4	37.1	22.7	40.4	-3.9	63.6	
Guposhan complex (Wang Z et al., 2017 AmJ of Sc 314 235-277)																											
Major phase																											
GP-2	72.8	0.3	14.0		2.7	0.1	0.2	1.4	2.6	5.1	0.1	1212	4.0	275.2	0.6	0.7	5.2	24.8	84.9	107.4	1.3	33.3	9.8	41.0	-5.3	64.3	
GP-4	72.2	0.3	14.0		3.1	0.1	0.3	1.4	2.7	5.1	0.1	1202	4.3	274.4	0.6	0.7	6.5	25.1	87.1	107.9	1.4	29.2	11.3	39.1	-4.2	65.1	
GP-9	74.8	0.4	12.7		3.0	0.1	0.2	1.6	1.9	4.3	0.1	1245	4.6	249.7	0.8	0.8	5.2	28.5	62.6	90.4	1.4	39.6	10.6	51.4	-6.5	55.1	
GP-22	73.9	0.3	13.7		2.6	0.1	0.1	1.5	2.2	4.8	0.1	1230	3.4	268.7	0.9	1.0	2.5	26.6	71.0	101.1	0.8	43.5	6.7	47.7	-7.9	60.2	
GP11-42	77.5	0.1	12.1		0.6	0.0	0.2	0.3	3.2	5.3	0.0	1289	0.8	236.8	0.1	0.1	4.0	4.6	103.9	111.5	0.1	12.1	4.8	38.6	-1.7	63.1	
GP11-43	75.0	0.2	12.8		1.8	0.0	0.3	0.9	3.9	4.6	0.0	1248	2.3	250.3	0.4	0.4	6.7	15.9	124.6	97.2	0.6	-3.2	9.3	30.9	2.1	67.0	
GP11-44	77.2	0.2	11.4		1.6	0.0	0.3	0.6	3.3	4.7	0.0	1285	1.9	223.0	0.4	0.4	6.2	10.2	106.8	98.7	0.4	-2.8	8.5	36.8	1.9	61.3	
GP11-48	74.2	0.2	13.2		1.7	0.0	0.3	1.0	3.7	5.3	0.0	1234	2.3	257.9	0.4	0.4	7.4	16.9	118.7	113.4	0.6	-8.1	10.1	27.2	3.1	69.7	
GP11-50	69.5	0.4	14.5		3.1	0.1	0.6	1.8	4.1	4.8	0.1	1157	5.1	285.2	0.6	0.7	15.1	31.2	132.0	101.9	2.0	-11.1	20.9	20.5	5.5	74.0	
GP11-53	72.6	0.3	13.4		2.5	0.0	0.4	1.2	3.8	5.2	0.1	1208	3.1	263.6	0.5	0.6	9.4	21.4	122.9	110.2	1.0	-12.3	13.1	24.3	4.5	71.2	
GP11-54	68.7	0.5	14.6		3.4	0.1	0.8	1.9	4.3	4.8	0.2	1143	5.8	287.2	0.9	1.0	19.1	33.2	140.0	102.8	2.8	-22.0	25.7	15.7	8.4	75.9	
GP11-60	74.7	0.1	13.2		1.3	0.0	0.3	0.6	3.6	5.8	0.0	1243	1.6	259.1	0.5	0.6	6.5	10.0	114.6	123.8	0.4	0.8	8.6	29.0	1.1	69.8	
GP11-98	73.7	0.2	12.9		2.1	0.0	0.3	0.8	3.9	5.1	0.0	1227	2.4	253.6	0.5	0.6	6.7	15.0	125.2	109.1	0.6	-10.7	9.6	26.3	3.6	70.1	
GP11-102	74.7	0.1	12.8		1.1	0.1	0.3	1.1	3.3	5.4	0.0	1242	1.1	251.5	0.6	0.7	6.5	19.3	104.9	113.6	0.1	-5.5	8.2	30.4	2.4	67.2	
GP11-103	73.6	0.2	13.0		2.2	0.0	0.4	1.0	3.9	4.8	0.1	1224	2.8	254.6	0.4	0.4	8.9	18.5	125.8	101.1	0.8	-9.4	12.1	27.3	3.7	69.0	
GP11-105	73.2	0.2	13.3		1.9	0.0	0.4	1.0	3.7	5.6	0.1	1218	2.6	261.1	0.4	0.4	8.7	17.1	120.4	118.5	0.8	-12.0	11.7	24.4	4.2	71.5	
MME																											
GP11-46	56.2	1.5	16.4		8.3	0.2	2.6	4.6	4.8	3.9	0.6	934.4	18.1	320.7	2.4	2.7	65.3	81.1	153.6	82.6	8.0	-77.7	85.8	-12.6	30.2	82.4	
GP11-47	58.4	1.2	16.0		7.6	0.2	2.1	3.5	4.5	4.8	0.6	971.4	15.3	313.8	1.9	2.1	51.6	62.1	145.2	100.8	8.2	-56.3	68.8	-5.3	22.9	82.4	
GP11-52	57.4	1.4	16.3		9.1	0.2	2.2	3.1	4.9	3.5	0.5	955.2	17.6	320.3	1.9	2.1	54.6	54.9	156.8	73.9	7.6	-20.2	74.1	5.0	16.4	78.5	
Late stage																											
GP-11	80.9	0.1	11.1		1.0	0.0	0.0	0.4	1.7	4.4	0.0	1345	0.6	217.5	0.1	0.1	0.2	7.8	53.2	93.4	0.1	55.2	1.0	63.0	-11.1	48.1	
GP-12	78.4	0.1	12.4		1.1	0.0	0.0	0.5	3.1	3.7	0.0	1304	0.9	242.3	0.1	0.1	0.2	9.1	99.4	78.8	0.0	45.9	1.2	52.6	-9.1	56.5	
GP-14	78.8	0.1	11.7		1.6	0.0	0.0	0.6	2.6	4.0	0.0	1311	1.1	229.9	0.3	0.3	0.2	10.7	82.3	84.3	0.0	41.9	1.6	54.5	-8.3	53.8	
GP-15	77.8	0.1	12.1		1.5	0.0	0.0	0.6	2.1	4.9	0.1	1295	1.1	237.5	0.3	0.3	0.2	10.0	67.8	103.6	1.4	46.2	1.6	53.8	-9.2	55.4	
GP-16	78.5	0.1	12.0		1.4	0.0	0.0	0.5	2.5	4.4	0.0	1307	1.1	234.8	0.1	0.1	0.2	8.6	81.6	92.8	0.0	43.3	1.5	53.2	-8.5	55.4	
GP-17	78.5	0.1	12.1		0.9	0.0	0.0	0.4	2.0	5.1	0.0	1306	0.8	237.2	0.1	0.1	0.2	7.1	65.8	107.9	0.1	49.2	1.1	54.7	-9.8	55.1	
GP-18	77.3	0.0	12.8		1.0	0.0	0.0	0.7	3.1	4.1	0.0	1287	0.4	251.3	0.4	0.4	0.2	12.1	101.3	86.4	0.1	39.3	1.0	48.1	-7.8	59.6	
GP11-31	76.9	0.1	12.3		0.1	0.0	0.2	1.2	3.0	5.7	0.0	1280	0.9	240.5	0.1	0.1	4.0	20.5	96.2	120.0	0.1	-16.6	5.0	30.2	4.1	65.7	
GP11-41	75.5	0.1	12.8		1.2	0.0	0.5	0.7	4.2	5.0	0.0	1256	0.9	250.3	0.3	0.3	11.7	11.6	133.9	107.0	0.0	-13.8	12.8	26.1	4.7	69.3	
GP11-62	76.7	0.1	12.3		1.0	0.0	0.2	0.5	3.6	5.1	0.0	1276	0.9	241.3	0.3	0.3	4.5	8.2	116.2	107.2	0.1	1.5	5.6	33.9	0.6	65.5	
GP11-68	75.6	0.1	12.5		0.9	0.0	0.2	0.5	2.8	6.4	0.0	1259	0.8	245.0	0.0	0.0	4.0	8.7	90.0	135.7	0.0	1.8	4.7	32.7	0.4	66.9	
GP11-71	75.6	0.1	12.4		1.4	0.0	0.3	1.0	3.4	4.9	0.0	1258	1.1	243.8	0.4	0.4	7.7	16.9	108.4	104.0	0.3	-2.5	9.2	33.2	1.9	64.9	
GP11-73	76.2	0.0	12.6		0.8	0.0	0.2	0.8	3.7	4.6	0.0	1268	0.5	247.9	0.4	0.4	6.0	13.7	118.1	98.3	0.3	4.1	6.8	34.5	0.2	65.2	
GP11-74	75.2	0.1	12.9		1.0	0.0	0.2	0.7	3.3	5.4	0.0	1251	0.6	253.4	0.4	0.4	5.2	12.8	104.9	115.3	0.1	7.6	6.2	33.9	-0.6	66.7	
Guposhan granite (Wang R.c et al., 2013 JAsESc 74 361-372)																											
Hn-Bt gr GP2-08	69.4	0.4	14.6	1.7	1.6	0.1	0.6	1.7	3.6	5.3	0.2	1155	5.3	286.8	23.8	0.8	15.4	31.0	115.8	113.2	2.5	-4.3	44.5	22.4	7.6	70.0	
Bt-gr GP09	72.1	0.3	13.7	2.0	0.6	0.1	0.2	1.3	3.4	5.3	0.1																

Chapter 1

XY5-1	76.9	0.1	12.6	0.8	0.1	0.1	0.7	3.5	4.5	0.0	1280	0.8	247.0	12.0	1.0	2.7	12.7	111.6	94.5	0.3	15.5	15.5	39.2	-0.7	61.5
XY5-3	76.0	0.1	13.2	0.7	0.1	0.2	0.7	3.4	5.0	0.0	1265	0.6	259.3	10.2	0.8	3.7	11.6	109.4	106.6	0.3	20.2	14.6	37.6	-1.8	64.2
XY6-2	77.0	0.1	12.7	0.6	0.0	0.2	0.4	2.9	4.8	0.0	1282	0.6	249.5	9.3	0.6	4.7	7.8	93.3	101.7	0.3	38.9	14.6	46.7	-5.6	58.8
Stage II 2m granite XY3-1	73.7	0.2	14.1	1.2	0.0	0.3	0.9	3.0	5.6	0.2	1226	2.4	276.4	16.9	0.4	8.4	16.8	96.5	118.7	2.1	27.7	27.7	36.6	-1.3	64.7
	XY3-3	73.7	0.2	14.3	1.1	0.0	0.4	0.7	3.0	5.6	0.2	1227	2.4	279.5	16.1	0.4	8.7	13.0	95.5	118.3	2.1	39.7	27.1	39.7	-3.8
XY3-6	74.1	0.2	13.9	1.4	0.1	0.4	1.0	3.1	5.1	0.2	1232	2.9	273.2	20.2	0.7	9.9	16.9	98.4	107.6	2.3	33.3	33.0	39.6	-1.6	62.1
XY3-7	74.2	0.2	13.9	1.2	0.0	0.4	0.8	2.9	5.7	0.2	1235	2.4	273.0	16.5	0.6	8.7	13.9	92.6	120.6	2.3	32.0	27.5	38.6	-2.2	63.6
XY4-3	73.6	0.3	14.0	1.3	0.0	0.5	0.8	2.7	5.8	0.2	1224	3.4	273.8	18.7	0.3	11.9	13.6	85.5	122.1	2.3	39.1	34.0	40.6	-2.6	62.1
XY4-4	73.1	0.3	14.0	1.4	0.0	0.5	0.5	2.4	5.7	0.2	1216	3.9	273.6	19.8	0.3	12.4	9.1	78.1	121.0	2.4	56.3	36.1	46.0	-5.8	59.8
XY4-5	73.4	0.3	14.5	1.1	0.0	0.4	0.6	2.5	5.9	0.2	1221	3.5	283.6	15.5	0.3	10.2	10.7	81.3	125.3	2.4	55.7	29.2	44.2	-6.7	62.4
XY4-7	75.4	0.2	13.7	1.0	0.0	0.3	0.9	2.8	5.0	0.1	1255	2.1	268.1	14.4	0.6	6.9	15.3	91.3	105.7	1.3	40.4	23.5	44.0	-4.5	60.4
XY8-1	70.0	0.5	14.7	2.7	0.1	1.0	1.8	2.8	5.2	0.2	1164	5.9	287.8	37.5	0.7	23.8	32.6	90.4	109.8	2.8	22.4	67.2	32.9	5.8	61.3
XY9-1	70.0	0.5	13.7	3.3	0.1	1.2	2.4	2.5	4.4	0.2	1165	6.8	269.5	46.5	1.0	28.5	43.3	80.0	93.8	2.1	9.0	81.8	33.9	10.8	55.3
Biotite granite XY9-3	69.1	0.6	14.3	3.4	0.1	1.2	2.6	2.8	4.5	0.2	1149	7.1	280.5	47.7	1.0	30.3	46.4	90.0	96.4	2.3	1.4	85.1	28.7	12.7	58.6
	XY9-5	70.0	0.5	14.1	3.1	0.1	1.1	2.4	2.7	4.7	0.1	1165	6.6	276.8	44.4	1.1	26.8	43.0	87.1	98.9	1.8	4.8	77.8	30.8	10.9
XY9-6	70.7	0.4	14.0	2.6	0.1	0.9	2.3	2.7	4.9	0.1	1176	5.5	273.8	37.7	1.1	23.3	40.5	85.5	104.0	1.7	3.3	66.5	30.8	9.5	59.7
XY9-9	71.7	0.3	14.2	2.2	0.1	0.8	2.1	3.1	4.7	0.1	1194	4.3	278.9	30.9	0.7	18.9	37.3	99.1	100.4	1.5	4.9	54.0	30.5	7.2	62.3
XY10-1	70.2	0.6	14.1	3.2	0.1	1.2	2.9	2.9	4.2	0.2	1168	6.9	275.6	44.9	0.8	29.0	51.9	93.3	89.0	2.1	-10.4	80.9	27.4	14.3	58.3
XY10-2	68.9	0.6	14.4	3.2	0.1	1.2	2.8	2.9	4.5	0.2	1146	7.1	282.3	45.4	0.8	29.8	50.5	93.6	96.0	2.3	-8.2	82.3	25.6	14.2	60.3
XY10-3	70.0	0.5	14.1	2.8	0.1	1.0	2.4	2.8	5.0	0.1	1165	6.3	277.4	39.3	0.8	25.3	43.3	88.7	106.2	1.8	-4.2	70.8	27.4	11.6	61.0

J2-3 mafic and felsic rocks from Hunan (Jiang T.h et al., 2009 Lithos 167 185-204)

Tongshanling																											
syenogranite																											
TSL-1	66.0	0.5	16.0		4.0	0.1	1.3	0.9	1.6	5.5	0.2	1098	5.9	313.3	50.6	1.3	32.3	16.0	50.0	116.1	2.8		115.0	88.7	57.7	-9.9	52.2
TSL-2	68.1	0.5	15.4		3.8	0.1	1.6	1.0	1.9	4.6	0.2	1133	5.8	301.1	47.3	1.0	40.2	17.1	60.7	98.1	2.5		108.1	93.3	58.8	-7.6	48.8
monzogranite																											
TSL-3	67.4	0.4	15.5		4.0	0.1	1.5	2.0	2.6	4.5	0.2	1121	5.4	303.3	49.5	1.3	36.7	35.0	83.3	95.5	2.4		54.6	91.6	40.8	3.1	56.1
TSL-4	67.6	0.4	15.3		4.1	0.1	1.5	1.1	2.1	4.5	0.2	1125	5.5	300.1	51.1	1.1	37.2	19.4	67.8	96.0	2.4		97.5	93.8	55.0	-5.4	50.3
MME																											
TSL-5	58.9	0.9	18.2		6.1	0.1	2.3	3.0	2.5	3.5	0.3	980.7	10.6	357.8	76.1	2.0	57.8	52.6	81.6	74.7	3.9		96.2	144.6	43.9	2.9	53.2
TSL-6	67.9	0.4	15.1		4.0	0.1	1.5	2.3	2.5	3.9	0.2	1129	5.1	296.8	49.8	1.3	36.0	40.3	81.0	82.0	2.3		53.2	91.0	43.4	3.3	53.4
TSL-7	68.8	0.4	14.8		3.8	0.1	1.4	2.3	2.6	4.1	0.2	1145	5.1	290.9	48.0	1.3	35.5	40.3	84.2	87.9	2.3		38.2	88.6	39.3	5.9	54.8
Jinjiiling																											
Kfs-granite																											
JJL-1	77.0	0.1	12.2		1.5	0.0	0.2	0.2	3.0	4.8	0.0	1281	1.6	238.7	18.4	0.4	3.7	3.7	96.5	101.7	0.4		33.1	23.8	45.5	-3.0	57.5
JJL-2	74.1	0.1	13.6		1.7	0.1	0.1	0.1	2.4	6.5	0.0	1232	1.6	267.6	20.7	1.0	3.5	1.6	78.4	138.9	0.4		47.1	25.8	42.5	-5.5	63.0
JJL-4	73.8	0.1	13.2		1.7	0.1	0.2	0.8	2.6	6.3	0.0	1228	1.8	259.5	20.8	1.0	5.0	13.9	82.3	133.1	0.6		16.3	27.5	35.0	0.9	64.0
syenogranite																											
JJL-3	74.5	0.2	12.9		1.7	0.0	0.1	0.9	3.4	4.8	0.0	1240	1.9	253.2	21.3	0.4	3.5	15.9	111.0	102.3	0.6		8.2	26.6	34.1	2.4	63.4
JJL-5	78.0	0.1	11.2		1.6	0.0	0.2	0.6	2.9	4.5	0.0	1298	1.6	219.7	19.5	0.6	3.7	10.9	92.9	96.2	0.4		8.8	24.9	42.4	2.0	55.5
JJL-6	73.6	0.2	13.5		1.9	0.0	0.3	0.9	3.1	5.5	0.0	1224	2.0	264.2	23.2	0.6	6.5	16.2	98.4	117.6	0.6		15.7	31.6	34.2	1.7	64.2
JJL-8	76.5	0.2	11.8		1.8	0.0	0.2	0.8	2.6	5.6	0.0	1273	2.1	232.1	22.7	0.4	4.5	14.4	84.9	119.5	0.6		-1.2	29.3	35.8	4.7	59.6
monzogranite																											
JJL-9	74.4	0.2	12.5		2.3	0.0	0.3	1.3	2.7	5.3	0.1	1238	3.0	245.4	28.2	0.6	7.9	22.8	86.8	112.7	1.0		0.2	39.1	34.1	5.9	60.0
Xishan																											
Kfs-granite																											
XS-2	74.3	0.2	12.5		2.4	0.0	0.5	0.5	2.7	5.7	0.1	1237	2.8	246.0	29.9	0.4	11.7	8.7	86.8	121.0	1.0		20.7	44.3	38.3	2.6	59.1
XS-3	71.8	0.2	12.8		2.6	0.0	0.5	0.3	2.9	5.9	0.1	1195	3.0	250.1	32.9	0.6	12.4	5.3	92.6	125.3	1.1		21.5	48.3	35.3	3.1	61.6
XS-8	75.1	0.1	12.8		1.7	0.0	0.2	0.5	3.1	5.0	0.1	1250	1.4	251.5	21.8	0.6	3.7	8.7	100.4	107.0	0.8		26.6	26.9	40.3	-1.2	60.9
XS-9	75.1	0.1	12.9		1.6	0.0	0.2	0.1	2.9	5.4	0.1	1250	1.4	253.2	19.8	0.4	4.7	2.5	94.9	113.8	0.7		39.6	25.9	43.5	-4.0	60.5
XS-10	74.8	0.2	12.8		2.1	0.0	0.2	0.4	2.4	5.8	0.1	1245	2.4	250.1	26.1	0.6	5.7	6.6	76.5	122.1	0.8		38.3	34.1	44.4	-2.5	58.1
syenogranite																											
XS-1	74.0	0.2	12.0		2.5	0.0	0.5	0.9	2.6	5.6	0.1	1232	2.9	234.4	31.1	0.6	13.2	16.2	82.3	118.3	1.3		1.4	47.1	34.8	7.0	58.2
XS-5	73.8	0.2	12.6		2.6	0.0	0.3	0.8	2.3	5.8	0.1	1229	2.8	247.7	32.3	0.6	7.7	14.4	73.9	123.8	1.1		21.2	42.8	39.3	2.3	58.4
XS-11	73.7	0.2	12.7		2.2	0.0	0.3	1.2	2.9	5.1	0.1	1226	2.3	249.9	27.1	0.6	6.2	21.6	92.6	108.1	1.1		6.1	35.5	34.7	4.2	61.0
XS-12	74.6	0.2	12.7		2.1	0.1	0.2	1.2	2.7	5.2	0.1	1241	2.0	249.7	26.8	0.7	4.5	21.2	88.1	111.3	1.0		7.9	33.3	36.0	3.5	60.5
monzogranite																											
XS-7	71.8	0.3	13.1		3.3	0.1	0.3	1.6	2.6	5.5	0.1	1194	3.9	257.7	41.7	0.7	8.2	28.0	83.3	116.3	1.5		2.2	53.8	31.5	7.8	60.6
Ninggyan shoshonite																											
TYS-1	51.5	1.5	15.7	4.9	3.5	0.2	5.1	6.6	3.8	2.4	0.7	857.7	18.4	307.2	111.8	2.8	125.3	118.4	123.6	51.0	10.1		-104.2	255.5	-15.6	61.0	54.6
TYS-2	50.2	1.5	15.5	6.4	3.3	0.2	6.5	6.7	3.8	2.3	0.7	834.9	18.9	303.1	130.4	2.4	162.0	119.8	121.7	48.4	9.9		-106.7	311.3	-16.6	69.2	47.4
Changchenling basalte																											
YZH-4	50.3	2.1	17.1	7.1	2.9	0.1	3.8	11.0	2.2	0.3	0.3	836.2	25.9	335.6	135.8	1.8	95.3	195.8	72.0	6.6	4.5		-134.5	257.0	-15.4	67.9	47.5
basalte andesitique																											

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YZH-8	52.0	2.0	17.3	6.7	3.0	0.1	3.9	9.4	2.5	0.5	0.3	864.5	24.5	339.9	130.9	1.8	97.8	167.6	80.7	11.0	4.1	-87.0	253.2	-2.9	57.1	45.9
YZH-13	49.9	2.0	17.0	7.4	2.6	0.1	3.2	11.3	1.8	0.6	0.3	829.9	25.5	334.1	134.9	1.7	80.4	202.0	58.7	11.7	4.5	-140.4	240.8	-16.5	67.6	48.9
Daoxian basalte																										
HZY-13	46.9	0.5	8.3	3.4	3.0	0.1	15.7	14.1	1.0	1.4	0.6	779.8	6.1	163.0	83.6	2.0	389.1	252.1	31.3	30.1	7.9	-402.7	478.9	-76.0	154.8	21.2
HZY-18	47.7	0.6	9.0	3.0	3.8	0.2	14.8	13.3	1.4	1.3	0.6	794.0	7.3	175.8	89.0	2.3	366.7	237.3	46.1	27.6	7.9	-372.7	463.0	-69.2	145.6	23.6
Guiyang lamprophyre																										
ZHC-10	49.8	0.6	9.4	4.7	3.0	0.1	15.1	6.9	1.1	4.2	0.5	829.3	7.3	184.0	103.1	2.0	375.4	123.8	35.2	89.2	7.5	-187.9	485.8	-25.7	111.8	13.9
ZHC-13	47.9	0.6	9.1	4.9	5.6	0.2	15.2	6.7	1.0	4.2	0.5	797.5	7.0	178.1	138.2	2.1	378.2	118.6	32.3	88.5	7.5	-179.9	523.4	-24.8	116.5	8.3
Qitianling complex (Xie L et al., 2010 Chem Geol 276 53-68)																										
first stage (I)																										
QT-38	66.7	0.9	13.7	5.2		0.1	1.2	3.1	3.0	4.4	0.3	1110	11.1	268.7	72.7	1.8	28.5	54.7	96.5	93.8	3.5	-31.1	112.4	18.5	23.5	58.0
QT-63	68.2	0.6	13.8	3.9		0.1	0.7	2.3	3.0	4.9	0.2	1134	7.1	270.3	53.9	1.1	16.4	41.5	97.1	103.2	2.3	-13.1	77.4	23.5	14.6	61.9
QT-110	70.0	0.5	13.6	3.5		0.1	0.6	1.9	3.1	5.2	0.2	1165	6.8	266.6	48.9	1.1	15.6	33.9	101.0	110.2	3.0	-12.4	71.3	24.0	13.4	62.6
QT-121	72.8	0.4	13.1	2.6		0.1	0.4	1.6	3.0	5.2	0.1	1211	4.8	256.4	35.9	0.7	9.9	29.3	95.3	109.4	1.6	-6.9	50.6	29.4	9.1	61.6
Cailling phase																										
QTL-6	67.3	0.9	13.9	3.3	2.2	0.1	1.0	3.1	3.3	4.1	0.3	1120	11.5	273.2	72.9	1.4	25.1	55.5	106.8	87.3	3.5	-31.7	109.5	18.0	23.0	59.0
QTL-14	67.3	0.7	14.0	2.9	1.7	0.1	0.9	3.3	3.8	5.0	0.2	1119	8.9	275.2	61.4	1.3	22.6	58.1	122.6	105.5	3.2	-69.2	92.9	4.3	27.6	68.1
QT-27	67.6	0.9	12.6	4.3	1.8	0.2	1.1	2.5	2.9	4.5	0.3	1126	10.8	247.2	82.6	2.1	27.3	44.6	94.2	94.7	4.2	-30.9	120.6	20.9	24.9	54.2
QT-29	67.0	0.9	13.1	4.2	1.7	0.2	1.2	2.6	3.2	4.4	0.3	1115	11.0	256.2	79.1	2.3	30.3	46.0	103.9	92.8	4.6	-32.5	120.4	18.4	25.1	56.5
QT-30	66.5	0.7	14.0	3.4	1.5	0.1	1.1	2.2	3.1	4.9	0.3	1107	9.0	274.8	65.6	1.6	27.3	39.2	99.1	104.9	4.2	-7.6	101.9	22.6	17.3	60.2
second stage (II)																										
QT-55	74.0	0.3	12.7	2.1		0.1	0.3	1.5	3.1	4.9	0.0	1231	3.3	249.5	29.8	1.1	6.5	27.1	98.4	103.8	0.6	-6.9	39.5	31.2	7.4	61.4
QT-72	69.4	0.5	13.3	3.4		0.1	0.6	2.3	3.1	4.7	0.1	1155	6.5	260.5	47.9	1.4	13.9	40.3	100.0	98.9	1.8	-19.1	68.3	23.8	14.4	61.7
QT-82	71.1	0.4	13.1	2.4		0.1	0.5	1.7	3.2	5.3	0.1	1184	4.4	257.7	33.3	0.8	11.2	30.3	103.6	111.7	1.1	-18.1	48.8	23.8	11.2	65.0
QT-15	70.1	0.5	13.7	3.1		0.1	0.6	2.1	3.0	5.2	0.1	1167	6.3	267.8	43.4	0.8	13.9	36.7	98.1	109.6	1.8	-13.4	63.6	24.3	12.4	63.3
QT-62	69.6	0.5	14.0	3.5		0.1	0.6	2.4	3.3	4.7	0.2	1158	6.8	273.8	48.5	1.6	14.9	42.3	105.2	100.6	2.1	-16.5	70.2	22.7	14.0	63.3
QT-94	74.6	0.3	12.7	1.9		0.0	0.3	1.4	2.9	4.8	0.1	1241	3.4	248.1	27.0	0.6	6.7	24.3	94.2	101.1	1.0	4.3	37.1	35.6	4.8	59.6
QT-52	75.7	0.1	12.0	1.3		0.0	0.1	0.8	3.3	5.0	0.0	1260	1.5	236.2	17.8	0.4	1.2	13.7	106.8	105.7	0.1	-3.8	20.5	33.4	3.9	62.7
QT-112	75.0	0.1	12.3	1.5		0.1	0.1	0.9	3.3	5.1	0.0	1249	1.3	241.9	21.5	0.7	2.5	15.3	105.8	107.6	0.6	-2.3	25.2	32.6	4.3	63.0
QT-118	75.3	0.2	12.0	1.8		0.1	0.3	0.9	2.7	5.0	0.1	1253	2.1	234.4	24.4	0.7	6.9	15.3	88.4	107.0	1.3	8.3	33.5	38.4	3.5	58.1
Furong phase																										
FR-63	67.4	0.7	14.3	3.2	1.5	0.1	0.8	2.7	3.4	5.1	0.2	1121	9.3	281.3	63.6	1.1	19.4	47.8	109.4	107.6	3.2	-31.3	92.2	15.0	20.2	64.8
FR-33	67.1	0.7	14.4	3.0	1.3	0.1	0.8	2.6	3.4	5.4	0.2	1116	8.8	281.7	58.1	1.1	20.8	47.1	108.7	114.4	3.0	-35.7	87.7	13.0	20.4	66.6
FR-12	67.3	0.7	14.4	2.9	1.3	0.1	1.0	2.4	3.1	5.4	0.2	1120	9.3	282.7	56.0	0.8	24.8	43.3	100.7	115.5	3.0	-20.2	90.1	17.9	17.7	64.3
FR-19	70.3	0.3	13.8	2.0	0.9	0.1	0.5	1.8	3.4	6.2	0.1	1170	3.5	270.1	39.4	1.3	11.4	32.1	108.4	131.8	1.3	-34.4	54.3	15.0	15.0	70.0
GT-2-1	70.1	0.5	14.1	1.1	3.4	0.1	0.6	2.0	3.2	5.0	0.1	1167	6.1	276.6	57.7	0.8	14.1	35.5	103.9	105.9	2.0	-4.2	77.9	25.6	12.6	61.8
FR-43	68.9	0.5	14.3	2.4	0.9	0.1	0.7	2.2	3.2	5.4	0.2	1147	6.6	280.3	43.8	0.8	16.1	38.3	103.6	115.1	2.3	-15.0	66.6	20.6	13.1	66.2
GT-4-4	67.1	0.6	15.1	2.3	0.9	0.1	1.0	1.4	3.1	5.8	0.2	1117	7.0	296.6	43.3	0.8	24.3	25.7	99.7	122.7	2.5	22.8	74.7	27.6	6.9	65.6
MME																										
QTL-1-B	63.0	1.6	12.1	4.9	4.5	0.2	1.9	3.9	2.7	3.4	0.5	1049	20.4	236.6	124.7	3.0	46.9	68.7	87.1	72.6	7.5	-60.5	192.0	12.9	42.4	44.6
QTL-2	60.0	1.7	12.9	5.9	4.0	0.2	2.1	4.3	3.0	4.1	0.6	997.8	21.8	252.3	131.7	3.1	51.9	77.4	95.2	86.0	7.7	-83.7	205.3	0.4	49.0	50.6
QTL-3	59.5	1.5	14.2	2.9	5.4	0.2	1.6	3.9	3.9	6.1	0.5	989.3	18.1	278.0	106.9	2.4	40.7	69.5	125.8	129.3	6.6	-116.3	165.7	-18.3	48.3	70.0
QTL-5	56.8	1.8	15.8	6.6	3.1	0.1	1.3	3.5	3.4	3.1	0.5	945.9	21.9	310.7	131.3	1.8	31.3	62.4	109.1	66.7	6.3	10.2	184.5	19.6	27.2	53.2
QTL-7	58.5	1.8	15.3	4.9	4.2	0.2	1.2	4.1	3.7	5.4	0.5	973.7	22.8	300.5	121.0	2.1	30.5	73.3	117.8	114.6	7.0	-78.5	174.3	-8.3	42.2	66.1
QTL-8	59.3	1.7	15.4	4.8	3.9	0.2	1.2	4.2	3.8	4.8	0.5	986.5	21.5	302.7	116.1	2.1	28.8	74.5	121.0	102.1	6.6	-69.5	166.4	-4.3	39.2	65.1
QTL-9	59.0	1.8	15.1	4.6	4.4	0.2	1.2	4.1	3.6	5.0	0.5	982.0	21.9	296.4	118.4	2.1	29.8	72.9	117.1	105.9	6.6	-72.6	170.1	-5.0	40.6	64.4
QTL-10	58.5	1.7	15.0	4.6	4.2	0.1	1.2	4.2	4.5	4.9	0.5	973.9	21.2	294.6	116.9	2.0	29.5	74.9	146.5	103.4	6.8	-105.1	167.6	-16.7	46.3	70.4
QTL-11	61.7	0.6	17.6	2.7	2.2	0.1	1.8	6.1	4.6	2.0	0.3	1027	6.9	344.4	64.5	1.3	44.4	108.4	148.4	43.3	3.5	-64.1	115.8	0.6	29.8	69.6
QTL-12	55.9	1.8	16.5	4.7	5.9	0.2	1.3	4.3	5.1	3.3	0.5	929.4	22.3	322.9	139.0	2.1	33.0	76.1	165.5	69.0	6.5	-64.0	194.2	-8.6	42.2	66.3
QTL-15	59.0	2.0	14.0	6.8	3.3	0.2	2.0	4.8	3.7	2.5	0.4	982.5	24.8	274.0	135.7	2.7	50.6	84.9	119.1	52.0	5.4	-66.8	211.1	3.8	46.2	50.0
third stage (III)																										
QT-66	73.5	0.1	12.5	1.7		0.1	0.1	1.3	3.6	4.7	0.0	1222	1.1	245.4	23.6	1.7	1.5	22.8	114.9	98.7	0.1	-13.9	26.3	28.2	6.8	65.0
QT-105	74.6	0.1	13.0	1.2		0.1	0.1	0.9	3.5	5.0	0.0	1241	1.1	254.4	17.3	0.7	3.5	15.2	112.6	106.6	0.1	4.9	21.9	32.6	2.3	65.1
QT-123	75.6	0.1	12.5	1.1		0.0	0.0	0.5	2.7	6.0	0.0	1258	1.7	245.7	15.6	0.3	0.5	8.4	86.8	127.6	0.4	14.6	17.8	37.2	-0.2	63.0
Mufushan complex (Wang L.x et al., 2014 Lithos 206 147-163)																										
Diorite																										
D0801-1	50.7	1.3	13.1	7.2	2.5	0.1	10.5	8.2	2.5	1.7	0.1	843.7	16.3	257.0	101.7	1.8	260.5	146.2	80.7	36.1	1.4	-152.2	378.5	-19.0	88.5	30.5
D0725-2	53.4	1.0	12.3	5.8	2.3	0.1	10.7	8.1	2.7	1.8	0.2	888.7	12.5	241.3	82.1	1.7	265.5	144.4	87.1	38.2	2.8	-172.9	360.1	-21.7	89.0	32.7
D0725-3	50.5	1.0	10.7	6.8	2.9	0.2	13.0	9.5	2.1	1.3	0.3	840.4	12.5	209.9	96.4	2.1	322.6	169.4	67.8	27.6	3.9	-224.3	431.5	-32.6	110.8	21.8
Granodiorite																										
GD0713-1	65.7	0.6	16.1	2.7	1.5	0.1	1.7	4.1	3.2	3.2	0.2	1093	7.5	315.8	38.5	1.1	42.2	73.1	103.3	67.9	3.4	-1.6	88.2	24.6	13.8	61.6
GD0723-1	65.2	0.6	16.1	3.3	1.1	0.1	1.9	4.2	3.0	3.2	0.3	1085	7.5	315.8	46.9	1.1	47.1	74.9	96.8	67.9	3.5	1.3	101.5	25.6	15.3	59.1
Biotite monzogranite																										

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BG0701-1	71.0	0.3	15.0	1.5	0.6	0.0	0.9	2.8	3.7	3.2	0.1	1182	3.8	294.2	21.3	0.6	22.3	49.9	119.4	67.9	1.8	7.0	47.4	30.9	5.8	63.3
BG0702-1	71.2	0.3	15.1	1.4	0.5	0.0	0.7	2.6	3.9	3.3	0.1	1185	3.8	296.2	19.8	0.4	17.4	46.4	125.8	70.1	1.4	7.6	41.0	30.1	4.7	65.2
BG730	71.4	0.4	14.8	1.6	0.4	0.0	0.8	2.3	3.6	3.7	0.1	1188	5.0	290.3	22.7	0.6	19.9	41.0	116.2	78.6	1.8	13.6	47.6	32.3	4.5	63.2
BG0733-1	72.1	0.3	14.7	1.8	0.1	0.0	0.6	2.2	3.7	3.6	0.2	1200	3.8	288.3	25.5	0.6	14.9	39.2	119.4	76.4	2.1	14.1	44.2	33.0	3.9	63.1
2m leucogranite																										
LG10718-1	72.8	0.2	14.7	1.2	0.1	0.0	0.5	1.8	3.9	4.0	0.1	1212	2.5	288.3	17.2	0.6	12.4	32.1	125.8	84.9	1.3	13.4	32.1	31.7	2.2	66.2
LG10705-1	74.0	0.1	14.2	0.7	0.3	0.1	0.2	0.8	3.7	4.8	0.2	1231	1.3	278.5	10.4	0.7	5.0	14.3	119.4	101.9	2.5	28.7	16.6	36.3	-3.2	66.9
LG10710-1	72.6	0.3	14.5	1.3	0.3	0.0	0.5	1.3	3.3	4.8	0.2	1208	3.8	284.4	18.6	0.6	12.4	23.2	106.5	101.9	2.4	29.7	34.7	36.4	-0.6	64.2
LG10728-1	72.9	0.2	14.5	1.2	0.2	0.0	0.4	0.9	3.4	5.2	0.2	1213	2.5	284.4	17.2	0.6	9.9	16.0	109.7	110.4	3.2	32.2	29.6	35.9	-1.9	66.0
LG20727-1	75.0	0.0	14.1	0.1	0.4	0.0	0.1	0.9	3.9	4.6	0.0	1248	0.0	276.6	1.6	0.3	2.5	16.0	125.8	97.7	0.4	21.0	4.1	35.0	-3.5	68.5
LG20706-1	75.1	0.1	13.9	0.5	0.2	0.0	0.1	0.4	3.6	5.0	0.1	1250	1.3	272.7	7.5	0.6	2.5	7.1	116.2	106.2	1.7	36.1	11.2	39.4	-5.5	66.1
LG20711-1	74.1	0.1	14.6	0.6	0.2	0.1	0.1	0.2	4.0	4.6	0.2	1233	1.3	286.4	9.3	1.1	2.5	3.6	129.1	97.7	2.1	52.5	13.1	41.4	-8.4	67.1
Mafic enclave																										
ME0803-1	50.3	1.3	15.7	8.5	3.8	0.3	5.6	6.1	2.1	3.8	0.4	837.1	16.3	308.0	121.4	3.7	139.0	108.8	67.8	80.7	5.9	-58.0	276.6	-1.7	56.0	45.7
Micaschist																										
MS0735-2	68.2	0.9	14.1	5.1	1.0	0.1	2.0	0.6	1.4	4.6	0.1	1135	11.3	276.6	72.0	1.3	49.6	10.7	45.2	97.7	1.5	112.3	132.9	63.5	-2.3	38.8

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	SiO ₂	TiO ₂	Al ₂ O ₃	FeO	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	Si	Ti	Al	Fe	Mn	Mg	Ca	Na	K	P	A	B	Q*3	B*3	F*3	
Middle Yanshanian (150-110 Ma)																											
Guizhou mafic dyke (Jiang Y.h et al., 2006 Geol Mag 143 457-474)																											
ZHC-4	52.7	0.6	12.7	3.7	3.0	0.2	6.6	9.6	2.4	4.0	0.6	877.5	8.0	249.3	53.3	2.5	164.8	170.8	78.4	85.8	7.7		-256.5	226.0	-50.6	88.9	61.7
ZHC-13	47.9	0.6	9.1	4.9	5.6	0.2	15.2	6.7	1.0	4.2	0.5	797.5	7.0	178.1	70.0	2.1	378.2	118.6	32.3	88.5	7.5		-179.9	455.2	-25.8	110.4	15.5
Taoshan granite (Zhao Z.f et al., 2015 Chem Geol 395 11-26)																											
10SC07A	68.0	0.8	14.8		4.3	0.1	1.0	2.2	3.0	4.7	0.3	1132	9.5	289.3	0.9	1.0	24.3	38.5	97.5	100.0	3.9		14.8	34.7	30.4	2.4	67.2
10SC10	70.4	0.4	13.8		2.9	0.1	0.5	1.5	2.9	5.1	0.2	1172	5.1	270.9	0.6	0.7	13.4	26.9	93.3	107.2	2.4		16.6	19.2	34.0	-0.4	66.4
10SC06A	76.0	0.2	12.7		1.5	0.1	0.4	1.6	2.9	4.0	0.1	1265	2.6	248.9	0.9	1.0	10.7	28.4	93.6	85.1	0.8		13.5	14.2	41.5	-0.5	59.1
10SC09	77.1	0.2	11.9		1.4	0.1	0.4	1.2	2.7	3.9	0.1	1282	2.4	234.2	0.9	1.0	9.7	21.6	88.1	83.2	0.8		19.7	12.9	46.1	-2.0	55.9
10SC01A	73.3	0.2	14.0		1.4	0.1	0.3	0.9	3.2	4.8	0.1	1220	2.0	274.4	0.6	0.7	7.7	16.8	104.2	102.3	1.7		34.3	10.3	39.8	-5.4	65.6
10SC01G	72.7	0.2	14.1		1.4	0.0	0.3	1.0	3.7	4.8	0.1	1211	1.9	276.2	0.5	0.6	6.7	18.5	118.7	102.3	2.0		18.0	9.1	33.0	-2.2	69.2
10SC02B	72.1	0.2	14.8		1.4	0.0	0.4	0.8	3.0	5.5	0.2	1200	2.5	290.9	0.4	0.4	8.9	15.0	96.5	115.7	2.1		48.7	11.8	40.7	-8.0	67.4
10SC05A	73.2	0.2	14.4		1.3	0.0	0.3	0.5	3.1	5.5	0.2	1218	2.0	281.9	0.4	0.4	6.9	8.6	100.7	115.9	2.3		48.2	9.3	41.5	-8.3	66.8
10SC01N	74.9	0.1	13.7		1.1	0.1	0.3	0.6	3.0	4.7	0.1	1246	1.4	269.5	0.6	0.7	6.2	11.4	97.1	99.6	1.5		50.0	8.2	46.7	-8.8	62.2
10SC04A	74.7	0.2	13.7		1.3	0.0	0.4	1.3	3.0	4.6	0.1	1243	1.9	268.7	0.5	0.6	8.7	23.0	97.1	97.9	0.7		27.7	11.1	40.7	-3.9	63.1
Tieshan complex (Deng Z. et al., 2014 Lithos 200-201 334-354)																											
diabase																											
11JX09-3	49.5	1.0	21.3		7.1	0.1	3.5	9.7	2.6	2.3	0.4	823.8	12.0	417.8	89.0	1.6	85.9	172.6	84.2	47.8	4.9		-59.4	186.9	-7.2	40.8	66.5
11JX09-4	48.2	0.9	21.7		6.9	0.1	3.4	9.8	3.1	2.2	0.3	802.1	11.4	425.7	86.4	1.6	83.4	174.2	99.7	46.7	4.8		-69.2	181.2	-13.2	41.9	71.2
monzodiorite																											
11JX09-5	53.8	0.8	19.7		5.8	0.1	2.3	5.3	3.0	4.9	0.3	895.3	9.9	386.4	72.3	1.8	57.6	94.0	96.8	103.8	3.9		-2.1	139.7	5.7	22.1	72.1
syenite																											
11JX09-6	60.7	0.7	17.7		4.6	0.1	1.3	2.7	4.4	5.1	0.2	1010	8.9	347.2	57.2	1.3	31.0	47.3	143.3	109.1	3.1		0.3	97.1	9.1	14.7	76.2
11JX09-7	61.7	0.7	17.3		4.4	0.1	1.1	2.2	4.9	5.4	0.2	1027	8.5	339.3	55.5	1.1	26.3	39.6	158.4	114.0	3.1		-12.3	90.3	4.9	16.0	79.0
Qtz monzonite																											
11CJP07-1	64.7	0.5	15.9		4.4	0.1	2.3	3.7	3.2	4.2	0.2	1077	6.8	311.9	55.1	1.3	55.8	65.6	104.2	89.0	2.7		-12.5	117.7	18.2	20.3	61.5
monzogranite																											
11JX04-1	74.8	0.1	12.9		1.1	0.0	0.2	0.8	3.6	4.9	0.0	1245	1.6	253.0	13.4	0.6	5.0	14.6	114.9	103.6	0.4		5.3	20.0	33.1	2.0	65.0
11JX09-1	75.6	0.1	13.0		1.1	0.0	0.2	0.3	3.9	4.8	0.0	1258	1.3	255.0	14.2	0.6	4.5	5.7	126.8	101.7	0.4		15.1	19.9	34.7	0.0	65.2
12CJP06-1	71.3	0.2	14.9		1.7	0.0	0.6	1.5	3.8	4.6	0.1	1187	2.9	292.3	20.9	0.6	15.1	26.0	121.3	97.5	0.7		21.4	38.9	31.4	1.6	67.0
granite																											
11JX01-4	74.2	0.1	13.0		1.5	0.2	0.2	0.9	3.0	5.6	0.0	1235	0.8	255.0	19.3	2.8	4.0	15.3	96.5	118.5	0.3		9.4	24.0	33.9	1.8	64.3
11JX01-3	77.1	0.1	12.7		1.4	0.0	0.2	0.1	2.1	5.0	0.0	1283	1.0	249.1	17.7	0.4	4.7	2.3	67.1	107.0	0.4		70.4	23.4	57.9	-10.6	52.7
12CJP02-2	73.9	0.2	13.5		2.0	0.0	0.1	0.6	2.5	5.4	0.0	1230	2.5	264.8	25.5	0.4	2.0	11.1	81.6	114.6	0.4		46.4	30.0	45.1	-4.8	59.6
12CJP02-3	76.5	0.1	13.0		1.2	0.0	0.0	0.7	3.0	4.2	0.0	1273	1.5	255.0	14.8	0.4	0.2	12.8	97.1	88.1	0.1		44.1	16.5	48.5	-6.3	57.8
Caiyuan-Hongong-Huangtuling complex (He Z and Xu X.s 2012 Chem Geol 328 208-221)																											
Hongong syenite																											
HG06	65.8	0.7	13.9	6.4		0.2	0.3	2.0	3.9	5.6	0.2	1096	8.1	273.2	90.5	2.3	7.2	35.7	124.9	118.5	2.4		-41.4	105.9	8.5	24.4	67.1
3394	64.0	0.6	14.6	5.5		0.1	0.4	1.6	4.0	6.0	0.2	1065	7.5	285.8	77.9	1.7	10.9	28.2	130.0	127.4	2.8		-28.0	96.4	8.0	20.5	71.5
6001	66.6	0.4	14.3	3.2		0.1	0.7	2.2	3.5	6.1	0.1	1108	5.0	280.5	44.8	1.0	16.1	39.6	113.6	130.4	1.4		-42.6	65.9	8.5	18.7	72.8
3738	66.1	0.6	15.2	5.0		0.2	0.5	0.8	4.1	5.9	0.2	1100	7.1	297.2	71.1	2.5	13.4	14.6	132.0	124.8	2.5		11.1	91.6	19.3	11.7	69.1
Caiyuan																											
CY01	66.8	0.5	15.4	3.7		0.1	0.5	1.8	3.7	6.1	0.1	1112	6.0	301.9	51.7	1.4	11.9	32.1	118.1	128.7	1.7		-9.1	69.6	15.6	12.3	72.0
CY02	61.8	0.7	16.9	4.7		0.1	1.1	3.1	3.5	6.0	0.3	1028	8.5	332.3	65.0	1.6	26.3	54.6	112.0	126.8	3.7		-15.6	99.9	8.5	18.3	73.2
Huangtuling gabbro																											
HTL01	49.5	1.3	17.2	10.3		0.2	4.9	9.5	2.6	1.5	0.4	823.1	16.6	337.8	145.6	2.3	121.1	169.9	84.2	32.1	6.2		-118.4	283.3	-16.2	67.6	48.6
HTL02	49.1	1.2	17.1	10.6		0.2	5.2	9.1	2.5	1.5	0.5	816.9	14.9	335.8	148.8	2.4	128.5	162.1	81.0	32.5	6.9		-101.8	292.2	-11.9	66.0	45.9
HTL04	51.3	1.4	17.1	10.7		0.2	4.4	8.1	2.9	1.9	0.5	853.2	17.9	335.8	150.3	2.4	108.2	144.3	93.9	40.1	7.0		-86.7	276.4	-8.1	59.7	48.4
Ganhang belt basalts (Yu X.q et al., 2006 Cres 27 663-672)																											
Ji'an-Taihe basin																											
alkali basalt																											
D74	49.5	1.8	14.8	6.7	3.8	0.1	7.9	9.4	2.8	1.3	0.3	822.9	22.0	290.3	140.3	2.0	196.0	168.3	89.4	27.0	4.5		-162.7	358.4	-24.6	86.8	37.8
D74-0	53.4	1.7	15.5	5.4	4.0	0.1	6.3	8.5	2.9	0.6	0.2	888.5	21.5	303.1	125.4	1.8	157.3	151.6	93.3	13.4	3.2		-106.7	304.3	-6.2	67.6	38.6
D74-2	49.8	1.8	15.0	5.2	5.3	0.1	7.9	9.7	2.8	1.3	0.3	828.4	22.4	295.0	137.7	2.0	194.8	172.8	90.4	27.0	4.5		-167.9	354.9	-25.8	86.7	39.1
Xinjiang & Jinhua-Qinzhou																											
tholeiite																											
D90-1	49.7	1.5	16.5	2.3	7.4	0.2	6.7	9.9	2.9	0.6	0.4	827.1	18.3	323.1	124.8	2.8	166.3	176.0	93.9	13.0	4.9		-135.8	309.3	-18.4	74.6	43.8
D240	50.2	1.3	17.4	3.2	6.7	0.1	5.4	9.2	3.0	1.3	0.3	834.7	16.3	341.7	128.3	1.6	134.0	164.8	95.8	27.8	3.9		-111.5	278.5	-14.7	65.0	49.7
alkali basalt																											

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D115	51.3	1.9	17.8	0.7	8.7	0.1	2.9	6.7	3.8	3.0	1.1	853.7	24.2	348.8	119.4	1.7	72.5	118.8	121.3	64.5	15.5	-74.6	216.0	-11.8	48.6	63.2
D241	49.3	2.1	16.7	3.0	7.6	0.2	4.6	7.3	3.3	2.6	1.0	819.8	26.7	327.4	136.8	2.5	114.4	130.3	107.1	54.1	14.4	-94.6	277.8	-14.9	62.4	52.5
D119	52.6	1.5	16.9	1.4	8.1	0.1	4.8	6.3	3.5	2.1	0.5	875.0	19.0	331.3	120.8	1.3	118.9	112.0	112.3	45.0	7.2	-50.0	258.7	0.2	49.9	49.9
D127	51.8	1.7	17.6	0.7	8.6	0.1	3.3	6.1	3.6	3.3	1.0	862.4	21.4	344.4	117.3	1.6	81.4	109.5	116.5	70.7	13.9	-61.7	220.1	-7.8	46.6	61.2
Maduan granite (Li Y.j et al., 2012 Min Dep 47 763-780)																										
MD-5	74.3	0.2	12.9	0.7	0.4	0.1	0.3	1.3	2.7	5.9	0.0	1236	2.0	252.8	14.3	1.1	6.9	23.2	87.8	124.2	0.4	-5.5	23.2	30.6	4.6	64.8
MD-6	73.7	0.2	13.2	0.7	0.5	0.1	0.3	1.5	3.1	5.1	0.0	1226	2.0	258.1	15.9	1.0	6.7	26.2	100.0	108.7	0.6	-3.0	24.6	30.8	4.4	64.8
MD-7	73.8	0.2	12.9	0.8	0.3	0.1	0.3	1.3	2.5	6.4	0.0	1228	2.3	253.2	14.2	1.3	6.9	22.6	81.3	135.0	0.6	-8.4	23.4	28.9	5.3	65.8
MD-8	74.7	0.2	12.5	0.4	0.5	0.1	0.3	1.2	2.4	6.4	0.0	1243	2.3	245.6	11.5	1.0	6.7	21.4	75.8	135.0	0.4	-8.1	20.5	31.0	4.8	64.3
MD-9	75.2	0.2	12.2	0.6	0.8	0.1	0.3	1.2	2.6	5.3	0.0	1251	2.3	239.1	18.4	1.1	7.2	22.1	82.3	112.3	0.3	0.3	27.9	36.0	4.2	59.8
MD-10	74.6	0.2	12.5	0.5	0.5	0.1	0.3	1.3	2.5	6.0	0.0	1242	2.3	244.2	14.1	1.0	6.5	23.0	79.4	127.0	0.4	-8.1	22.8	31.5	5.1	63.3
Xiangshan rhyodacite (Yang S.y et al., 2010 ScChina ES 53 1411-1426)																										
rhyodacite																										
XS-30-1	69.6	0.4	13.7	1.2	3.5	0.1	0.4	1.9	3.7	3.1	0.2	1157	5.4	269.3	60.3	1.6	10.4	33.7	120.7	66.0	2.4	15.2	76.1	33.8	8.7	57.5
X9-18	68.5	0.4	14.8	0.4	3.1	0.1	0.9	1.7	2.9	5.1	0.2	1140	4.9	290.7	43.7	1.0	21.6	30.5	94.2	107.6	2.5	27.9	70.2	32.9	5.2	61.9
X9-19	68.6	0.4	14.8	0.4	3.3	0.1	0.6	1.9	2.8	5.0	0.2	1141	5.5	289.9	46.9	1.0	14.4	33.0	89.4	105.5	2.7	29.0	66.8	34.3	4.4	61.2
rhyodacite porph																										
XS-29-17	71.4	0.5	16.8	0.8	0.9	0.1	0.2	0.8	0.7	4.4	0.2	1189	5.9	329.9	21.7	0.7	6.0	14.4	23.9	92.6	2.4	184.6	33.6	86.5	-33.1	46.5
XS-30-3	67.8	0.4	14.3	1.8	0.9	0.1	0.6	3.5	2.3	3.4	0.2	1128	5.3	280.1	37.0	1.1	15.6	61.5	74.9	72.8	2.1	9.4	57.8	35.5	7.2	57.3
X9-20	67.8	0.4	14.4	1.9	1.7	0.1	0.9	2.0	3.2	4.9	0.2	1128	4.6	281.5	46.4	1.6	23.1	35.0	102.3	104.5	2.5	4.8	74.1	26.4	10.5	63.2
X9-25	68.6	0.4	14.6	1.5	1.8	0.1	0.9	1.4	3.6	4.8	0.2	1142	5.0	285.6	43.9	1.0	21.6	25.1	115.5	101.5	2.4	18.3	70.5	29.0	7.1	63.9
X9-26	69.0	0.4	14.3	1.2	2.4	0.1	0.8	0.6	3.0	5.0	0.2	1148	4.8	279.6	46.5	1.0	18.9	10.5	96.8	105.5	2.3	56.2	70.1	41.9	-0.6	58.7
X9-27	67.5	0.4	15.1	1.4	1.8	0.1	0.9	1.8	3.7	4.9	0.2	1123	4.4	296.2	41.6	1.1	21.6	31.6	119.4	104.5	2.7	9.2	67.6	24.1	8.5	67.4
Sanqingshan-Damaoshan complex (Sun F.j et al., 2015 JAsES 9809-224)																										
Kfs-granite																										
10sjs162	76.1	0.1	12.2		1.4	0.0	0.0	0.5	3.3	5.4	0.0	1267	1.5	239.5	17.7	0.4	0.7	9.3	105.5	114.9	0.3	0.6	19.9	33.5	2.9	63.5
10sjs163	74.9	0.1	13.2		1.1	0.0	0.0	0.6	3.9	5.3	0.0	1247	1.4	258.5	13.3	0.4	0.7	10.7	126.2	113.2	0.4	-2.2	15.4	28.2	2.8	69.0
10sjs165	76.4	0.2	12.2		1.5	0.0	0.1	0.3	3.1	5.1	0.0	1271	2.1	239.5	19.3	0.6	3.2	6.1	101.3	107.2	0.6	18.8	24.6	40.0	0.0	60.0
SQS01	75.8	0.1	12.3		1.4	0.0	0.1	0.1	3.3	4.9	0.0	1261	1.4	241.7	18.0	0.6	2.7	2.1	106.8	103.4	0.3	27.2	22.1	41.5	-2.1	60.6
SQS02	73.6	0.1	11.9		1.2	0.0	0.1	0.2	3.2	5.0	0.0	1224	1.1	233.8	14.5	0.4	3.0	4.3	102.6	105.3	0.1	17.3	18.6	38.6	-0.7	62.0
SQS03	73.9	0.1	12.0		1.2	0.0	0.1	0.7	3.4	4.7	0.0	1230	1.3	234.4	15.4	0.6	2.7	11.8	110.0	100.0	0.1	0.8	19.4	34.0	2.9	63.2
SQ04	76.0	0.1	11.9		1.2	0.0	0.1	0.5	3.5	4.6	0.0	1264	1.3	232.6	15.0	0.4	2.2	9.5	112.0	97.9	0.3	3.9	18.5	36.3	2.1	61.7
SQ02	76.9	0.0	12.6		0.4	0.0	0.1	0.3	3.5	4.7	0.0	1280	0.3	247.5	5.1	0.0	1.5	5.9	113.6	99.6	0.1	22.6	6.9	40.4	-3.5	63.1
SQ05	77.5	0.2	11.1		1.6	0.0	0.2	0.9	2.6	4.3	0.0	1290	2.1	216.9	19.5	0.4	4.2	16.8	83.9	92.1	0.3	7.4	25.9	43.7	2.5	53.8
SQ07	80.1	0.2	9.2		2.0	0.0	0.3	0.7	1.9	3.9	0.0	1332	2.9	180.9	25.5	0.4	6.2	11.9	61.6	83.2	0.4	12.1	34.6	53.7	2.9	43.3
SQ08	78.4	0.1	10.8		1.3	0.0	0.2	0.5	2.7	4.1	0.0	1305	1.4	211.3	15.7	0.4	4.0	8.0	87.1	87.9	0.3	20.2	21.0	48.5	-0.9	52.3
10sjs172	74.2	0.3	12.5		2.2	0.1	0.1	1.0	3.6	5.2	0.0	1234	3.1	246.0	27.3	0.7	3.2	17.8	114.6	109.3	0.3	-13.6	33.7	27.3	7.8	64.9
10sjs173	75.7	0.1	12.4		1.2	0.0	0.1	0.8	3.5	5.2	0.0	1260	1.6	242.6	15.0	0.4	1.7	15.0	113.3	109.3	0.3	-9.9	18.4	30.0	4.8	65.2
10sjs175	77.1	0.2	11.4		1.7	0.0	0.1	0.7	3.1	4.8	0.0	1283	2.0	223.2	21.5	0.4	1.7	12.8	99.1	102.8	0.4	-4.3	25.3	36.5	4.7	58.8
10sjs177	76.3	0.1	12.1		1.3	0.0	0.0	0.6	3.6	5.2	0.0	1270	1.4	237.2	15.9	0.4	1.0	10.7	114.6	109.3	0.1	-8.1	18.3	31.2	4.4	64.4
syenogranite																										
13SQS03	75.9	0.1	12.8		1.1	0.0	0.1	0.2	3.6	4.7	0.0	1263	1.0	250.3	13.8	0.6	2.0	3.0	115.2	100.4	0.1	28.6	16.8	40.6	-3.2	62.6
13SQS04	76.5	0.1	12.4		1.2	0.0	0.1	0.1	3.5	4.6	0.0	1273	1.0	244.0	14.8	0.6	2.2	2.0	113.6	98.5	0.3	28.0	18.0	41.8	-2.9	61.1
13SQS05	76.5	0.1	12.2		1.3	0.0	0.1	0.4	3.7	4.2	0.0	1272	1.0	240.1	16.4	0.6	1.5	7.3	119.7	90.0	0.1	15.7	18.9	39.1	-0.3	61.2
Dazhou high-Zr rhyolite (Yang S.y et al., 2013 JAsES 74 303-315)																										
DZ-6	74.3	0.3	13.0	1.2	1.0	0.1	0.1	0.3	2.4	6.7	0.1	1236	3.6	255.4	28.8	1.7	1.5	5.7	77.4	141.4	0.8	25.1	34.0	37.5	0.1	62.4
DZ-8	75.5	0.3	12.4	1.4	0.8	0.1	0.1	0.6	2.3	6.0	0.1	1256	3.4	242.5	29.9	1.1	1.2	10.0	72.9	126.3	1.0	23.3	34.5	41.3	0.6	58.1
DZ-9	74.8	0.3	12.8	1.1	1.8	0.1	0.1	0.5	2.0	5.3	0.1	1245	3.6	250.7	37.4	1.1	1.5	9.5	64.9	112.1	1.0	54.8	42.5	51.4	-4.5	53.1
DZ-10	75.9	0.3	12.5	1.3	1.3	0.1	0.1	0.5	1.8	5.6	0.1	1263	3.3	245.4	33.6	1.1	1.5	8.7	57.8	118.9	1.1	51.3	38.3	51.7	-4.5	52.8
DZ-11	76.1	0.3	12.2	1.2	1.3	0.1	0.0	0.5	2.1	5.6	0.1	1267	3.4	239.5	32.4	1.4	1.0	8.7	68.1	117.8	1.1	36.1	36.8	47.1	-1.6	54.6
DZ-15	73.9	0.3	12.9	0.4	2.9	0.0	0.0	0.5	1.8	6.4	0.0	1230	3.5	252.1	41.9	0.3	1.0	8.9	57.1	136.7	0.6	40.4	46.3	44.6	-1.0	56.4
DZ-17	74.8	0.3	12.7	0.3	2.0	0.0	0.0	0.5	1.6	6.5	0.0	1245	3.6	249.7	28.3	0.3	1.0	9.3	50.7	137						

Chapter 1

Rucheng basalt (Meng L.f et al., 2012 Lithos 132-133 127-140)

07SC10-4	50.8	1.5	15.6	10.1	0.1	8.2	11.4	2.0	0.2	0.1	845.4	18.5	306.0	126.5	2.0	202.7	203.3	64.2	4.2	2.0	-169.0	347.7	-20.2	85.8	34.5
07SC10-5	49.6	1.5	16.0	10.7	0.1	8.4	10.9	2.1	0.6	0.1	825.4	19.1	313.8	134.0	2.0	207.2	194.4	66.8	12.3	2.0	-154.0	360.4	-19.2	84.6	34.6
07SC10-6	50.2	1.5	15.7	11.0	0.2	8.3	10.9	1.7	0.3	0.1	835.4	19.1	308.0	137.8	2.3	206.9	194.4	56.1	5.5	2.0	-142.4	363.9	-13.4	83.3	30.1
07SC10-7	50.6	1.5	15.8	10.7	0.2	8.0	10.8	2.1	0.3	0.1	842.1	18.9	309.9	134.0	2.1	198.0	192.6	66.8	5.3	2.0	-147.3	350.9	-15.6	82.1	33.4
07SC10-8	50.4	1.5	15.9	10.8	0.2	8.4	10.7	1.8	0.2	0.1	838.7	18.8	311.9	135.3	2.3	208.4	190.8	57.8	4.2	2.0	-131.7	362.5	-10.8	80.8	29.9
07SC10-9	50.0	1.7	15.5	11.6	0.2	7.8	10.7	2.2	0.2	0.2	832.1	20.9	304.0	145.3	2.3	193.3	190.8	71.6	4.5	2.1	-153.7	359.5	-17.9	84.9	33.0

Suzhou complex (Gao Y.y 2015 PhD dissertation)

Hn-porphyrritic gr																									
09YY101	71.4	0.4	13.7	2.3	0.1	0.8	1.6	3.6	5.0	0.1	1188	4.8	267.8	32.4	1.0	20.3	29.2	117.1	106.8	1.0	-14.7	57.5	23.0	11.6	65.4
09YY102	72.4	0.3	13.7	2.4	0.1	0.6	1.3	3.7	5.0	0.1	1205	4.1	268.1	32.9	0.7	14.9	22.6	117.8	106.2	1.0	-1.1	52.0	27.3	8.0	64.7
09YY103	70.2	0.5	14.1	3.2	0.1	0.9	1.6	3.7	4.9	0.1	1168	5.8	277.0	44.3	1.0	21.6	28.5	119.4	103.4	1.4	-2.9	71.7	24.5	11.4	64.1
09YY105	78.2	0.1	11.9	1.1	0.0	0.1	0.5	3.4	4.6	0.0	1301	1.1	232.6	15.1	0.3	1.5	8.2	109.4	96.6	0.0	10.2	17.8	39.8	0.7	59.6
09YY107	73.4	0.2	13.6	1.9	0.1	0.4	0.9	3.6	5.5	0.0	1221	2.9	265.8	26.3	1.1	9.2	16.8	116.8	116.1	0.6	-0.7	38.3	27.4	5.9	66.7
09YY108	73.6	0.2	13.5	1.8	0.1	0.4	0.9	3.5	5.3	0.0	1224	2.8	265.6	24.7	1.3	9.2	16.4	112.9	113.2	0.6	6.7	36.7	30.3	4.2	65.5
09YY109	73.1	0.3	13.6	2.1	0.1	0.5	1.1	3.6	5.2	0.0	1217	3.1	266.0	28.8	1.0	11.7	18.9	115.5	109.3	0.6	3.3	43.6	29.1	5.9	65.0
09YY110	73.6	0.2	13.3	1.6	0.1	0.4	1.2	3.6	5.1	0.0	1225	2.8	260.9	21.8	0.7	9.7	21.8	114.9	108.3	0.4	-5.8	34.3	27.9	6.3	65.8
09YY111	75.9	0.2	13.1	0.9	0.1	0.3	0.7	3.8	4.7	0.0	1263	2.1	257.4	12.6	1.0	7.2	13.2	122.6	99.8	0.3	8.6	22.0	33.7	1.6	64.7
Bt-Kfs gr																									
09YY85	74.5	0.2	13.0	1.9	0.1	0.2	0.7	3.9	5.0	0.0	1239	2.6	254.4	26.7	0.7	5.0	12.5	124.9	107.0	0.4	-2.4	34.3	28.8	5.6	65.6
09YY87	73.9	0.2	13.3	2.2	0.1	0.2	0.7	3.9	5.0	0.0	1229	2.9	260.3	30.0	0.7	4.7	12.3	125.5	106.6	0.4	3.6	37.6	29.4	5.0	65.7
09YY89	74.8	0.2	12.9	2.1	0.1	0.3	1.0	4.0	4.8	0.0	1244	3.0	252.3	28.9	0.7	6.2	16.9	127.5	101.9	0.6	-11.0	38.1	27.2	7.9	65.0
09YY90	74.6	0.2	13.4	1.5	0.0	0.2	0.6	4.0	5.1	0.0	1242	2.0	262.7	20.3	0.4	4.5	11.4	129.4	108.7	0.3	1.7	26.8	28.7	3.7	67.7
09YY91	76.3	0.1	12.9	1.3	0.0	0.1	0.3	4.3	4.3	0.0	1270	0.8	253.6	17.4	0.6	2.7	6.1	138.1	90.9	0.0	12.5	20.9	34.5	0.7	64.8
09YY92	73.6	0.2	13.6	1.7	0.0	0.3	0.9	3.3	5.2	0.0	1225	2.8	265.8	23.9	0.6	8.4	15.2	105.2	110.4	0.4	19.9	35.1	35.2	1.4	63.5
09YY93	76.4	0.0	13.2	0.8	0.0	0.2	0.2	3.8	4.7	0.0	1272	0.5	258.9	10.6	0.6	4.7	3.2	123.3	99.8	0.0	29.5	15.8	39.5	-3.4	64.0
09YY98	77.2	0.1	12.9	0.4	0.0	0.1	0.3	3.9	5.0	0.0	1284	0.6	253.8	5.1	0.1	2.0	5.7	127.1	106.8	0.0	8.5	7.8	33.6	-0.5	66.9
09YY99	74.8	0.2	13.2	1.4	0.0	0.2	0.9	3.9	5.0	0.0	1244	2.3	258.9	19.9	0.4	5.5	15.2	125.8	106.6	0.3	-3.8	27.6	28.3	4.9	66.8
albitized Kfs-gr																									
09YY95	74.8	0.2	13.1	1.5	0.0	0.3	0.8	3.6	5.1	0.0	1245	2.5	256.0	20.6	0.6	6.2	14.6	117.5	108.3	0.4	1.0	29.3	30.6	4.2	65.2
09YY96	73.8	0.2	13.6	1.8	0.0	0.2	0.7	4.1	5.3	0.0	1228	2.4	266.6	24.5	0.6	4.5	12.3	131.7	112.3	0.3	-2.0	31.3	26.1	5.1	68.9
09YY97	77.4	0.1	12.1	1.0	0.0	0.1	0.5	3.6	4.8	0.0	1288	1.1	237.0	13.8	0.3	2.7	9.5	116.2	100.8	0.0	1.0	17.6	35.1	2.5	62.4
09YY100	77.0	0.1	12.7	0.8	0.0	0.1	0.6	3.8	4.5	0.0	1281	1.1	248.9	11.3	0.3	3.0	10.5	123.3	96.4	0.0	8.2	15.4	35.5	0.7	63.9
09YY112	74.7	0.2	13.0	2.0	0.1	0.2	0.7	3.8	4.8	0.0	1243	2.8	254.6	27.7	0.7	6.0	12.1	122.9	102.5	0.4	4.9	36.4	31.6	4.5	63.9
09YY113	75.1	0.2	12.9	1.6	0.0	0.2	0.7	3.8	5.1	0.0	1250	2.3	252.1	21.7	0.6	5.2	12.5	122.0	107.6	0.3	-2.5	29.1	29.7	4.9	65.4

Baijuehuajian granite (Wong J et al., 2009 Lithos 112 289-305)

06ZFR01	76.2	0.1	11.5	2.8	0.1	0.1	0.6	2.4	3.9	0.0	1268	0.8	226.2	35.4	1.7	2.5	10.0	76.2	81.7	0.0	48.3	38.7	55.5	-3.9	48.4
06ZFR02	75.2	0.1	12.4	1.9	0.0	0.1	1.0	3.0	5.0	0.0	1252	1.6	244.0	23.7	0.4	2.7	18.0	98.1	106.8	0.0	3.1	28.0	35.0	3.7	61.3
06ZFR08	76.9	0.1	11.6	1.2	0.0	0.1	0.6	4.0	4.1	0.0	1280	1.3	228.3	15.3	0.6	1.5	9.8	128.8	86.4	0.0	-6.5	18.0	33.8	4.0	62.1
07ZFR01	76.2	0.1	11.7	1.4	0.0	0.1	0.7	3.7	4.2	0.0	1268	1.5	229.5	18.0	0.3	3.0	11.6	117.8	89.8	0.0	-1.3	22.5	35.5	3.7	60.8
07ZFR02	76.6	0.1	11.8	1.3	0.0	0.1	0.5	3.9	4.0	0.0	1275	1.4	230.5	16.7	0.3	2.7	9.5	125.5	85.8	0.0	0.3	20.8	35.7	3.1	61.2
07ZFR03	77.3	0.1	12.0	1.4	0.0	0.1	0.6	3.9	4.0	0.0	1286	1.4	234.4	16.9	0.3	2.5	10.2	124.9	84.3	0.0	4.9	20.8	37.2	2.2	60.6
07ZFR04	77.3	0.1	12.4	0.6	0.0	0.1	0.5	3.5	5.0	0.0	1286	0.9	242.6	6.9	0.1	1.7	9.1	112.9	106.6	0.0	4.9	9.5	35.4	0.5	64.1
07ZFR05	77.1	0.1	12.0	1.2	0.0	0.1	0.6	3.5	4.6	0.0	1284	1.3	235.0	15.5	0.3	2.7	10.5	111.3	97.2	0.0	5.4	19.5	37.3	1.9	60.8
07ZFR06	76.9	0.1	12.0	0.9	0.0	0.0	0.5	3.7	4.6	0.0	1279	1.3	236.0	11.8	0.1	0.0	9.6	119.4	97.9	0.0	-0.6	13.0	34.5	2.1	63.4
07ZFR07	77.1	0.1	11.9	1.4	0.0	0.1	0.6	3.6	4.3	0.0	1284	1.3	232.4	17.0	0.3	2.2	9.8	116.5	90.4	0.0	5.9	20.5	37.9	1.9	60.2
07ZFR08	77.1	0.1	12.1	1.4	0.0	0.1	0.6	3.5	4.4	0.0	1283	1.3	237.5	17.9	0.3	2.2	10.0	112.9	92.6	0.0	12.1	21.4	39.2	0.8	60.0
07ZFR09	76.7	0.1	11.8	1.6	0.0	0.1	0.6	3.5	4.2	0.0	1276	1.5	231.7	19.7	0.3	2.2	10.0	112.9	89.6	0.0	9.2	23.4	39.0	1.7	59.2
07ZFR10	77.7	0.1	11.5	0.8	0.0	0.1	0.6	4.0	4.2	0.0	1292	1.3	225.6	9.8	0.3	1.2	10.0	128.8	90.0	0.0	-13.2	12.3	32.4	4.5	63.1
07ZFR11	77.3	0.1	11.5	1.1	0.0	0.1	0.6	3.7	4.3	0.0	1287	1.1	225.0	14.3	0.6	2.2	10.0	119.1	91.1	0.0	-5.1	17.6	35.3	3.7	60.9
07ZFR12	76.9	0.1	12.0	1.1	0.0	0.1	0.5	3.8	4.4	0.0	1280	1.3	236.2	13.5	0.1	1.7	9.5	122.0	93.4	0.0	1.9	16.5	35.4	2.1	62.5
07ZFR13	76.6	0.1	12.2	1.1	0.0	0.1	0.5	3.6	4.5	0.0	1275	1.3	238.7	13.7	0.1	1.7	9.6	117.5	96.2	0.0	5.8	16.6	36.2	1.4	62.4

Kuokeng monzogabbro (Li B t al., 2015 Tectonophysics 661 136-155)

GT-07	53.2	1.1	15.6	5.9	0.8	0.1	5.5	5.9	2.8	3.6	0.4	885.5	13.3	305.8	91.9	1.6	135.7	104.5	90.0	77.1	6.1	-70.3	240.9	-4.2	52.1	52.0
GT-08	47.7	1.3	14.6	6.8	1.6	0.2	7.8	8.5	2.6	2.6	0.5	794.1	16.6	286.8	114.8	2.3	193.1	151.9	82.9	56.1	7.5	-156.1	324.5	-27.9	83.2	44.7

Chapter 1

Daiqianshan-Pingtang gabbro (Chen C.y et al., 2004 Trs. Roy. Sc. Ed 95 237-248)

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Fujian gabbros (Wang Z.h et al., 2002 CMP 144 230-240)																											
Pingtan group																											
4.3	48.1	0.9	17.4	6.9	5.2	0.2	6.4	10.5	2.3	0.7	0.1	800.0	11.0	341.1	159.8	3.0	158.8	186.9	75.2	14.9	1.4	-122.7	329.7	-15.6	74.9	40.8	
13.3	52.4	0.8	18.3	7.8	1.2	0.1	5.4	9.3	2.9	0.8	0.2	872.0	10.4	358.0	123.6	1.6	134.0	165.8	92.0	17.0	2.1	-82.6	268.0	-4.3	56.6	47.6	
24-30	49.1	1.0	18.0	6.7	4.3	0.1	6.6	10.5	2.0	0.7	0.1	816.6	12.3	353.9	146.7	1.8	163.5	187.8	65.2	14.4	1.4	-101.3	322.5	-8.7	69.0	39.7	
24-31	52.1	0.9	18.2	6.5	2.3	0.1	5.9	9.0	2.4	0.8	0.1	866.5	10.6	357.8	118.2	1.3	145.2	159.8	78.7	16.6	1.4	-57.1	274.0	3.5	53.1	43.4	
24-32	48.5	1.0	17.7	7.1	3.7	0.2	6.5	10.5	2.1	0.5	0.1	807.1	12.0	347.2	144.9	2.1	161.0	186.5	69.1	11.5	1.4	-106.4	318.0	-10.3	70.2	40.2	
17.3.2	51.7	0.9	18.2	6.9	3.9	0.2	3.8	9.0	3.2	0.8	0.4	861.0	10.9	357.2	143.8	2.5	94.0	160.8	102.3	16.6	5.5	-83.3	248.8	-6.2	54.7	51.6	
17.3.1	52.1	1.2	17.6	6.3	3.8	0.2	3.8	9.1	3.3	0.9	0.4	867.0	15.4	344.6	136.1	2.1	95.3	163.0	106.8	20.0	5.2	-108.1	246.8	-12.4	59.3	53.2	
17.1.2	52.5	1.1	17.5	6.3	3.9	0.2	3.8	8.6	3.0	1.2	0.3	873.7	13.5	343.3	136.1	3.0	95.3	153.9	98.1	25.1	4.4	-87.7	244.9	-6.3	55.2	51.1	
Dongcai to Taohuashan grp																											
Dongcai																											
DC1	40.9	1.7	13.5	9.5	10.8	0.4	8.2	11.5	1.2	0.3	0.0	680.0	21.5	265.4	267.0	5.2	203.5	204.2	37.1	6.2	0.4	-186.2	492.0	-30.3	116.6	13.7	
DC2	40.8	1.7	15.2	8.9	10.3	0.4	7.3	12.1	1.1	0.3	0.0	678.1	21.5	298.5	251.9	6.2	180.4	215.4	35.8	6.2	0.3	-174.2	453.8	-28.9	107.8	21.1	
DC3	42.6	1.4	19.1	7.0	8.3	0.3	5.4	12.8	1.3	0.3	0.0	708.6	16.9	374.1	201.4	3.7	134.7	227.4	41.9	6.2	0.3	-128.7	353.0	-19.9	81.3	38.6	
DC4	43.5	1.1	22.4	5.4	6.4	0.2	3.9	13.7	1.3	0.3	0.0	723.9	14.1	440.0	155.5	2.4	95.8	244.1	42.6	7.2	0.3	-98.1	265.4	-14.9	61.0	53.9	
Donghueshan																											
C430-1	45.6	0.8	16.9	6.3	6.1	0.2	7.9	12.5	1.3	0.5	0.0	759.2	9.6	332.3	163.5	2.1	195.5	222.4	41.6	10.0	0.1	-164.0	368.6	-23.9	89.8	34.1	
C430-2	43.1	0.9	18.4	5.8	6.4	0.1	7.5	14.2	1.3	0.3	0.0	716.4	10.6	360.5	161.0	1.8	185.6	253.6	42.6	6.2	0.6	-195.3	357.3	-35.7	94.2	41.5	
Taohuashan																											
C430-5	42.2	1.2	16.3	8.5	8.9	0.2	7.1	12.6	1.1	0.4	0.0	702.9	14.5	320.1	229.4	2.4	175.7	224.3	36.5	8.5	0.4	-173.5	419.6	-28.5	100.8	27.7	
Quinglanshan																											
D58-6	44.8	0.8	17.0	6.8	4.9	0.2	9.2	12.7	1.3	0.3	0.0	745.5	10.3	333.3	154.8	2.5	228.8	225.9	41.6	6.4	0.3	-166.6	393.9	-24.8	93.5	31.3	
Daiqianshan group																											
H53-5(1)	38.8	1.6	19.5	6.5	8.3	0.2	6.6	14.0	1.6	0.3	0.5	645.9	20.3	381.7	194.4	2.3	163.8	250.0	51.6	6.4	6.9	-176.3	378.5	-37.6	94.9	42.8	
H53-5(2)	38.7	1.6	20.5	6.0	8.0	0.2	5.7	14.5	1.6	0.3	0.6	644.7	19.6	402.1	184.5	2.1	142.4	258.2	51.3	6.4	8.9	-172.0	346.6	-37.8	89.3	48.5	
H53-5(3)	38.7	1.6	19.5	6.6	8.9	0.2	7.0	13.4	1.5	0.3	0.3	644.5	19.8	382.9	202.3	2.4	173.4	238.6	48.7	6.4	4.4	-149.4	395.5	-30.3	91.9	38.4	
H53-5(4)	39.4	1.5	19.1	6.7	8.8	0.2	7.5	12.9	1.7	0.2	0.1	655.4	18.4	373.9	203.4	2.3	185.1	230.6	53.9	5.1	1.3	-146.2	406.9	-28.7	92.5	36.1	
Pu4-4	39.1	0.7	16.7	8.7	10.4	0.1	7.4	13.6	1.0	0.5	0.1	649.9	9.3	328.0	250.9	1.7	182.6	241.8	33.6	9.8	0.8	-198.9	442.8	-38.7	110.0	28.6	
Pu4-6	39.7	1.4	15.5	8.5	10.0	0.1	8.1	13.9	1.4	0.4	0.1	661.2	17.4	304.0	242.9	1.8	202.0	248.2	43.9	9.1	0.7	-245.4	462.2	-49.4	121.0	28.4	
Pu4-8	39.6	2.4	18.6	8.0	6.9	0.1	7.7	12.6	2.3	0.5	0.1	659.7	29.4	364.8	197.7	1.7	191.1	224.0	72.6	9.8	1.7	-165.5	418.2	-35.1	96.9	38.3	
Pu4-7	40.0	1.5	15.4	8.4	10.1	0.1	8.2	13.8	0.9	0.4	0.1	666.2	18.4	301.1	243.1	1.7	204.5	246.8	29.7	9.1	0.8	-231.3	465.9	-43.8	119.4	24.4	
31-261	39.6	2.1	18.6	7.9	8.0	0.1	7.5	12.0	1.8	0.2	0.2	659.7	25.9	364.8	209.4	2.0	186.1	214.0	57.8	4.7	3.4	-125.5	421.4	-22.9	90.8	32.2	
G95-2-2	40.3	2.0	19.7	5.9	7.6	0.2	7.0	13.3	1.7	0.3	0.1	670.0	24.7	385.4	177.6	2.4	173.2	237.9	54.2	5.7	1.3	-150.2	375.4	-29.6	88.3	41.3	
Quanzhou-Huaco (Li Z et al., 2012 GeolMag 149 459-482)																											
Quanzhou																											
Hn-gabbro																											
QZ-1	46.7	0.7	15.7		13.1	0.2	11.5	10.3	0.9	0.3	0.1	777.2	8.9	307.6	164.6	2.7	284.9	183.1	27.8	5.3	1.1	-91.7	458.3	-0.5	87.7	12.8	
QZ-2	42.9	0.2	18.3		9.9	0.1	14.7	11.6	0.7	0.2	0.1	713.9	2.0	358.8	123.7	1.8	364.5	206.0	21.6	4.0	1.0	-78.8	490.3	-2.8	88.2	14.6	
QZ-3	47.0	0.8	20.6		9.9	0.1	7.7	10.5	1.4	1.1	0.1	782.7	9.9	403.3	124.1	1.6	190.6	186.7	44.2	22.5	0.8	-36.8	324.6	4.6	56.4	39.1	
QZ-4	44.8	1.2	17.9		15.9	0.1	6.7	10.5	1.2	0.8	0.1	745.9	14.5	351.3	199.0	1.8	165.3	187.4	38.7	16.1	1.3	-78.4	378.8	-3.9	74.7	29.2	
QZ-5	44.4	0.8	22.1		12.2	0.1	6.1	11.1	1.4	0.6	0.1	739.6	9.6	433.1	153.3	1.6	150.9	197.2	44.2	12.5	1.1	-18.1	313.8	6.4	51.7	41.9	
QZ-6	43.5	1.3	16.7		18.2	0.2	6.4	11.5	1.3	0.4	0.1	723.4	16.1	327.8	227.4	2.7	159.6	205.6	42.9	9.3	1.7	-135.7	403.1	-18.7	90.5	28.1	
granite																											
QZ-7	75.9	0.1	12.9		0.7	0.1	0.2	0.9	3.4	5.1	0.1	1264	1.5	253.2	8.6	0.7	4.5	15.5	110.4	108.7	0.8	3.1	14.6	33.2	1.6	65.2	
QZ-8	70.6	0.2	15.4		1.9	0.1	0.5	2.3	3.9	4.0	0.1	1174	3.0	302.5	23.3	0.7	12.9	41.4	125.8	84.5	1.7	9.4	39.2	27.9	4.1	68.0	
Huaco																											
Hn-gabbro HC-2	44.5	1.0	17.7		13.6	0.2	7.6	13.0	1.3	0.4	0.1	739.9	13.0	347.4	170.8	2.3	187.3	231.5	42.3	8.5	1.7	-166.3	371.2	-26.3	90.3	36.0	
Hn-gabbro HC-3	45.4	1.0	17.5		14.1	0.2	6.7	12.7	1.4	0.6	0.1	755.9	12.6	344.1	177.0	2.1	166.3	227.0	44.9	12.7	1.7	-167.5	355.9	-26.3	88.2	38.1	
granite HC-4	72.8	0.2	13.8		1.7	0.1	0.5	1.8	4.0	4.5	0.1	1212	2.8	270.3	21.4	0.8	11.9	31.4	127.5	95.8	1.4	-15.7	36.1	23.9	8.5	67.5	
Putuoshan granite (Zhao J.I et al., 2016 Lithos 240 16-33)																											
12PT-3	75.9	0.2	13.3		1.1	0.1	0.4	0.9	4.2	4.4	0.0	1263	2.0	260.5	1.3	1.4	9.9	15.5	136.2	92.8	0.4	0.5	13.2	30.7	1.9	67.4	
12PT-5	77.5	0.2	12.6		1.1	0.1	0.4	0.5	4.4	4.1	0.0	1290	2.3	247.0	1.3	1.4	8.9	9.1	140.4	86.6	0.3	1.8	12.4	33.4	1.5	65.1	
03P-10	76.9	0.1	12.5		0.7	0.1	0.0	0.5	4.0	4.8	0.0	1279	1.5	244.6	0.6	0.7	0.2	8.0	127.9	101.9	0.1	-3.1	2.4	31.6	1.0	67.5	
03P-11	77.5	0.1	12.4		0.7	0.1	0.0	0.4	3.9	4.7	0.0	1290	1.3	243.6	0.6	0.7	0.2	7.1	125.2	99.6	0.1	4.6	2.1	34.9	-0.6	65.6	
03P-13	76.6	0.1	12.5		0.9	0.1	0.1	0.6	4.1	4.5	0.0	1275	1.6	244.8	0.6	0.7	1.2	11.4	131.7	95.5	0.1	-5.2	3.5	31.3	1.6	67.1	
03P-14-3	77.1	0.1	12.6		0.9	0.0	0.0	0.4	4.2	4.7	0.0	1284	1.5	247.0	0.4	0.4	0.7	7.3	135.5	99.2	0.1	-2.3	2.6	31.4	0.9	67.8	
03P-15	75.8	0.1	12.7		0.9	0.1	0.1	0.8	4.1	4.8	0.0	1261	1.6	249.3	0.8	0.8	1.5	14.4	131.0	102.1	0.1	-12.7	3.9	27.7	3.1	69.2	
03P-17	76.9	0.1	12.8		0.8	0.1	0.1	0.6	4.1	4.7	0.0	1280	1.4	251.5	1.0	1.1	1.7	10.0	133.3	100.2	0.1	-2.0	4.1	31.1	1.0	67.0	

Chapter 1

Putuoshan complex (Gao Y.v 2015 PhD dissertation)

alk-gr																																	
09YY134	77.3	0.1	12.8	0.0		0.1	0.1	0.4	4.0	4.7	0.0	1287	1.3	251.5	0.0	0.7	2.7	6.2	130.4	98.7	0.0						9.9	4.0		35.0	-1.4	66.4	
09YY135	77.4	0.1	12.8	0.0		0.1	0.1	0.3	3.8	4.7	0.0	1289	1.4	251.3	0.0	0.8	3.0	6.1	121.3	100.2	0.0						17.6	4.4		38.0	-2.8	64.8	
09YY137	75.4	0.2	13.7	0.9		0.1	0.2	0.5	3.8	4.7	0.0	1255	2.1	267.9	13.1	0.7	5.7	8.4	122.9	100.2	0.3						28.0	20.9		37.6	-2.4	64.8	
09YY138	78.1	0.1	12.4	0.6		0.0	0.1	0.3	3.9	4.6	0.0	1300	1.0	243.4	8.9	0.4	1.7	5.0	124.2	97.9	0.3						11.3	11.6		37.2	-0.5	63.3	
09YY142	75.6	0.2	13.2	0.8		0.0	0.3	0.5	3.7	5.3	0.0	1258	2.4	259.1	11.1	0.6	6.2	8.7	119.1	113.2	0.3						9.4	19.7		32.4	1.1	66.5	
09YY143	76.7	0.1	12.9	0.5		0.1	0.2	0.5	4.0	4.6	0.0	1276	1.5	253.4	7.1	0.8	4.0	9.6	130.0	98.3	0.1						5.8	12.6		33.3	0.7	66.0	
09YY159	77.4	0.1	12.9	0.4		0.0	0.1	0.4	3.8	4.6	0.0	1288	1.3	253.8	5.3	0.4	2.5	6.2	122.6	98.3	0.0						20.4	9.0		38.5	-2.7	64.2	
09YY162	77.6	0.1	12.7	0.0		0.1	0.1	0.5	4.0	4.5	0.0	1291	1.4	248.7	0.0	1.0	2.7	8.7	128.8	95.5	0.0						7.0	4.1		35.2	-0.8	65.6	
09YY165	77.1	0.1	12.3	1.2		0.1	0.1	0.5	3.8	4.8	0.0	1283	0.8	240.3	16.1	1.4	2.0	9.6	123.9	101.1	0.1						3.9	18.9		32.4	3.6	64.0	
09YY166	77.4	0.1	12.6	0.8		0.1	0.1	0.5	3.8	4.8	0.0	1288	1.5	247.9	11.1	1.1	2.0	9.6	123.9	101.1	0.1						3.7	14.6		34.0	1.5	64.6	
09YY163	77.4	0.1	12.9	0.4		0.0	0.1	0.4	3.9	4.6	0.0	1287	1.3	252.5	6.1	0.4	2.5	6.2	125.8	98.3	0.0						15.8	9.8		36.9	-1.6	64.7	
Bt-gr																																	
09YY136	76.9	0.1	13.1	0.5		0.0	0.1	0.5	3.9	4.7	0.0	1279	1.3	257.0	6.8	0.3	3.5	8.9	125.8	100.6	0.0						12.6	11.5		35.1	-0.8	65.7	
09YY157	75.5	0.2	13.6	1.0		0.1	0.3	0.6	3.8	4.7	0.0	1256	2.1	267.4	14.0	0.8	6.2	10.5	122.3	98.9	0.4						25.1	22.4		37.1	-1.6	64.5	
09YY160	75.7	0.2	13.1	0.9		0.1	0.3	0.7	3.7	4.8	0.0	1260	2.1	256.8	12.2	0.7	6.7	12.1	119.7	102.5	0.4							10.3	21.1		34.1	1.1	64.7
09YY161	75.9	0.2	13.1	0.9		0.1	0.3	0.7	3.8	4.8	0.0	1263	2.1	257.7	12.6	1.0	7.2	13.2	123.6	100.8	0.4							6.9	22.0		33.0	1.9	65.1
170.00	73.0	0.3	14.7	1.2		0.3	0.4	1.1	4.2	4.4	0.0	1215	3.3	288.9	16.4	4.1	9.7	20.0	134.9	94.3	0.6						19.8	29.3		31.2	0.5	68.3	
MME																																	
09YY139	51.2	1.1	18.6	7.8		0.1	4.4	5.5	4.0	2.6	0.3	851.6	13.5	364.1	108.7	1.6	108.7	98.3	130.4	54.1	4.2						-16.9	230.9		2.4	39.0	58.5	
09YY164	54.2	1.0	17.3	8.4		0.1	4.6	7.4	3.2	1.8	0.3	901.8	12.5	339.9	116.1	2.0	115.1	131.1	102.6	38.4	3.9						-63.2	243.7		-0.3	49.6	50.7	
09YY167	53.6	0.9	15.2	8.3		0.2	7.9	7.6	3.3	1.2	0.3	891.2	11.6	297.2	115.6	3.0	195.3	134.8	107.5	25.1	3.7						-104.9	322.6		-8.1	69.0	39.0	
09YY168	65.0	0.7	16.0	4.4		0.1	2.0	4.4	3.8	2.7	0.2	1081	8.5	314.0	61.3	1.3	48.4	78.3	122.6	56.3	2.3							-12.4	118.2		17.7	22.0	60.3
09YY169	68.3	0.5	15.4	3.0		0.1	1.0	2.4	4.1	4.0	0.1	1137	6.6	301.3	41.6	0.7	25.6	42.1	132.3	85.6	2.0							-0.7	73.7		22.4	11.2	66.4

Yandangshan complex (He Z et al., 2009 Int Geol Rw 51 556-582)

Augite quartz porphyritic syenite

YS01	65.0	0.6	16.6	2.9	0.1	0.7	2.0	5.1	5.4	0.1	1081	7.4	325.4	41.7	1.8	16.4	36.0	164.6	113.6	1.8	-24.8	65.4	4.9	14.6	80.5
YS02	68.9	0.6	16.6	2.7	0.1	0.7	1.9	5.0	5.5	0.1	1147	7.3	325.6	39.5	1.6	17.4	33.7	161.0	116.6	1.8	-19.4	64.1	9.7	12.9	77.4
YS03	66.1	0.6	16.7	2.6	0.1	0.7	1.8	5.2	5.4	0.1	1099	7.0	327.6	37.7	1.6	16.4	31.7	167.5	115.3	1.5	-18.7	61.0	6.8	12.7	80.6
S17	65.8	0.5	16.1	2.7	0.1	0.7	1.9	5.2	5.4	0.2	1095	5.9	314.8	38.5	1.1	17.4	33.7	167.8	113.8	2.1	-34.2	61.8	3.5	15.9	80.6
YD05	65.7	0.6	17.0	2.8	0.1	0.7	1.8	5.1	5.2	0.1	1093	7.4	333.7	39.7	1.6	17.1	31.4	165.9	109.6	1.7	-4.5	64.2	10.4	10.4	79.1
S18	66.1	0.5	17.5	2.5	0.1	0.7	1.7	4.5	5.0	0.2	1101	5.8	342.5	36.1	1.4	16.4	29.6	145.5	106.1	2.5	31.6	58.3	22.3	2.5	75.2
Rhyolitic volcanics																									
S15	74.5	0.2	13.7	1.2	0.1	0.3	0.4	4.0	5.4	0.0	1240	2.1	268.3	17.3	1.1	6.5	7.7	129.1	113.8	0.1	10.1	25.8	29.8	1.9	68.3
S11	74.4	0.1	13.3	1.3	0.1	0.2	0.5	4.2	5.3	0.0	1238	1.8	260.9	18.0	0.7	4.7	9.1	136.8	112.1	0.1	-6.2	24.5	25.3	4.9	69.8
S7	74.9	0.1	13.6	1.1	0.0	0.3	0.2	2.4	5.8	0.0	1246	1.8	267.0	14.7	0.1	8.4	3.2	77.4	123.6	0.1	59.5	24.9	48.5	-8.1	59.6
YD02	73.1	0.2	13.6	1.3	0.1	0.2	0.7	4.3	5.2	0.0	1217	2.9	267.0	18.5	1.0	4.2	12.3	137.5	111.0	0.3	-6.1	25.6	24.0	5.1	70.9
S5	70.3	0.4	14.6	2.0	0.0	0.7	1.5	4.0	5.4	0.1	1170	4.6	287.2	28.0	0.6	17.4	26.0	130.4	114.2	1.0	-9.5	50.0	19.7	9.4	70.9
S3	73.2	0.2	14.1	1.3	0.0	0.3	0.3	4.4	4.4	0.0	1218	3.0	277.0	18.6	0.4	7.7	5.5	143.3	93.4	0.4	29.2	29.3	33.9	-1.4	67.4

Kuqi complex (he Z.y and Xu X.s 2012 ChemGeol 328 208-221)

Nanyu diorite

F3429	54.6	1.4	15.9	8.3	0.1	3.2	6.1	3.3	2.9	0.7	908.6	17.3	311.3	104.0	2.0	79.4	108.2	107.5	60.9	10.4	-73.6	200.6	-4.2	46.7	57.5
model	55.7	0.6	15.6	9.3		3.9	7.1	2.8	2.2		926.9	7.5	306.0	116.5	0.0	96.8	126.6	90.4	46.7	0.0	-84.3	220.8	-1.9	51.5	50.4
Danyang monzogranite																									
CM23	70.4	0.4	14.3	2.9	0.1	0.9	2.3	3.9	3.8	0.1	1172	4.8	280.5	36.7	0.8	22.8	41.7	125.5	80.3	1.7	-8.7	64.3	25.0	11.4	63.6
CM24	69.8	0.4	14.5	2.5	0.1	0.8	2.1	3.5	5.0	0.1	1162	5.4	283.8	30.9	1.0	19.1	37.3	112.6	106.2	1.3	-9.5	55.4	22.6	10.3	67.1
CM25	71.0	0.4	14.3	2.2	0.1	0.7	1.9	3.6	4.9	0.1	1181	4.8	280.5	27.9	0.7	17.1	34.6	116.2	104.7	1.3	-9.5	49.8	23.5	9.4	67.1
CM26	70.9	0.4	14.5	2.3	0.1	0.7	2.0	3.5	5.0	0.1	1180	5.1	285.2	28.4	1.1	17.4	36.2	111.3	105.3	1.3	-3.8	50.9	25.1	8.4	66.5
CM28	71.5	0.3	14.0	1.6	0.1	0.6	2.1	3.5	4.3	0.1	1189	3.4	274.8	20.4	1.1	14.4	37.8	114.2	91.3	1.1	-6.3	38.2	27.2	7.1	65.7
margins																									
CM22	67.3	0.6	15.1	3.3	0.1	1.2	2.8	3.4	4.8	0.1	1120	7.0	297.0	41.2	1.1	29.8	49.4	110.0	102.8	2.0	-14.6	78.0	18.9	14.7	66.4
CM27	63.1	0.7	16.1	4.5	0.1	1.8	4.1	3.7	4.2	0.2	1049	8.5	316.2	56.6	1.3	44.2	73.6	120.4	89.0	2.8	-40.2	109.3	7.6	24.6	67.8
Fuzhou syenogranite																									
CM29	73.9	0.2	13.0	1.4	0.1	0.3	1.2	4.1	3.8	0.1	1229	2.9	254.8	17.7	1.3	7.9	21.4	132.3	81.5	1.0	-1.8	28.5	30.8	4.7	64.5
CM32	75.7	0.2	12.1	1.4	0.1	0.2	0.5	4.2	4.1	0.0	1260	3.0	236.6	17.8	1.7	3.7	9.1	136.8	86.2	0.3	-4.6	24.5	31.7	4.7	63.6
CM33	75.5	0.2	12.6	0.9	0.1	0.2	0.7	4.0	4.5	0.0	1256	2.0	247.0	10.9	0.8	4.0	12.3	128.8	96.0	0.3	-2.4	16.9	31.3	3.0	65.7
margin																									
CM35	62.8	0.7	15.9	5.4	0.1	2.6	4.5	4.0	3.6	0.2	1045	9.3	311.1	67.5	1.7	65.3	81.0	127.8	76.4	3.4	-55.0	142.0	4.4	32.0	63.6
enclave																									
F3469	54.8	0.9	16.8	6.0	0.2	2.9	5.5	4.4	3.0	0.3	912.5	10.8	328.8	75.5	2.1	72.5	97.7	142.0	63.7	3.8	-72.4	158.7	-9.0	39.7	69.3

Chapter 1

Kuiqi peralkaline granite																									
CM10	76.9	0.1	11.5	1.1	0.0	0.1	0.0	4.3	4.4	0.0	1280	1.8	226.2	13.9	0.4	2.0	0.0	139.1	94.3	0.1	-7.2	17.6	31.6	4.1	64.3
CM11	76.6	0.2	11.5	1.4	0.1	0.0	0.0	4.2	4.4	0.0	1275	1.9	225.8	17.5	0.7	1.0	0.0	135.5	94.1	0.1	-3.8	20.4	32.7	3.9	63.4
CM12	76.6	0.2	11.4	1.3	0.1	0.0	0.1	4.3	4.3	0.0	1275	1.9	222.6	16.5	1.8	0.2	1.6	137.5	91.5	0.0	-9.5	18.7	31.6	4.8	63.7
CM13	76.7	0.1	11.6	1.3	0.1	0.0	0.1	4.2	4.5	0.0	1277	1.6	227.5	15.8	0.7	0.5	2.1	136.5	94.7	0.0	-7.9	17.9	31.4	4.3	64.3
CM14	76.5	0.2	11.1	1.4	0.1	0.0	0.1	4.4	4.3	0.0	1273	2.1	217.5	17.9	2.0	0.5	1.6	142.9	90.9	0.1	-19.5	20.5	28.7	7.0	64.3
CM15	76.0	0.2	11.9	1.3	0.1	0.0	0.1	4.6	4.4	0.0	1264	2.0	234.2	15.8	1.6	0.0	1.6	148.8	92.8	0.0	-10.5	17.8	28.4	4.8	66.8
CM62	77.1	0.1	11.1	1.3	0.1	0.0	0.1	4.3	4.3	0.0	1283	1.6	218.5	15.7	1.6	0.2	1.8	137.1	91.3	0.0	-13.5	17.5	31.2	5.4	63.4
CM37	76.5	0.2	12.1	1.3	0.1	0.0	0.1	4.1	4.8	0.0	1273	1.9	236.8	16.0	1.1	0.7	1.1	131.7	100.8	0.1	2.1	18.7	32.9	2.4	64.7
CM64	76.7	0.1	11.5	1.5	0.1	0.0	0.0	4.2	4.3	0.0	1276	1.4	225.0	18.8	1.1	0.0	0.0	134.2	92.1	0.0	-1.4	20.2	33.9	3.4	62.8
CM66	76.9	0.1	11.4	1.0	0.0	0.0	0.1	3.7	4.9	0.0	1279	1.0	224.0	12.8	0.6	0.0	2.3	120.4	103.6	0.0	-4.6	13.8	33.6	3.0	63.3
CM67	76.2	0.1	11.9	1.5	0.1	0.0	0.1	4.4	4.4	0.0	1268	1.1	232.8	18.2	1.1	0.2	2.1	142.0	92.8	0.0	-6.2	19.5	30.6	4.2	65.2
CM69	75.3	0.1	11.3	2.4	0.2	0.0	0.0	4.7	4.2	0.0	1253	1.6	220.9	30.3	3.2	0.0	0.2	152.3	88.1	0.0	-19.9	31.9	26.5	8.9	64.7
CM70	75.3	0.2	12.6	0.9	0.1	0.1	0.4	4.3	4.4	0.0	1253	2.0	246.6	11.6	0.8	2.2	7.7	138.1	94.1	0.3	-0.9	15.9	30.7	2.6	66.7
CM72	76.0	0.2	11.7	1.3	0.0	0.0	0.1	4.3	4.7	0.0	1265	2.6	229.9	16.2	0.6	0.5	1.6	138.1	100.6	0.1	-12.1	19.3	28.7	5.3	66.0
CM73	76.0	0.2	11.7	1.3	0.0	0.1	0.1	4.3	4.7	0.0	1265	3.0	230.1	15.8	0.6	1.2	1.8	139.1	100.6	0.1	-13.2	20.0	28.2	5.7	66.1
grey dikes																									
CM63	74.8	0.1	10.3	4.3	0.1	0.0	0.0	4.8	4.3	0.0	1245	1.4	201.1	54.0	1.8	0.0	0.2	154.2	91.1	0.0	-44.6	55.4	20.2	17.4	62.4
CM65	77.4	0.1	12.2	1.1	0.2	0.0	0.2	4.3	4.3	0.0	1288	1.0	238.3	13.2	3.4	0.2	2.7	139.4	91.9	0.1	1.6	14.4	33.5	1.8	64.7
Bijashan peralkaline granite																									
CM44	76.8	0.1	12.1	0.8	0.1	0.1	0.3	4.4	4.4	0.0	1278	1.1	237.3	9.8	1.3	1.2	5.2	140.4	93.2	0.1	-6.6	12.1	30.8	3.1	66.1
CM45	77.0	0.1	12.0	0.6	0.0	0.0	0.2	4.2	4.4	0.0	1282	1.3	235.6	7.6	0.6	0.2	3.2	135.9	94.1	0.1	-0.7	9.1	33.1	1.5	65.3
CM47	76.3	0.1	12.1	0.8	0.0	0.0	0.3	4.3	4.6	0.0	1270	1.4	237.0	9.9	0.4	0.7	5.0	139.7	96.6	0.1	-9.4	12.0	29.4	3.7	66.9
CM48	76.2	0.1	12.0	1.1	0.1	0.0	0.2	4.6	4.5	0.0	1268	1.8	234.4	13.4	0.8	0.5	3.0	147.1	95.1	0.1	-13.9	15.6	27.6	5.1	67.3
dike																									
CM20	71.5	0.4	14.4	1.9	0.1	0.2	0.3	4.1	5.4	0.1	1189	4.6	283.2	23.2	0.8	5.2	5.2	132.6	113.8	0.7	26.5	33.0	30.3	-0.3	70.0
Taohuadao granite (Zhao J.I et al., 2016 Lithos 240 16-30)																									
12THD-1	78.5	0.1	11.6	1.1	0.1	0.2	0.2	4.3	4.3	0.0	1306	1.3	226.6	1.0	1.1	5.5	3.6	137.8	90.9	0.1	-9.2	7.7	32.7	3.0	64.3
12THD-4	78.2	0.1	11.8	0.9	0.0	0.2	0.1	4.3	4.5	0.0	1301	1.5	232.1	0.5	0.6	4.7	1.4	137.5	96.2	0.1	-4.4	6.7	32.8	1.9	65.3
T-1*	77.8	0.1	11.4	1.7	0.1	0.1	0.5	4.1	4.3	0.0	1295	1.1	223.2	1.4	1.6	2.0	8.7	131.0	91.7	0.1	-17.0	4.5	31.3	4.1	64.6
T-3*	76.5	0.1	11.5	1.7	0.1	0.1	0.5	4.3	4.4	0.0	1273	1.5	225.4	1.5	1.7	1.7	9.3	137.5	92.8	0.1	-23.4	4.7	27.6	5.4	66.9
T-9*	77.0	0.1	11.6	1.7	0.1	0.1	0.5	3.8	4.7	0.0	1281	1.4	227.1	1.8	2.0	2.0	8.7	122.6	100.4	0.1	-13.4	5.1	31.4	3.5	65.1
T-10*	77.0	0.1	11.5	1.8	0.1	0.0	0.4	4.7	4.4	0.0	1281	1.1	226.2	1.6	1.8	0.2	6.2	153.0	94.1	0.1	-33.3	3.0	23.3	7.1	69.6
Taohuadao aeg-granite (Gao Y.y 2015 PhD dissertation)																									
09YY118	78.1	0.2	12.1	0.9	0.0	0.1	0.1	4.1	4.5	0.0	1299	2.0	237.0	12.0	0.3	1.7	1.4	133.3	96.0	0.0	4.9	15.7	35.1	1.4	63.5
09YY119	77.8	0.2	12.0	1.3	0.0	0.1	0.1	4.3	4.2	0.0	1294	2.1	234.4	17.8	0.6	2.5	1.6	139.1	88.3	0.0	3.8	22.4	34.9	2.6	62.4
09YY120	77.8	0.1	11.9	1.1	0.1	0.1	0.2	4.2	4.4	0.0	1294	1.8	234.0	15.4	1.0	1.7	2.7	136.8	94.3	0.0	-2.4	18.9	33.0	3.3	63.7
09YY121	77.5	0.2	11.8	1.2	0.2	0.1	0.3	4.2	4.4	0.0	1290	1.9	231.7	16.1	2.8	1.7	5.0	136.2	93.2	0.0	-7.7	19.7	31.8	4.5	63.7
09YY122	77.5	0.1	12.2	0.9	0.1	0.1	0.2	4.6	4.2	0.0	1289	1.8	238.3	13.1	0.7	2.0	4.3	147.5	89.2	0.0	-6.9	16.8	30.6	3.9	65.5
09YY125	77.6	0.2	11.8	1.1	0.0	0.3	0.2	4.3	4.2	0.0	1291	2.0	231.5	15.8	0.4	6.9	4.1	137.8	89.2	0.0	-3.7	24.8	33.1	4.4	62.4
09YY126	77.0	0.2	12.0	1.2	0.1	0.1	0.2	4.5	4.5	0.0	1281	2.0	235.6	16.4	1.1	3.5	4.1	144.6	96.4	0.0	-13.5	21.9	28.2	5.9	65.9
09YY127	76.8	0.2	12.0	1.2	0.1	0.3	0.2	4.5	4.5	0.0	1277	2.1	235.6	16.4	1.1	6.7	4.3	145.9	96.4	0.0	-15.2	25.2	27.4	6.8	65.9
09YY128	77.4	0.2	12.2	1.1	0.1	0.1	0.1	4.4	4.5	0.0	1288	1.9	238.3	14.6	1.3	2.2	1.4	142.0	96.0	0.0	-2.5	18.7	31.5	3.3	65.2
09YY129	77.4	0.2	12.2	1.1	0.1	0.1	0.1	4.4	4.5	0.0	1287	2.4	239.7	15.3	0.7	2.2	2.1	140.7	95.3	0.0	-0.6	19.9	32.1	3.1	64.8
09YY130	78.0	0.1	11.9	1.2	0.0	0.1	0.2	3.8	4.4	0.0	1299	1.5	233.0	16.4	0.3	3.0	2.9	123.3	92.4	0.0	11.7	20.9	38.8	0.8	60.4
09YY131	78.0	0.1	11.9	1.2	0.0	0.1	0.2	3.8	4.3	0.0	1298	1.6	233.8	16.4	0.1	3.2	2.9	123.6	92.1	0.0	12.4	21.3	38.8	0.8	60.4
09YY132	75.2	0.1	13.8	1.0	0.1	0.3	0.3	3.2	5.5	0.0	1252	1.6	269.9	13.9	1.0	7.9	4.6	102.0	116.1	0.1	42.5	23.5	41.7	-4.9	63.2
09YY146	76.5	0.1	12.4	1.2	0.2	0.1	0.3	4.7	4.5	0.0	1273	1.6	243.2	16.3	2.3	2.5	4.5	151.0	95.8	0.0	-12.5	20.4	26.8	5.5	67.7
09YY147	78.1	0.1	12.0	1.0	0.0	0.1	0.1	3.2	4.7	0.0	1300	1.8	234.4	14.0	0.1	2.5	1.6	103.9	100.2	0.0	27.1	18.3	44.3	-2.6	58.3
09YY148	78.0	0.1	12.0	1.0	0.0	0.1	0.2	3.9	4.3	0.0	1299	1.5	235.4	14.3	0.3	2.5	3.7	127.1	91.5	0.0	9.3	18.3	37.6	0.9	61.5
09YY149	78.2	0.1	11.9	0.9	0.0	0.1	0.3	4.0	4.3	0.0	1301	1.5	233.0	13.1	0.1	2.0	4.5	127.5	91.1	0.0	5.6	16.6	37.0	1.4	61.6
09YY150	78.0	0.1	11.9	1.0	0.0	0.1	0.2	4.0	4.4	0.0	1298	1.5	233.8	14.5	0.3	2.2	3.7	129.4	93.4	0.0	3.5	18.2	35.7	2.0	62.2
09YY151	77.7	0.1	11.9	1.2	0.0	0.1	0.2	4.3	4.2	0.0	1293	1.4	233.8	17.2	0.3	2.0	4.1	137.1	90.0	0.0	-1.5	20.6	33.6	3.4	63.0
09YY152	77.8	0.1	11.7	1.5	0.0	0.1	0.4	4.2	4.2	0.0	1294	1.8	229.9	20.6	0.1	1.2	6.6	133.9	88.5	0.0	-5.8	23.6	33.4	4.7	61.9
09YY153	74.9	0.2	13.1	1.0	0.0	0.1	0.4	3.6	6.4	0.0	1246	2.6	257.7	14.0	0.1	2.5	6.4	116.8	136.5	0.1	-8.4	19.1	24.9	4.5	70.6
09YY154	77.7	0.1	12.0	0.9	0.3	0.3	0.1	4.3	4.3	0.0	1293	1.5	234.8	12.0	4.8	7.6	2.5	138.4	91.9	0.0	-0.6	21.1	3		

Chapter 1

Gejiu complex (Chang Y.b et al., 2012 CMP 164 659-676)

Longchahe granite

7056	70.3	0.4	14.8	2.4	0.1	0.8	1.8	3.1	5.2	0.2	1171	5.5	290.7	29.4	0.7	19.4	32.1	99.7	111.3	2.1	15.5	54.3	30.0	5.1	64.8
7057	63.8	0.7	16.7	3.8	0.1	1.4	3.3	3.3	5.8	0.3	1062	8.1	326.6	47.8	1.0	35.7	58.5	106.2	123.8	3.7	-20.3	91.7	10.4	17.8	71.7
7058	70.8	0.4	14.8	2.1	0.1	0.7	1.8	3.2	5.2	0.1	1179	4.4	289.5	25.9	0.7	16.9	31.7	102.3	110.4	2.0	13.4	47.2	29.8	4.5	65.7
7059	63.4	0.7	16.1	4.0	0.1	1.5	3.3	3.4	5.6	0.3	1055	8.5	315.8	49.6	1.1	36.7	58.5	108.1	117.8	3.9	-27.1	94.8	9.5	19.9	70.7
7060	67.7	0.5	15.2	3.0	0.1	1.1	2.5	3.2	5.2	0.2	1127	6.6	298.4	37.4	0.7	27.3	44.2	101.6	111.3	2.8	-3.0	71.4	22.2	11.4	66.3
7061	63.0	0.8	16.4	4.4	0.1	1.6	3.5	3.2	5.5	0.3	1048	9.4	321.7	54.5	1.0	40.2	62.8	102.6	116.3	4.4	-22.8	104.1	10.6	20.4	69.1
7062	59.9	0.9	16.9	5.7	0.1	2.1	3.7	3.2	6.2	0.4	996.3	11.4	330.5	70.8	1.3	50.9	66.5	103.6	131.0	5.4	-37.1	133.0	1.7	27.5	70.8
7063	65.9	0.7	15.5	4.0	0.1	1.4	3.1	3.4	4.8	0.3	1097	8.1	303.6	49.5	1.1	34.2	55.5	108.4	102.1	3.5	-17.8	91.8	16.6	17.5	65.9
7064	67.8	0.6	14.7	3.9	0.1	1.4	2.4	3.3	4.3	0.3	1129	8.0	288.0	48.2	1.0	34.0	42.8	106.5	91.5	3.5	4.4	90.2	26.6	12.9	60.5
7065	64.9	0.8	16.1	4.2	0.1	1.6	3.2	3.1	4.9	0.3	1080	9.4	315.8	52.1	1.0	38.7	57.1	101.3	104.7	4.4	-4.3	100.2	18.9	16.0	65.0
7072	74.9	0.1	14.8	0.2	0.0	0.3	0.4	3.6	4.6	0.2	1247	1.8	289.3	2.9	0.1	7.2	7.3	116.2	98.1	2.5	60.5	11.8	45.3	-10.2	64.9
7079	62.0	0.7	15.9	3.9	0.1	1.5	3.0	5.4	6.0	0.3	1031	8.6	312.5	49.2	0.8	37.2	54.2	175.2	127.0	4.2	-98.1	95.1	-18.2	33.3	85.0

Jiasha gabbro

7053	47.0	0.9	13.0	7.4	0.2	5.4	13.4	2.4	4.2	1.5	781.8	10.8	255.6	92.4	2.5	134.0	239.5	76.8	90.0	21.0	-390.2	237.2	-93.0	118.9	74.1
7054	45.5	1.2	12.1	9.4	0.2	9.4	12.7	1.6	3.9	0.9	757.9	15.1	237.2	118.1	2.3	234.2	225.7	52.0	82.8	12.7	-349.1	367.5	-76.6	127.8	48.8
7067	44.0	1.2	11.1	8.9	0.2	7.9	19.6	1.0	1.8	1.4	732.1	14.6	217.7	111.6	2.1	195.5	349.1	33.2	39.1	20.3	-552.9	321.8	-125.1	165.0	60.1
7068	44.7	1.3	11.9	9.8	0.2	9.2	13.5	1.3	3.9	0.9	743.9	16.3	234.2	122.1	2.3	229.3	240.9	43.2	83.0	12.4	-373.9	367.7	-83.2	133.6	49.6
7070	44.2	1.5	11.8	9.6	0.2	8.8	16.6	1.1	2.9	1.4	735.6	18.6	230.7	120.4	2.1	217.9	295.8	36.1	60.5	19.4	-457.6	356.9	-101.9	148.8	53.0
8107	47.1	1.8	17.4	10.2	0.1	4.7	9.2	2.2	4.4	1.2	783.3	22.0	341.5	127.1	1.8	117.1	163.3	72.3	92.6	17.3	-150.0	266.3	-32.7	71.8	60.9
8108	43.0	2.7	14.5	13.9	0.2	5.8	10.6	1.9	3.6	1.8	715.1	33.8	284.2	174.5	2.3	144.9	188.3	62.3	76.2	25.5	-230.9	353.2	-52.7	104.2	48.5
8109	52.0	1.5	19.0	7.8	0.1	2.9	7.4	3.0	4.0	0.7	864.9	18.8	372.9	98.2	1.6	70.7	132.5	95.5	84.1	10.3	-71.7	187.7	-10.9	43.2	67.7
8110	44.1	2.7	15.9	12.5	0.2	5.7	9.7	2.0	3.9	1.5	734.2	33.8	311.3	156.8	2.1	140.9	173.5	65.5	82.0	21.3	-183.2	331.5	-41.0	89.8	51.2
8111	45.1	2.0	16.6	11.6	0.1	5.4	10.2	2.1	3.8	1.4	749.9	24.9	325.8	145.8	2.0	132.8	181.0	67.1	80.5	19.7	-183.8	303.4	-40.9	85.0	55.8

Chapter 1

	SiO ₂	TiO ₂	Al ₂ O ₃	FeO	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	Si	Ti	Al	Fe	Mn	Mg	Ca	Na	K	P	A	B	Q*3	B*3	F*3	
Late Yanshanian (110-70 Ma)																											
Western																											
Dulong granite (Xu B et al., 2015 Lithos 218-219 54-72)																											
fine-grained granite																											
11TJ-62	73.2	0.1	14.5		1.0	0.0	0.2	0.8	3.6	4.4	0.2	1218	1.3	283.6	12.4	0.6	5.5	13.7	117.5	93.6	3.4		45.1	19.1	41.0	-6.1	65.1
11TJ61	72.5	0.1	14.4		0.9	0.0	0.2	0.9	3.2	5.6	0.2	1207	1.4	282.1	11.3	0.6	5.2	16.6	102.9	117.8	2.8		28.1	17.9	35.0	-2.9	67.9
11TJ-58	71.8	0.2	15.0		1.4	0.0	0.3	0.9	3.4	5.0	0.2	1195	2.1	293.6	16.9	0.6	6.7	16.8	109.4	106.2	3.4		44.6	25.7	38.4	-5.0	66.6
11TJ-55	71.3	0.1	15.4		1.5	0.1	0.3	0.8	3.6	4.7	0.2	1187	1.3	302.1	18.4	0.8	8.4	15.0	115.8	99.6	3.0		56.7	28.1	40.6	-7.1	66.5
11LJS-96-2	72.1	0.1	14.6		1.4	0.1	0.3	0.8	3.1	5.1	0.3	1199	1.6	286.4	17.4	0.7	7.4	14.6	98.7	109.1	3.7		49.3	26.5	41.4	-5.9	64.4
11LJS-68	73.6	0.1	14.0		1.2	0.0	0.3	0.8	3.2	4.9	0.2	1225	1.6	273.6	14.5	0.6	6.5	13.9	101.6	103.2	3.2		41.0	22.6	41.7	-4.8	63.1
DL-43	73.1	0.1	14.7		0.9	0.0	0.2	0.8	3.8	4.4	0.2	1217	1.1	287.4	11.3	0.4	5.2	14.6	123.6	93.8	3.1		40.7	17.6	38.6	-5.4	66.8
DL-30	73.8	0.1	14.4		0.7	0.0	0.2	0.8	3.8	4.4	0.2	1229	0.9	282.1	8.9	0.4	3.7	14.4	121.7	93.6	3.1		37.9	13.5	39.2	-5.5	66.4
DL-29	73.1	0.1	14.5		0.9	0.1	0.2	0.8	3.8	4.6	0.2	1216	1.1	284.2	10.9	0.7	3.7	14.6	122.0	96.6	2.8		36.4	15.7	37.7	-4.9	67.2
DL-28	73.5	0.1	14.6		0.8	0.0	0.2	0.9	3.8	4.4	0.2	1224	1.0	286.2	9.5	0.4	4.5	15.7	121.7	93.8	3.2		39.3	15.0	38.9	-5.6	66.6
DL-27	73.9	0.1	14.5		0.9	0.0	0.2	0.7	3.4	4.7	0.2	1230	1.0	283.4	11.3	0.4	4.5	12.5	110.4	100.2	3.0		47.9	16.7	42.3	-7.0	64.8
13MG-11	72.9	0.1	14.7		1.1	0.0	0.3	0.7	4.0	4.4	0.2	1212	1.3	287.6	13.3	0.6	6.2	12.8	129.7	93.0	3.4		39.2	20.7	37.3	-4.7	67.3
coarse-grained granite																											
13MG-12	72.7	0.1	14.7		1.0	0.0	0.2	0.7	3.9	4.4	0.3	1209	1.3	288.5	12.5	0.6	5.7	12.8	126.2	93.8	3.7		42.9	19.5	38.5	-5.6	67.1
13MG-13	73.1	0.1	14.7		1.1	0.0	0.2	0.7	4.0	4.5	0.2	1217	1.3	289.1	13.7	0.6	4.7	12.1	129.4	94.5	3.4		41.0	19.6	37.7	-5.2	67.5
13MG-14	73.1	0.1	14.5		0.9	0.0	0.3	0.7	3.9	4.6	0.2	1217	1.3	284.0	11.8	0.6	7.2	12.3	125.5	97.5	3.4		36.4	20.2	37.1	-4.2	67.1
13HST-5	72.3	0.1	15.1		1.1	0.0	0.2	0.7	3.9	4.6	0.2	1203	1.3	296.2	13.9	0.6	4.5	13.0	127.1	97.7	3.2		45.4	19.6	37.6	-6.1	68.4
13HST-6	73.0	0.1	14.8		1.0	0.0	0.3	0.7	3.7	4.4	0.3	1214	1.4	290.1	12.9	0.6	6.5	12.1	118.1	93.0	3.5		54.8	20.7	42.8	-7.8	65.0
13HST-1	70.4	0.2	14.8		1.9	0.1	0.5	1.1	3.4	5.2	0.3	1171	2.8	290.3	23.2	0.8	11.4	18.7	108.7	110.0	3.7		34.1	37.3	34.4	-1.1	66.8
porphyritic granite																											
13HST-2	71.7	0.2	14.7		2.0	0.1	0.5	1.0	3.4	4.7	0.3	1193	3.0	288.0	24.7	0.8	12.2	18.5	109.7	99.4	3.9		41.8	39.8	38.6	-2.3	63.7
13HST-3	70.7	0.3	14.9		1.7	0.1	0.5	1.1	3.7	5.3	0.2	1177	3.1	292.7	21.8	0.8	12.2	19.8	117.8	112.3	3.4		23.0	37.1	30.0	1.0	68.9
13HST-4	71.6	0.2	14.7		1.9	0.1	0.4	1.1	3.6	4.5	0.3	1192	2.9	287.8	23.3	0.8	9.4	19.6	116.8	94.5	3.7		37.2	35.6	37.2	-2.0	64.8
13HST-7	71.0	0.2	14.4		1.7	0.1	0.3	1.6	2.8	5.1	0.3	1182	2.9	282.1	21.8	1.3	8.4	28.4	89.4	108.5	3.8		27.5	33.1	36.4	-0.5	64.0
13HST-20	72.9	0.1	14.5		1.1	0.0	0.2	0.7	3.9	4.4	0.2	1214	1.5	284.4	13.4	0.4	6.0	11.6	124.9	94.1	3.4		42.3	20.9	39.0	-5.3	66.3
13HST-23	72.3	0.2	15.4		1.2	0.1	0.4	0.7	2.2	6.8	0.3	1203	1.9	301.5	15.2	1.0	8.9	11.9	71.6	143.7	3.9		62.2	26.0	42.7	-8.4	65.7
Longchahe complex (Cheng Y.b et al., 2012 CMP 164 659-676)																											
Longchahe granite																											
7056	70.3	0.4	14.8		2.4	0.1	0.8	1.8	3.1	5.2	0.2	1171	5.5	290.7	29.4	0.7	19.4	32.1	99.7	111.3	2.1		15.5	54.3	30.0	5.1	64.8
7057	63.8	0.7	16.7		3.8	0.1	1.4	3.3	3.3	5.8	0.3	1062	8.1	326.6	47.8	1.0	35.7	58.5	106.2	123.8	3.7		-20.3	91.7	10.4	17.8	71.7
7058	70.8	0.4	14.8		2.1	0.1	0.7	1.8	3.2	5.2	0.1	1179	4.4	289.5	25.9	0.7	16.9	31.7	102.3	110.4	2.0		13.4	47.2	29.8	4.5	65.7
7059	63.4	0.7	16.1		4.0	0.1	1.5	3.3	3.4	5.6	0.3	1055	8.5	315.8	49.6	1.1	36.7	58.5	108.1	117.8	3.9		-27.1	94.8	9.5	19.9	70.7
7060	67.7	0.5	15.2		3.0	0.1	1.1	2.5	3.2	5.2	0.2	1127	6.6	298.4	37.4	0.7	27.3	44.2	101.6	111.3	2.8		-3.0	71.4	22.2	11.4	66.3
7061	63.0	0.8	16.4		4.4	0.1	1.6	3.5	3.2	5.5	0.3	1048	9.4	321.7	54.5	1.0	40.2	62.8	102.6	116.3	4.4		-22.8	104.1	10.6	20.4	69.1
7062	59.9	0.9	16.9		5.7	0.1	2.1	3.7	3.2	6.2	0.4	996.3	11.4	330.5	70.8	1.3	50.9	66.5	103.6	131.0	5.4		-37.1	133.0	1.7	27.5	70.8
7063	65.9	0.7	15.5		4.0	0.1	1.4	3.1	3.4	4.8	0.3	1097	8.1	303.6	49.5	1.1	34.2	55.5	108.4	102.1	3.5		-17.8	91.8	16.6	17.5	65.9
7064	67.8	0.6	14.7		3.9	0.1	1.4	2.4	3.3	4.3	0.3	1129	8.0	288.0	48.2	1.0	34.0	42.8	106.5	91.5	3.5		4.4	90.2	26.6	12.9	60.5
7065	64.9	0.8	16.1		4.2	0.1	1.6	3.2	3.1	4.9	0.3	1080	9.4	315.8	52.1	1.0	38.7	57.1	101.3	104.7	4.4		-4.3	100.2	18.9	16.0	65.0
7072	74.9	0.1	14.8		0.2	0.0	0.3	0.4	3.6	4.6	0.2	1247	1.8	289.3	2.9	0.1	7.2	7.3	116.2	98.1	2.5		60.5	11.8	45.3	-10.2	64.9
7079	62.0	0.7	15.9		3.9	0.1	1.5	3.0	5.4	6.0	0.3	1031	8.6	312.5	49.2	0.8	37.2	54.2	175.2	127.0	4.2		-98.1	95.1	-18.2	33.3	85.0
MME																											
7040	58.6	0.7	17.5		5.6	0.1	1.7	4.0	3.6	6.8	0.4	974.5	8.1	343.3	70.4	1.7	41.9	71.0	117.5	143.9	5.1		-60.1	120.5	-9.1	30.0	79.1
7041	58.6	0.8	16.9		5.7	0.1	2.0	4.5	3.3	6.3	0.5	975.2	10.1	332.3	71.5	1.4	49.9	81.0	107.5	134.4	7.0		-71.5	131.5	-9.2	34.1	75.1
7042	57.6	0.8	17.8		6.2	0.1	2.0	4.1	3.5	6.6	0.5	959.1	9.5	348.2	77.8	1.8	48.6	73.1	112.9	139.5	6.3		-50.5	135.9	-6.8	30.5	76.4
7043	49.0	1.3	16.7		9.3	0.2	5.0	7.9	2.5	4.9	1.0	815.6	16.3	327.8	116.4	2.3	124.3	141.4	80.7	104.9	13.9		-140.6	256.9	-29.9	68.2	61.7
7045	56.9	1.1	18.8		6.8	0.1	1.8	4.1	3.1	5.9	0.5	946.6	13.8	367.8	84.9	1.4	43.9	73.3	101.3	125.9	6.5		-6.0	142.6	5.5	22.8	71.6
7046	51.4	1.4	16.9		8.6	0.1	4.8	7.1	2.6	5.2	0.9	855.4	16.9	331.7	107.2	1.8	118.1	127.1	85.2	110.0	12.0		-117.7	242.2	-22.6	60.4	62.2
7047	54.9	1.3	17.5		7.9	0.1	4.8	7.1	2.6	5.2	0.6	913.3	16.3	342.9	98.7	1.8	118.1	127.1	85.2	110.0	8.0		-106.6	233.1	-16.5	54.9	61.6
7048	50.1	1.2	17.9		9.0	0.2	3.9	7.1	3.6	4.1	1.1	833.4	14.9	350.7	113.2	2.3	95.8	126.8	114.6	86.0	15.9		-103.4	223.9	-22.1	55.3	66.8
7050	54.0	1.3	17.0		8.3	0.1	3.6	6.1	2.7	4.9	0.7	898.5	15.6	332.5	103.6	1.8	89.6	109.3	86.5	104.2	10.1		-76.9	208.8	-9.3	47.5	61.8
7051	52.2	1.1	17.7		8.6	0.2	3.0	5.8	3.0	5.9	0.9	868.5	13.6	346.4	107.3	2.1	75.4	103.8	97.8	125.1	12.1		-84.0	196.4	-17.3	47.0	70.3
7052	48.5	1.6	17.2		9.8	0.2	4.1	7.7	2.6	5.5	1.1	807.8	19.9	336.4	122.4	2.1	100.5	136.9	85.2	116.3	15.2		-139.0	242.8	-32.3	65.7	66.6

Chapter 1

Matou Dalai monzonite (Liu L et al., 2014 Lithos 196-197 242-260)

Matou	65.7	0.6	15.0	3.3	0.0	1.2	2.0	3.7	4.5	0.2	1093	7.1	294.8	41.1	0.6	30.8	34.8	119.1	96.2	3.0	10.0	79.0	24.1	10.2	65.7
MT-3	67.6	0.5	16.2	3.2	0.1	1.0	1.9	4.6	4.5	0.2	1125	6.6	317.0	39.5	1.1	24.8	34.6	148.1	96.4	2.8	3.3	70.9	18.6	9.9	71.5
MT-4	66.4	0.5	15.7	3.0	0.1	1.1	2.7	4.3	4.1	0.2	1105	6.1	307.6	37.9	1.1	26.3	47.3	137.8	86.4	2.8	-11.1	70.4	17.0	12.9	70.0
MT-5	66.3	0.5	15.6	3.5	0.1	1.2	2.7	4.3	4.3	0.2	1103	6.6	306.0	43.6	1.3	30.5	48.3	137.5	90.2	3.1	-18.3	80.7	14.7	15.8	69.5
MT-6	66.8	0.4	16.1	2.9	0.1	0.9	2.7	4.5	4.0	0.2	1111	5.5	316.4	35.7	1.0	22.8	48.5	144.6	84.7	2.5	-9.9	64.0	16.5	11.6	71.9
MT-7	66.8	0.5	15.6	3.2	0.1	1.1	2.6	4.4	4.0	0.2	1112	6.4	305.6	39.8	1.1	27.3	46.9	141.0	84.5	2.7	-13.7	73.5	16.6	13.8	69.5
MT-9	68.6	0.4	14.9	2.6	0.1	0.9	2.2	3.9	4.4	0.2	1141	5.4	292.1	33.1	1.1	22.6	39.8	124.2	94.3	2.3	-6.0	61.0	22.0	10.5	67.5
MT-10	67.9	0.5	15.2	4.0	0.1	1.4	1.9	3.9	5.0	0.2	1130	6.1	298.2	49.8	1.8	34.5	33.2	126.2	105.7	2.1	-0.1	90.5	20.6	13.5	65.9
1	66.9	0.5	15.9	3.5	0.1	1.3	2.8	4.1	4.3	0.2	1114	6.3	311.3	43.7	1.0	32.3	50.6	132.9	90.9	3.1	-13.8	82.2	16.4	15.0	68.6
2	66.6	0.5	16.0	3.7	0.1	1.2	1.5	4.6	4.5	0.2	1109	6.6	314.4	46.2	1.3	28.5	26.4	147.5	95.5	3.4	18.6	81.4	22.1	8.6	69.3
3	66.7	0.5	16.6	3.8	0.1	1.1	2.9	4.1	4.3	0.2	1110	6.3	325.0	47.1	1.3	27.5	50.8	132.0	91.9	3.4	-0.5	80.9	18.7	12.2	69.2
4	66.5	0.5	15.6	5.0	0.1	1.2	2.8	4.2	4.4	0.2	1107	6.3	305.4	62.0	1.1	30.8	49.0	134.9	92.4	3.1	-19.9	99.0	14.4	18.7	66.9
5	67.5	0.5	15.4	4.1	0.1	1.3	2.5	4.2	4.3	0.2	1123	6.3	302.3	51.7	1.3	31.8	44.4	135.2	90.4	2.8	-12.2	89.7	17.6	15.8	66.6
6	67.3	0.5	15.4	3.6	0.1	1.2	2.5	4.2	4.3	0.2	1120	6.3	301.9	45.5	1.1	30.8	44.9	135.2	92.1	3.1	-15.3	82.5	16.6	15.4	68.0
7	66.5	0.6	15.8	3.7	0.1	1.2	2.6	4.1	4.2	0.2	1106	6.9	309.5	46.6	1.8	29.8	46.7	132.9	90.0	2.8	-6.9	83.3	18.1	13.9	68.0
8																									
Dalai	63.7	0.8	15.8	4.2	0.1	1.8	3.2	4.0	4.9	0.2	1060	9.8	309.7	52.0	1.1	43.9	57.1	128.4	103.2	3.1	-36.0	105.7	7.1	23.1	69.8
DL-1	59.5	1.0	16.0	5.9	0.1	3.1	4.6	3.5	3.9	0.3	989.7	12.8	313.8	74.4	1.4	75.7	82.2	114.2	83.4	4.4	-48.2	162.8	3.6	34.5	61.8
DL-2	57.2	1.1	17.1	6.6	0.1	3.9	6.1	4.0	2.9	0.4	951.7	14.0	335.0	83.2	1.4	97.3	107.9	130.0	61.8	5.9	-72.6	194.4	-5.2	43.4	61.9
DL-3	58.2	1.0	16.0	6.2	0.1	3.7	5.5	3.8	3.1	0.4	968.7	13.0	313.7	77.9	1.4	91.3	98.4	122.0	65.4	5.2	-70.6	182.2	-2.1	41.9	60.3
DL-5	59.8	1.0	16.3	6.2	0.1	3.1	4.8	3.8	3.9	0.3	995.0	12.3	319.1	77.9	0.7	76.4	85.1	123.6	82.2	3.9	-56.7	166.6	0.5	36.3	63.2
9																									

Muchen complex (Liu L et al., 2013 Lithos 160-161 145-163)

Qtz monzonite																									
MC-1(1)	64.8	0.4	16.6	4.0	0.2	1.1	2.0	4.5	5.5	0.2	1078	5.0	325.0	51.0	2.0	28.0	35.0	146.0	116.0	3.0	-7.0	84.0	11.1	13.9	75.0
MC-2(3)	64.0	0.4	16.6	4.7	0.2	1.0	1.7	4.8	5.8	0.2	1065	5.0	326.0	58.0	2.0	25.0	30.0	153.0	124.0	3.0	-11.0	88.0	7.6	15.3	77.1
MC-3	67.1	0.2	16.4	3.2	0.2	0.5	1.3	5.2	5.8	0.1	1116	3.0	322.0	40.0	2.0	11.0	23.0	167.0	124.0	2.0	-15.0	54.0	8.0	10.9	81.1
MC-4	63.8	0.5	16.3	4.9	0.1	1.4	3.3	4.3	5.0	0.3	1062	6.0	319.0	61.0	1.0	35.0	58.0	138.0	106.0	4.0	-41.0	102.0	3.9	23.3	72.8
MME																									
MC-2(1)	55.4	0.7	18.0	7.9	0.3	2.4	4.9	4.9	4.3	0.6	921.0	9.0	352.0	99.0	4.0	60.0	87.0	157.0	91.0	8.0	-70.0	168.0	-13.6	39.0	74.6
MC-2	54.8	0.9	17.5	8.0	0.4	2.4	5.5	5.1	2.4	0.5	913.0	11.0	343.0	100.0	6.0	59.0	99.0	165.0	50.0	8.0	-70.0	170.0	-10.1	40.1	69.9

Daheshan syenite (He Z.y and Xu X.s 2012 ChemGeol 328 208-211)

DH02	64.7	0.5	15.3	4.9	0.1	0.4	1.8	4.7	5.1	0.2	1077	6.6	299.3	61.6	1.7	9.7	32.8	152.3	107.6	2.3	-26.24	77.93	8.0	17.1	74.8
DH03	66.8	0.5	14.5	4.7	0.1	0.4	1.6	4.3	5.3	0.1	1111	6.4	284.6	59.2	1.7	10.2	28.0	140.0	111.5	2.0	-22.88	75.8	12.6	16.1	71.3

Jiepailing complex (Xie L et al., 2016 OGR 78 300-321)

Granite porph																										
JPL-40	63.5	0.0	14.9	1.2	0.4	0.3	0.1	4.0	0.3	10.8	0.0	1057	0.3	291.7	22.2	3.8	3.0	71.7	8.1	230.1	0.1	-89.9	25.5	-6.9	22.7	84.2
JPL-43	73.5	0.0	14.2	0.2	0.1	0.1	0.0	2.2	0.1	8.1	0.0	1223	0.1	278.9	4.1	1.0	0.7	38.9	4.5	171.3	0.1	25.3	4.9	41.0	-4.4	63.3
JPL-31	70.6	0.0	14.0	1.0	0.3	0.4	0.1	1.5	0.2	8.4	0.0	1175	0.1	273.6	17.7	5.4	2.0	26.6	7.4	178.1	0.1	34.9	19.8	40.9	-4.1	63.2
JPL-38	68.1	0.0	14.8	0.4	0.2	0.1	0.1	1.7	0.2	11.5	0.0	1133	0.1	290.5	8.5	1.6	2.5	29.4	5.8	244.4	0.1	-18.5	11.1	15.1	5.5	79.4
Toz-granite																										
JPL-28	64.2	0.0	18.8	0.5	0.3	0.9	0.0	0.4	4.0	8.3	0.0	1069	0.0	369.4	10.6	12.4	1.0	6.8	128.8	175.2	0.1	51.9	11.6	18.5	-8.6	90.1
Znw JPL-17	71.8	0.0	15.2	1.1	0.2	0.1	0.0	0.1	3.6	5.6	0.0	1195	0.1	298.5	18.5	2.0	0.5	2.0	116.2	118.3	0.1	60.2	19.1	39.9	-9.1	69.2
Znw JPL-21	72.7	0.0	15.4	0.5	0.4	0.2	0.0	0.2	3.9	4.6	0.0	1209	0.0	301.3	12.0	2.3	0.5	3.4	126.8	96.8	0.1	70.9	12.5	44.6	-12.3	67.7
Greisen																										
JPL-14	74.5	0.0	13.9	3.8	0.9	0.7	0.0	0.2	0.1	2.9	0.0	1240	0.1	271.9	63.9	9.2	0.5	2.7	2.9	60.5	0.0	203.1	64.5	106.9	-32.8	25.9

Hengyang basalt (Meng L.f et al., 2012 Lithos 132-133 127-140)

07SC32-1	52.5	1.3	16.6	10.2	0.1	6.3	7.8	3.2	1.7	0.3	873.7	16.8	325.6	127.7	1.8	155.3	139.6	103.9	35.9	3.9	-93.4	299.9	-8.5	63.3	45.2
07SC32-2	52.4	1.3	16.4	10.2	0.1	6.5	7.8	3.3	1.8	0.3	872.0	16.3	321.7	127.7	1.8	160.5	139.8	104.9	37.4	3.7	-100.1	304.6	-10.4	65.3	45.1
07SC32-3	51.3	1.4	16.1	10.6	0.1	6.7	8.4	3.3	1.8	0.3	853.7	17.3	315.8	132.8	1.8	165.5	150.0	106.2	38.6	3.9	-128.9	315.5	-18.6	72.6	46.0
07SC32-4	52.3	1.3	16.5	10.1	0.1	6.5	7.9	3.3	1.7	0.3	870.4	16.5	323.7	126.5	1.7	161.8	141.6	105.8	35.5	3.7	-100.8	304.8	-10.6	65.4	45.2
07SC32-5	52.9	1.3	16.9	9.7	0.1	6.0	7.9	3.3	1.7	0.3	880.3	16.3	331.5	121.1	1.8	148.4	141.6	105.5	35.2	3.5	-92.4	285.8	-8.3	61.0	47.4
07SC32-6	52.5	1.4	16.6	9.8	0.1	6.3	8.2	3.2	1.7	0.3	873.7	17.3	325.6	122.9	1.7	155.1	146.8	104.2	35.0	3.8	-107.2	295.2	-11.9	65.3	46.7
07SC34-1	54.1	1.5	17.8	8.4	0.1	4.8	7.8	3.4	1.9	0.3	900.3	19.3	349.2	104.8	1.6	118.6	138.7	108.1	39.3	3.9	-75.7	242.7	-4.7	51.3	53.4
07SC34-2	53.5	1.4	16.9	9.2	0.1	5.8	8.0	3.2	1.7	0.3	890.3	17.8	331.5	115.6	1.6	143.2	142.1	102.3	35.9	3.5	-90.9	276.5	-7.1	59.3	47.8
07SC34-3	54.2	1.5	17.3	8.5	0.1	5.0	8.1	3.3	1.7	0.3	902.0	18.1	339.3	106.6	1.6	125.1	143.9	104.9	36.9	3.5	-90.3	249.8	-7.2	55.3	51.9
07SC34-4	53.9	1.5	17.2	9.4	0.1	4.9	7.7	3.3	1.8	0.3	897.0	18.6	337.4	117.5	1.8	120.3	137.3	104.9	38.0	3.8	-80.1	256.5	-4.9	54.4	50.4
07SC34-5	54.1	1.5	17.5	8.2	0.1	5.1	8.2	3.2	1.8	0.3	900.3	18.6	343.3	102.7	1.4	126.1	146.6	103.9	37.2	3.7	-90.9	247.4	-7.6	55.0	52.6
07SC34-6	53.4	1.5	16.8	9.6	0.1	4.8	7.4	4.3	1.8	0.3	888.7	18.1	329.5	120.4	2.0	120.1	132.3	138.8	38.0	3.5	-111.8	258.6	-16.7	60.5	56.1
07SC35	51.7	1.4	15.6	9.8	0.1	6.5	7.0	5.9	1.8	0.3	860.4	17.0	306.0	122.1	1.7	161.3	125.2	190.7	38.6	3.5	-173.7	300.4	-37.7	77.4	60.3

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Part 1 – Petrography and mineral geochemistry

Preamble

Micas are known to be good recorders of magmatic processes in granites and pegmatites from different types of environments. In W-Sn deposits, micas are ubiquitous and are often observed as several successive generations among pre-, syn- and post-mineralizing stages. The aim of this first part of manuscript is to give an insight of the potential of micas as tracers of hydrothermal processes when integrated into detailed paragenesis. Chapter 2 is devoted to the study of the Maoping deposit, located in the southern part of the Jiangxi province in SE China. In this chapter, we aim to provide a complement to fluid inclusions studies by the study of the evolution of chemical compositions of micas through the emplacement of the deposit. These micas are highly enriched in lithium and raised the problematic of measuring lithium content in hydrothermal system through the Tischendorf regressions. This chapter states that these regressions are not adapted to this kind of environment and proposes an alternative method to correct the bias induced by these regressions. Chapter 3 is devoted to the study of the Piaotang deposit, located in the same district as the Maoping deposit. This chapter aims to test and compare our previous observations and interpretations at the Maoping deposit and raise the possibility of common processes involved at the district scale.

Chapter 2: Detailed paragenesis and Li-mica compositions as recorders of the magmatic-hydrothermal evolution of the Maoping W-Sn deposit (Jiangxi, China)

Article published in Lithos 264 (2016) 108-124.

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Abstract

Li-micas have been used as indicators of the evolution of granites. However, hydrothermal Li-micas are less documented. World-class W-Sn deposits associated with Early Yanshanian granites (South Jiangxi, China) show magmatic and hydrothermal Li-micas which could help unravelling the magmatic-hydrothermal evolution of rare metal deposits. Six types of Li-micas have been identified in the vein system of the Maoping W-Sn deposit through detailed petrography and EPMA and LA-ICP-MS analyses, by chronological order: (i) late-magmatic Li-micas in feldspar veins, associated with late crystallization of a peraluminous melt; (ii) hydrothermal Fe-Li micas (Fe-Li mica veins and selvages); (iii) hydrothermal Fe-Li micas in W-Sn veins; (iv) Fe-Li micas in later banded quartz veins (v) Li-muscovite in the final stages; and finally (vi) micas associated with alteration at each stage. Based on oscillatory variations and trends in major elements composition, the chemical variations in Li-micas from the successive stages and in hydrothermal micas that crystallized in the veins are interpreted

to reflect mixing between at least three fluids of possible magmatic, meteoric and metamorphic origins. The crystallization of zircons and REE minerals, combined with variations of major and trace element concentrations in the Li-micas, notably an enrichment of rare metals (W-Sn-Ta-Nb) in the Li-micas, implies emplacement of a hidden peralkaline REE-rich magma during the crystallization of the banded quartz veins, a source which was different to the pre-existing peraluminous granites. The possible involvement of both peraluminous and peralkaline intrusives suggests the existence of polyphase magmatic-hydrothermal systems in the Maoping deposit, during the Yanshanian event (190-80 Ma).

Keywords

Maoping – Yanshanian – W-Sn deposit – Lithium-micas – REE – Magmatic-hydrothermal

1. Introduction

Li-micas have been widely used as indicators of the magmatic evolution of evolved granites and pegmatites within peraluminous or peralkaline series (Tischendorf *et al.*, 1997 and references therein). However, their use in hydrothermal systems is poorly documented. Moreover, most studies that have used magmatic or hydrothermal micas for this purpose were based on the characterization of only one object and/or on a comparison of chemical compositions with other case studies. Compositional trends are well documented for both magmatic trioctahedral (from iron-biotite to zinnwaldite) and dioctahedral (from Li-muscovite to lepidolite) micas (Tischendorf *et al.*, 1997 and references therein; Alfonso *et al.*, 2003; Roda-Robles *et al.*, 2006; Roda *et al.*, 2007; Vieira *et al.*, 2011). A few examples of hydrothermal alteration affecting magmatic Li-micas have been described (Johan and Johan, 2001; Van Lichtenvelde *et al.*, 2008; Johan *et al.*, 2012) and a Li-muscovite-bearing episyenite has been identified in Brazil (Costi *et al.*, 2002). A number of publications have described the post-magmatic evolutions of Li-micas (Henderson *et al.*, 1989; Cuney *et al.*, 1992; Neiva *et al.*,

2012; Neiva *et al.*, 2013; Marchal *et al.*, 2014; Jin-Ung and Kideok, 2015). Hydrothermal lepidolite is observed at Yellowstone (Bargar *et al.*, 1973) and F-rich micas have been described from acid leaching alteration in the Henderson Mo deposit (Gunow *et al.*, 1980). Finally, hydrothermal Li-micas in W-Sn deposits have been documented only in a few occurrences in Portugal (Neiva, 1987).

Li-micas are common in W-Sn deposits, which are usually emplaced during multiple magmatic and hydrothermal events that affect granites and their country rocks (Giuliani, 1985; Tischendorf, 1997; Costi *et al.*, 2002; Johan *et al.*, 2012; Neiva, 2013). Here, we show that the compositional variations of Li-micas can be correlated with the detailed paragenesis in order to determine both magmatic and hydrothermal influences within a single system and allow the identification of multiple magmas and fluid interactions throughout the history of a rare metal deposit. The Maoping Sn-W deposit in the southern Jiangxi Metallogenic Province (South China) provides an opportunity to (i) characterize magmatic and hydrothermal Li-micas of various compositions present at the different stages of the paragenetic sequence defined in the veins, (ii) relate the micas to the compositions of and interactions between the hydrothermal fluids responsible for the deposition of wolframite and cassiterite, and (iii) to use, for the first time, the evolution in hydrothermal Li-mica composition through the paragenetic stages to characterize the fluids involved in terms of timing and interactions as well as to characterize the magmatic influences present within the system.

2. Geological setting and sampling

2.1. The southern Jiangxi W-Sn Metallogenic Province

The southern Jiangxi W-Sn Metallogenic Province of southern China is located in the Nanling range in the central part of the Cathaysia Block, which is separated from the Yangtze Block to the northwest by the Jiangshan-Shaoxi fault zone (Figure 19) (Wang *et al.* 2013 and references therein). The Cathaysia Block is a former Paleoproterozoic craton which stabilized

at around 1.85-1.78 Ga (during the Nuna supercontinent amalgamation) and which includes Archean nuclei (presently identified in the lower crust; Xu et al., 2016). The Cathaysia and Yangtze blocks were sutured together during the Neoproterozoic Jiangnan (Sibao) orogeny (at around 1.0-0.83 Ga), coeval with the amalgamation of the Rodinia supercontinent, leading to the formation of the South China Craton (Charvet, 2013 and references therein). The Cathaysia Block underwent strong Phanerozoic reworking (i.e. metacratonization, Liegeois et al., 2013), involving tectono-metamorphic events and granite emplacement in the upper crust linked to lithospheric delamination processes. Consequently, the present day Sub-Continental Lithospheric Mantle (SCLM) is interpreted to be a “Tecton” signature inherited from the strong metasomatism of the Archean depleted mantle (Li et al., 2014; Liu et al., 2012). Three main orogenies contributed to the metacratonization: the Kwangsi (465-400Ma), the Indosinian (260-200Ma) and the Yanshanian (190-80Ma).

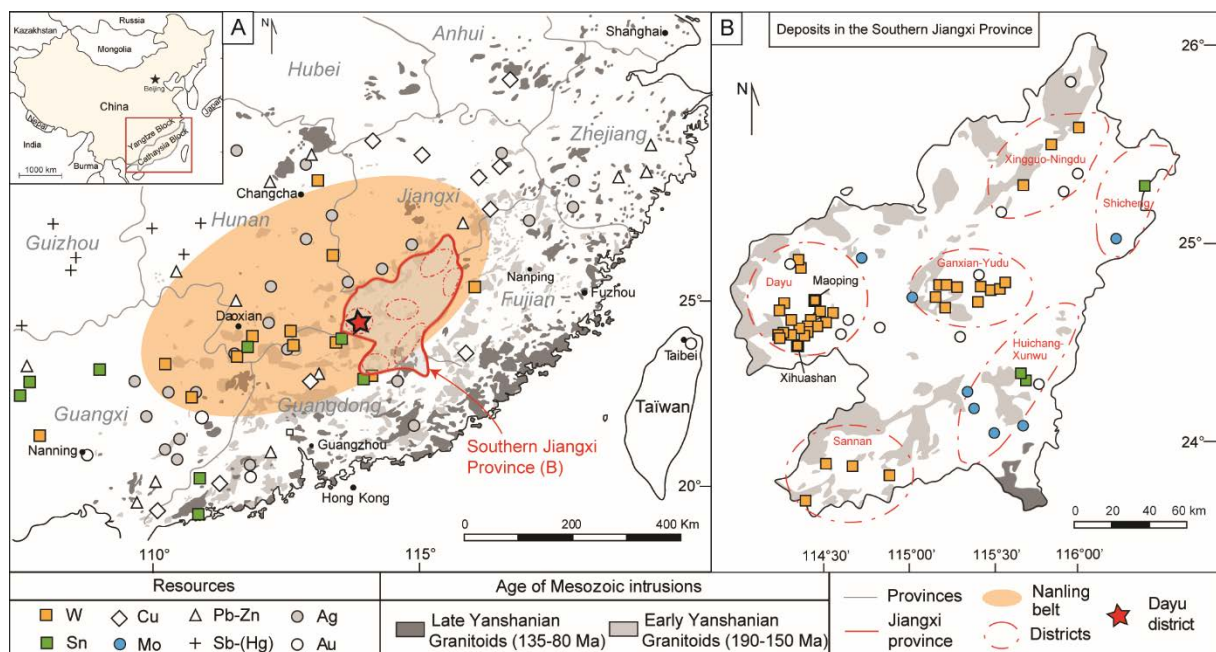


Figure 19. (A) Ore mining districts and metal deposits of the Cathaysia Block and (B) detailed map of the southern part of the Jiangxi Province in South-East China, showing the distributions of Early Yanshanian granitoids (peraluminous dominant) and Late Yanshanian granitoids (peralkaline dominant). The Maoping deposit is located in the Dayu district in the south-western part of the Jiangxi province. (A: modified and compiled from Sun et al. (2012), Jiang et al. (2006), Mao et al. (2006), Wu et al. (1993) and Wang and Fan (1987). B: modified after Huang et al., 2014 and Lecumberri-Sanchez et al., 2014)).

The Kwangsi orogen is interpreted to be the result of either the inversion of the Neoproterozoic to Early Paleozoic Nanhua rift (Charvet, 2013) or intracontinental subduction (Faure et al., 2009) under an overall Paleotethyan tectonic regime (Wang et al., 2012). The orogen was associated with pervasive emplacement of peraluminous granites derived from partial melting of the Cathaysia Block crust (Xia et al., 2014). The Indosinian event, which overprinted the Kwangsi orogen, yielded both metaluminous and peraluminous granites, the latter associated with Nb-Ta mineralization in the Nanling range (Mao et al., 2013) at around 390 Ma, according to the ages of Nanping pegmatites (U-Pb on columbite-tantalite; Che et al., 2015). The Indosinian orogeny was characterized in particular by the formation of metamorphic core complexes (Faure et al., 1996; Shu et al., 2008), and the orogeny might be considered a far-field effect of the collision between the South and North China Cratons (Wang et al., 2003; Shu et al., 2008).

After the late Triassic, the history of Cathaysia was dominated by the oblique subduction of the Paleo-Pacific (Izanagi) plate beneath the South China Craton (e.g., Zhou *et al.* 2002). This resulted: (i) in two extensional episodes (basin and range tectonics): the early (190-150 Ma) and late (135-80 Ma) Yanshanian events, separated by a seemingly compressional episode at the Jurassic-Cretaceous boundary (Shu *et al.* 2009, Liu *et al.* 2012, Li *et al.* 2014) and (ii) in the emplacement of the giant Yanshanian igneous province (up to 1300 km wide), which spreads across the whole of the Cathaysia and into the Yangtze Block, and is one of the largest granite provinces in the world (~ 180,000 km²; Chen *et al.* 2008).

The Early Yanshanian event, associated with flat slab subduction followed by slab foundering (Li and Li, 2007; Chen *et al.* 2008, Wang *et al.* 2013), was by far the most significant event in terms of both granite surface area (~ 100,000 km²) and the density of W-(Sn) occurrences and deposits (including the Maoping deposit) at 160-150 Ma. In particular, three E-W granite belts define the “Nanling Metallogenic Belt”, which includes most of the largest W-(Sn) deposits of Southern China (Figure 19). With a total of 429 deposits amounting to 1.7 Mt

WO₃, the southern Jiangxi Province hosts 90% of the Chinese tungsten resources (Feng *et al.*, 2011; Zeng *et al.*, 2007).

Whereas peraluminous granites represent the most abundant evidence for the Early Yanshanian event, the Late Yanshanian magmatic activity produced many highly potassic calc-alkaline and peralkaline and metaluminous (A-type) granitic massifs. The latter mainly outcrop along the Southeast China Coastal Volcanic Belt but are also found in other parts of Cathaysia. In the western Nanling Range, a major Sn-(W)-polymetallic mineralization event was associated with the Late Yanshanian granites (100-80 Ma) (Hu and Zhou, 2012). The Late Yanshanian event may have been the result of a major change in the subduction vector of the Izu plate and the transition from extension to transpression in the Cathaysia domain (Li *et al.*, 2014).

2.2. The Maoping deposit

The Maoping deposit (Hu *et al.*, 2011) hosts 63,000 t of WO₃ and 15,000 t of Sn, with average grades of 0.93% WO₃ and 0.3% Sn (Feng *et al.*, 2011). Maoping is one of the many large W-Sn deposits found in the southern Jiangxi Province, which also hosts the world-class Xihuashan deposit (Giuliani, 1985; Guo *et al.*, 2012).

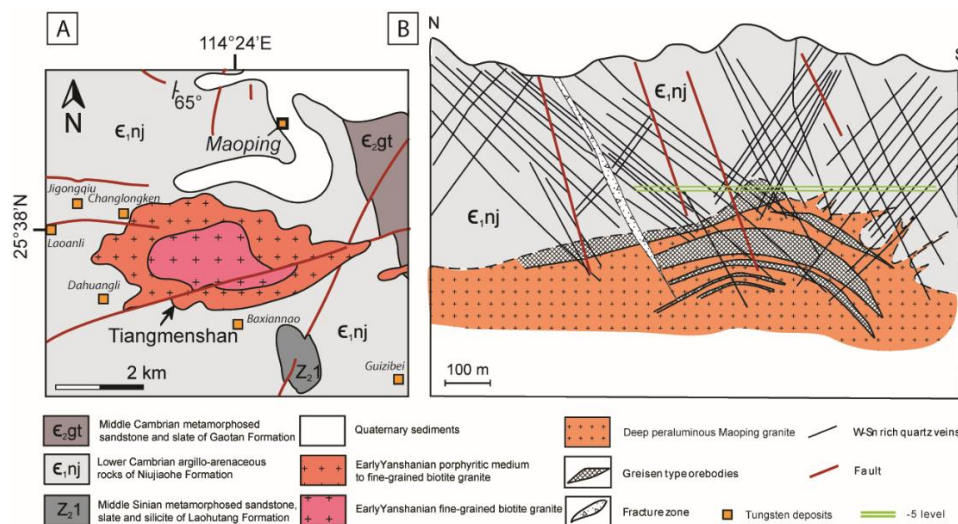


Figure 20. Location (A) and cross-section (B) of the Maoping W-Sn deposit, showing in particular the relationships between the deep granite, Lower Cambrian host rocks, greisenized granite and W-Sn rich quartz veins, as well as the sampling zone (-5 level, in green) (modified after Feng *et al.*, 2011).

The Maoping deposit is made up of more than 400 W-Sn-bearing quartz-dominated veins, each between 5 and 100 cm thick and up to 400 m in length, hosted by Cambrian metamorphosed sandstones and schists. The veins are associated with a buried porphyritic rare metal peraluminous granite that extends for about 4 km in an E-W direction and is thought to be the north-plunging extension of the Early Yanshanian Tianmengshan peraluminous granite intrusion (Feng *et al.*, 2011) (Figure 20). The granite cupola contains greisen-type disseminated W-Sn orebodies in its apical part. The veins have been described as radially distributed relative to the granite by Feng *et al.*, (2011) (Figure 20), although when observed at a depth of 200 m (i.e. at the sampling level), the veins are quite shallow dipping (Figure 21). According to Feng *et al.* (2011), the main ore-bearing faults are E-W trending and northward or southward dipping from 50° to 75°, with the mineralized veins dipping from 40° to 82°. Still according to Feng *et al.* (2011), the vein mineralogical assemblage gradually evolves from quartz-W-Mo-(Sn) veins within the greisen to W-Sn-sulphide veins above the granite and eventually to an upper fluorite-muscovite veinlet zone.

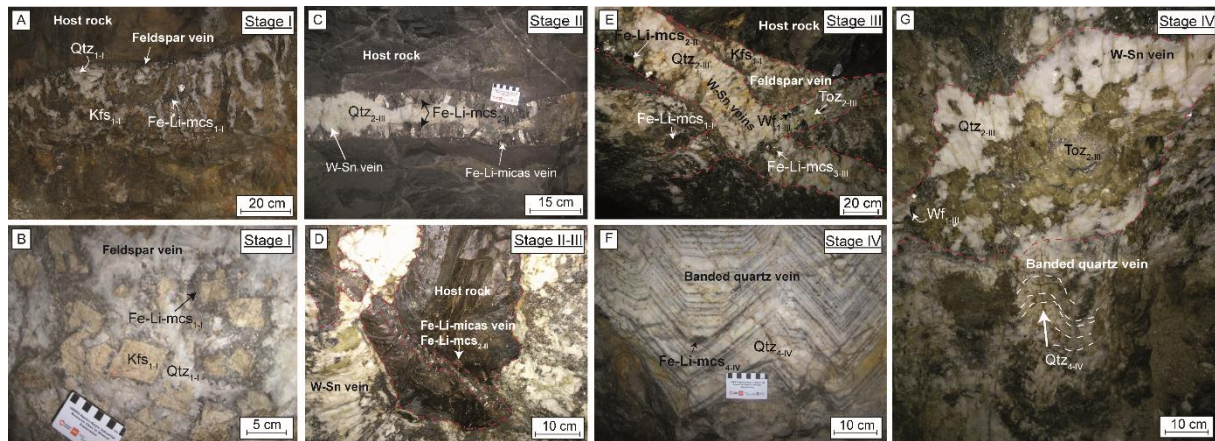


Figure 21. Relationships between vein types and paragenetic stages in the Maoping deposit. (A) Feldspar vein (type I) crystallized at stage I. (B) Transversal section of the Feldspar vein, showing euhedral K-feldspar crystals. (C) Fe-Li-mica vein (type II) reopened and filled with quartz from stage III. (D) W-Sn vein (type III) cross-cutting the Feldspar vein. (E) W-Sn vein at stage III, cross-cutting a Fe-Li-mica vein. (F) Banded quartz vein (type IV) showing the centimeter-scale banding of quartz and Fe-Li-micas at stage IV. (G) Crystallization of the banded quartz vein at stage IV, invading the W-Sn vein shown in 3E. IMA abbreviations: Qtz = Quartz; Fe-Li-mcs = Fe-Li-micas; Kfs = K-feldspar; Toz = Topaz; Wf = Wolframite.

The Maoping rare metal granite has been dated at 151.8 ± 2.9 Ma (SHRIMP U-Pb on zircon, Feng *et al.*, 2011), and the deep greisen bodies and the W-Sn-bearing veins at 155.3

± 2.8 Ma and 150.2 ± 2.8 Ma, respectively (Re-Os on molybdenite, Feng *et al.*, 2011). The respective timings of granite emplacement, greisen and W-Sn deposition are therefore indistinguishable within error.

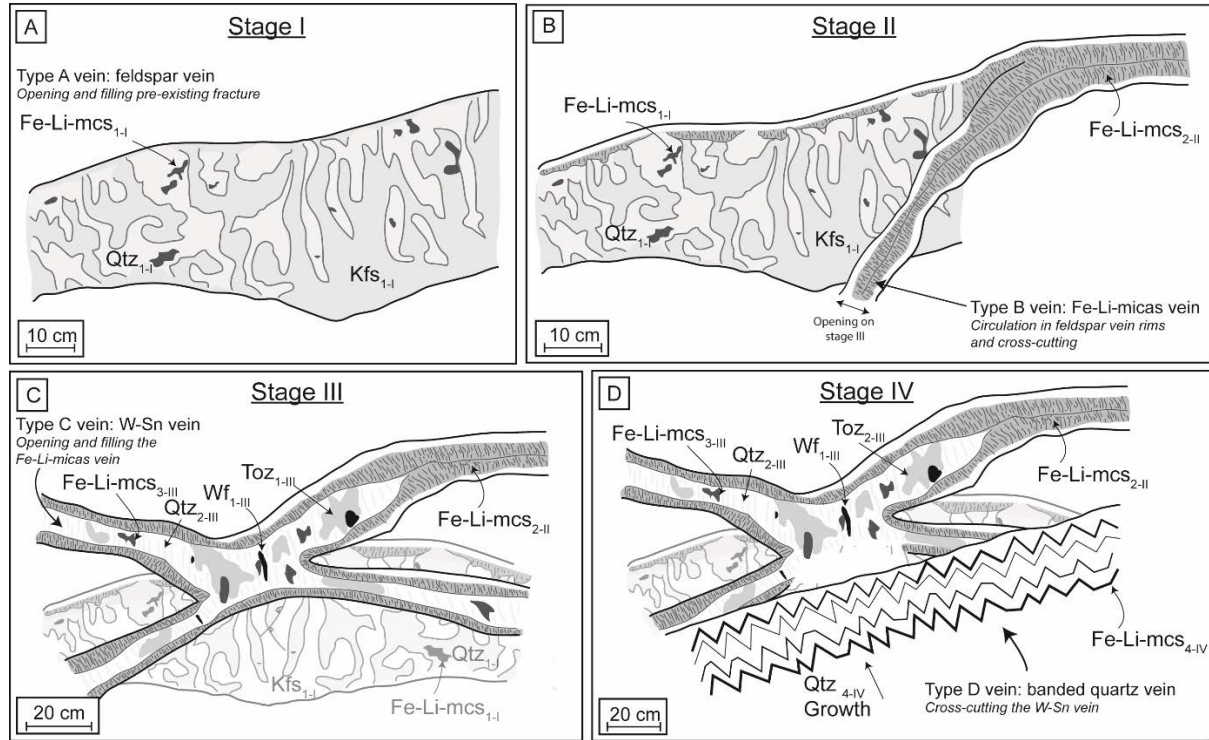


Figure 22. Sketches showing the evolution of the veins through time according to the paragenetic sequence observed at the Maoping deposit. (A) Sketch of a feldspar vein (type-I), emplaced at stage I. (B) Sketch of a feldspar vein (type-I) cross-cut by a Fe-Li mica vein (type-II) emplaced at stage II. (C) Sketch of conjugated type-III veins, emplaced by reopening of a type-II vein during stage III, cross-cutting a type-I vein. (D) Sketch showing type I to IV veins and the cross-cutting relations between them. Abbreviations according to the IMA: Qtz = Quartz; Fe-Li-mcs = Fe-Li-micas; Kfs = K-feldspar; Toz = Topaz; Wf = Wolframite.

2.3. Sampling

Thirty-five samples from both greisen-type and W-Sn-rich quartz vein orebodies were collected at the -5 level of the mine (at approximately 200 m depth), where the relationships between the host-rock, greisen and veins can be observed and the mineral assemblages are well-developed. Four types of veins, as defined by cross-cutting relationships, were sampled. The mining walls made sampling difficult in some areas due to the hardness of the rocks (see below, the feldspar veins for example). Unfortunately, the buried peraluminous granite could

not be sampled *in situ* and was therefore picked from the mine dumps. The origin of these granite samples was certified by the local miners.

3. Analytical methods

All preparations and analyses described below were carried out at the GeoRessources laboratory (Nancy, France).

Petrographic data were obtained from observations of polished thin-sections using conventional transmitted and reflected light microscopy and a HITACHI FEG S4800 scanning electron microscope (SEM) equipped with an energy dispersive spectrometer (EDS), using a Si(Li) semi-conductor detector. Cathodoluminescence (CL) was also performed on thin-sections, using a CITL cold cathode instrument CL8200 Mk4 (15 kV and 400 mA).

Electron microprobe analyses (EPMA) of Si, Al, Fe, Mn, Mg, Ca, Na, K, Ti, Rb and F were performed on Li-bearing micas using a CAMECA SX100 instrument equipped with a wavelength dispersive spectrometer (WDS) and calibrated using natural and synthetic oxides and silicates (albite, olivine, Al_2O_3 , orthose, andradite, MnTiO_3 , hematite, cassiterite, RbTiPO_5 , topaz, scheelite, LiTaO_3 and LiNbO_3). A current of 12 nA and an accelerating voltage of 15 kV was used for silicates (20kV and 20nA for W and Sn) with a counting time of 10 s per element. Special attention was paid to the fluorine measurements and a spectrometer was used for this element only (average detection limit of 1300 ppm). Total Fe is presented as FeO.

Major, minor and trace elements (^7Li , ^{28}Si , ^{39}K , ^{43}Ca , ^{85}Rb , ^{88}Sr , ^{89}Y , ^{93}Nb , ^{118}Sn , ^{133}Cs , ^{181}Ta , ^{182}W) were measured in Li-bearing micas using a LA-ICPMS instrumental setup similar to that described in Leisen et al. (2012) and Lach et al. (2013), with a GeoLas excimer laser (ArF, 193 nm, Microlas, Göttingen, Germany) (Günther et al., 1997) and an Agilent 7500c quadrupole ICP-MS. The laser beam was focused onto the sample within a 24.5 cm^3 cylindrical ablation cell using a Schwarzschild reflective objective (magnification $\times 25$; numerical aperture 0.4) mounted on an optical microscope (Olympus BX41) equipped with a X–Y motorized stage

and a CCD camera. Silica (determined by EPMA) was chosen as an internal standard as it is abundant in both the samples and the external standards and can be accurately quantified by EPMA. NIST610 (values from Jochum et al., 2011) was chosen as the external standard (accuracy verified with NIST612) (Longerich et al., 1990). The following parameters were used for the LA-ICPMS analyses: a fluence of 8J/cm² and a laser shot frequency of 5 Hz; He = 0.5 L.min⁻¹ as a carrier gas, mixed with Ar = 0.7 L.min⁻¹ via a cyclone mixer prior to entering the ICP torch; spot sizes from 44 µm to 120 µm (depending on the grain size); and an ablation duration of 40 s. Data was processed using the Iolite software (Paton et al., 2011).

4. Results

4.1. Paragenetic sequence

The paragenetic sequence was constructed using cross-cutting relationships and the textures of granite, greisen and vein infillings in the mine galleries, as well as petrographic observations made in the laboratory. Four types of veins (I to IV) and seven successive paragenetic stages were identified, separated by plastic deformation, corrosion/dissolution and vein reopening events (Figure 23).

Some minerals from the veins were found to occur repeatedly (e.g. up to 5 generations for quartz) whereas others occurred within only one stage (e.g., the W-Sn minerals). Minerals are hereafter referred to by their recommended IMA mineral abbreviations, their generation number (1 to 5, from the oldest to the youngest) and the paragenetic stage (I to VI, from the oldest to the youngest) to which they belong (e.g., Cst_{1-III} stands for the first generation of cassiterite in the whole paragenetic sequence, occurring during stage III, when veins III were emplaced).

The primary granite mineralogy consists of quartz, albite, K-feldspar, Fe-Li mica and topaz. Most of the visible micas form symplectic associations with secondary quartz and developed at the expense of feldspar and sometimes of topaz. The micas contain inclusions

of fluorite rimmed by Y-carbonate aggregates (possibly synchysite). Quartz grains show undulating extinction and micas exhibit kinking, indicative of a weak plastic deformation event (Figure 24A).

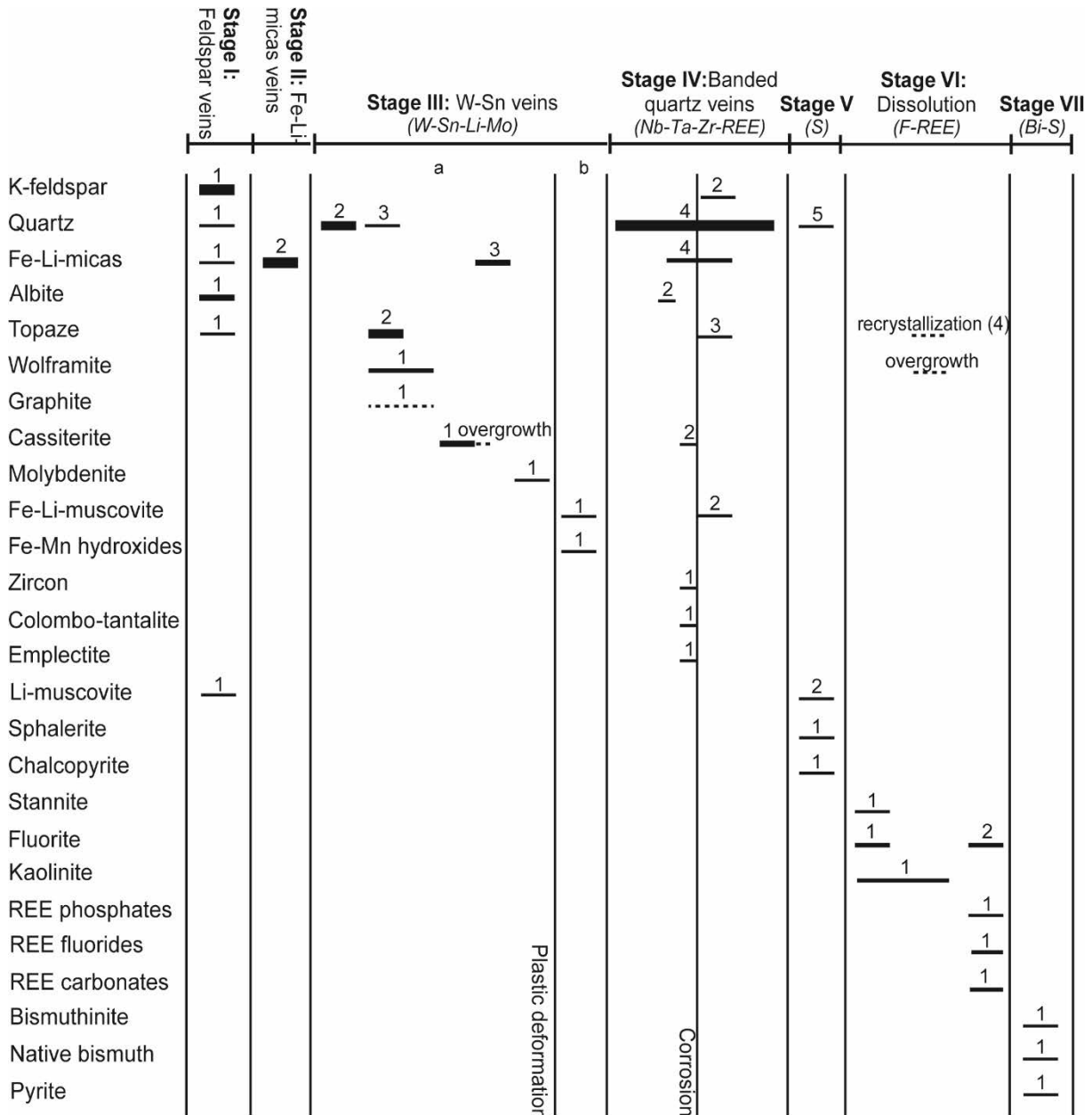


Figure 23. Paragenetic sequence of the Maoping W-Sn deposit based on observations and sampling conducted in mine gallery at level -5 (i.e. -200 m). See text and previous figures for details of the different stages, mineral abbreviations and notations.

The upper greisen body in the -5 level (i.e. -200 m) is a typical quartz-Fe-Li-mica-topaz greisen, with symplectic mica-quartz relationships. The micas contain late inclusions of fluorite-monzonite and uranothorite-monzonite assemblages. Undeformed kaolinite (developed at the

expense of topaz) and fluorite are also present (Figure 24B). Like the granite, the greisen underwent weak plastic deformation. The granite-greisen relationship could not be observed but all granite samples display evidence for incipient greisenisation, consistent with the development of the greisen at the expense of primary magmatic minerals in the peraluminous granite (Figure 20B).

Stages I to IV are associated with veining, whereas stages V and VI correspond to recrystallization, overgrowth, replacement of pre-existing minerals and cavity filling within the veins. Vein-types II and III are ubiquitous in the sampling area.

Stage I consists of the early formed type I veins (feldspar veins). These are half a meter thick and contain intergrown microcline (Kfs_{1-I}), quartz (Qtz_{1-I}) and Fe-Li-mica ($Fe-Li-mcs_{1-I}$). Feldspar crystals (up to 20 cm in size) are euhedral and show exsolution of $Ab_{1,I}$ and growth zones marked by Qtz_{1-I} , Toz_{1-I} and $Fe-Li-mcs_{1-I}$ (Figure 21A,B, Figure 22 and Figure 24C).

Stage II consists of the type II veins (Fe-Li-micas veins), which cross-cut the type I veins (Figure 22). The type II veins are composed of large (1 to 5 cm) feather-like Fe-Li-micas ($Fe-Li-mcs_{2-II}$; Figure 21C). The veins are sometimes crosscut by type III veins (Figure 21D) but are mostly reopened and filled with Qtz_{2-III} (Figure 21C, Figure 22 and Figure 24D).

Stage III consists of the ore-bearing type III veins (W-Sn veins). These are mostly sub-horizontal and tens of centimetres to a metre in thickness, and occur as conjugated sets (Figure 21E, Figure 22 and Figure 24F). Some type III veins are clearly observed in the field as having formed from reopening of the type II veins. The stage III minerals are quartz (Qtz_{2-III}), wolframite (Wf_{1-III}), cassiterite (Cst_{1-III}), topaz (Toz_{2-III}), Fe-Li-micas ($Fe-Li-mcs_{3-III}$), molybdenite (Mlb_{1-III}), Fe-Li-muscovite ($Li-Fe-ms_{1-III}$) and Fe-Mn hydroxides. Evidence for plastic deformation within the type III veins allows two successive mineral assemblages (IIIa and IIIb) to be distinguished.

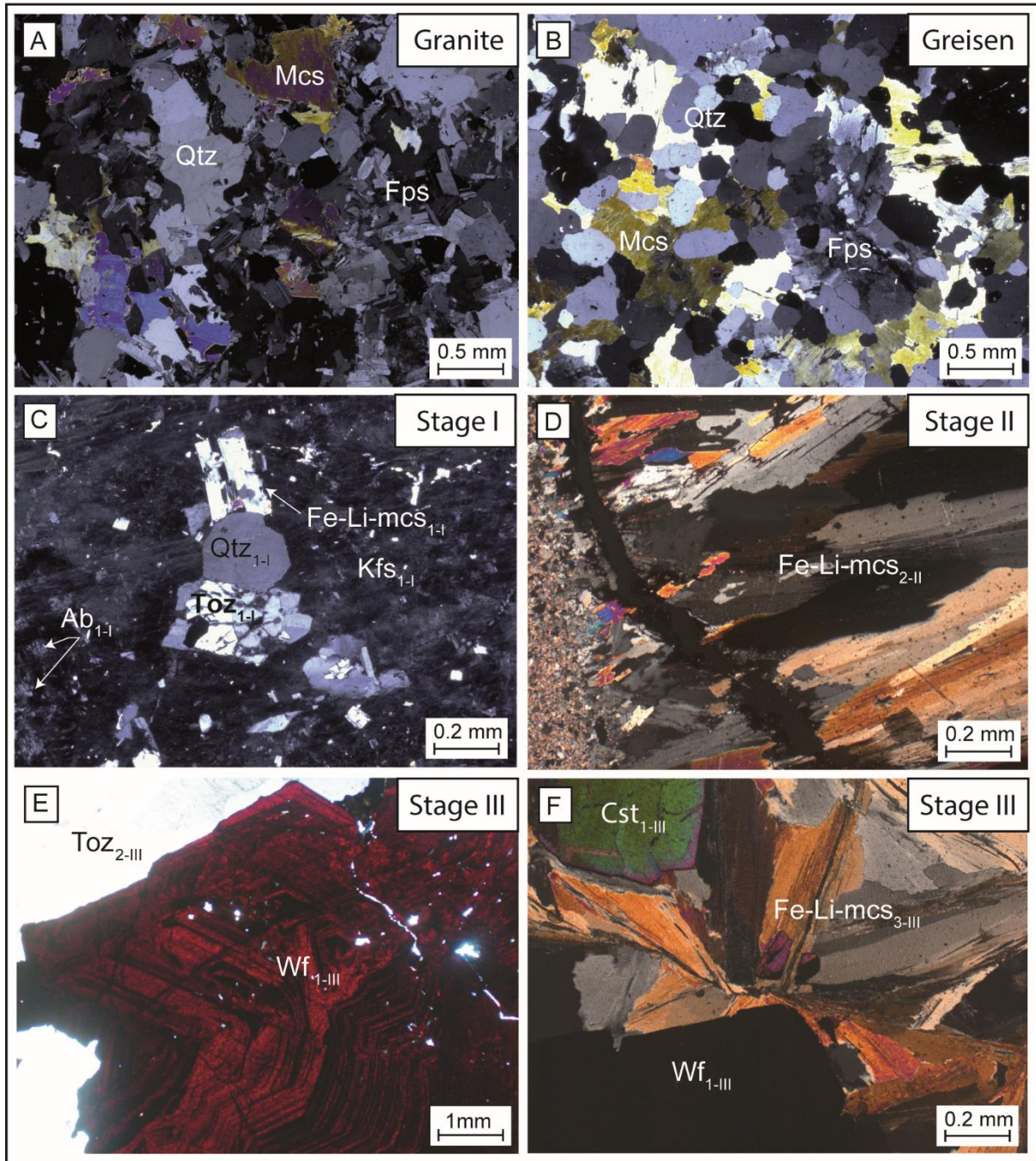


Figure 24. Mica textures and associations from the granite, greisen and stages I to III (type I to type IV veins). (A) Deformed quartz, corroded micas and euhedral feldspars from the granite (crossed polars). (B) Quartz sub-grains, corroded micas and altered feldspars from the greisen (crossed polars). (C) Qtz1-I, Toz1-I and Fe-Li-mcs1-I observed in centimetric Kfs1-I from stage I (type-I vein) (crossed polars). (D) Feather-like centimetric Fe-Li-mcs2-II and junction with the host rock from stage II (type-II vein) (cross polars). (E) Complex zoning in the Mn-rich wolframite Wf1-III of stage III (type-III vein) (natural light). (F) Fe-Li-mcs3-III from stage III associated with the crystallization of Wf1-III and Cst1-III (crossed polars). Abbreviations according to the IMA: Ab = albite; Cst = Cassiterite; Fe-Li-mcs = Fe-Li-micas; Fps = feldspars; Kfs = F-feldspar; Mcs = micas; Qtz = quartz; Toz = topaz; Wf = Wolframite.

During stage IIIa, quartz (Qtz_{2-III}), topaz (Toz_{2-III}), wolframite (Wf_{1-III}), cassiterite (Cst_{1-III}), Fe-Li-micas (Fe-Li-mcs_{3-III}) and molybdenite (Mlb_{1-III}) were successively deposited. Qtz_{2-III} and Toz_{2-III} are the most abundant minerals. Topaz, wolframite, cassiterite and Fe-Li-micas fill dissolution cavities in the Qtz_{2-III}. Wf_{1-III} is blood red under transmitted light microscopy and is seen to be porous in the SEM images. Both Wf_{1-III} and Cst_{1-III} are euhedral and show complex zoning under transmitted light microscopy and by cathodoluminescence (CL; Figure 24E). However, no compositional variation was found using SEM or EPMA. The wolframite (Wf_{1-III}) is Mn-rich (17-18 wt.% MnO and 6-7 wt.% FeO, with the following structural formula: Fe_{0.25}Mn_{0.75}WO₃; EPMA analyses). Graphite was locally observed by SEM and may be coeval with Wf_{1-III}. The Qtz_{2-III} and, to a lesser extent the Toz_{2-III}, exhibits an undulating extinction with the formation of lamellar neograins. At the quartz/wolframite contact, the deformed Qtz_{2-III} is overgrown by smaller (~100 µm) non-deformed euhedral Qtz_{3-III} crystals. Plastic deformation is also demonstrated by the kinking of Li-mcs_{3-III} and Mlb_{1-III} (Figure 25B). Stage IIIb is marked by limited replacement of the previously kinked Fe-Li-micas by Li-Fe-ms_{1-III} and Fe-Mn hydroxides.

Stage IV is represented by the type IV veins (banded quartz veins), which consist of meter-scale euhedral Qtz_{4-IV} crystals growing perpendicular to the sub-horizontal to sub-vertical walls. The crystals are zoned at the centimeter-scale, defining a broad banding (Figure 21F). Type IV veins clearly invade the type III veins (Figure 21G and Figure 22), with undeformed Qtz_{4-IV} growing onto the plastically deformed Qtz_{2-III}. The macroscopic banding formed by repeated episodes of accumulation of small aggregates of quartz and Fe-Li-mica crystals on corroded quartz surfaces, followed by quartz precipitation and dissolution (Figure 26). These aggregates have undergone plastic deformation (kinking of the Fe-Li-micas), and the Fe-Li-micas have been partially replaced along the cleavage planes by Li-Fe-ms and Fe-Mn hydroxides and therefore likely represent fallen fragments of the overprinted type III veins. Together with these aggregates, inherited anhedral and plastically deformed quartz grains (likely equivalent to Qtz_{2-III}) are observed.

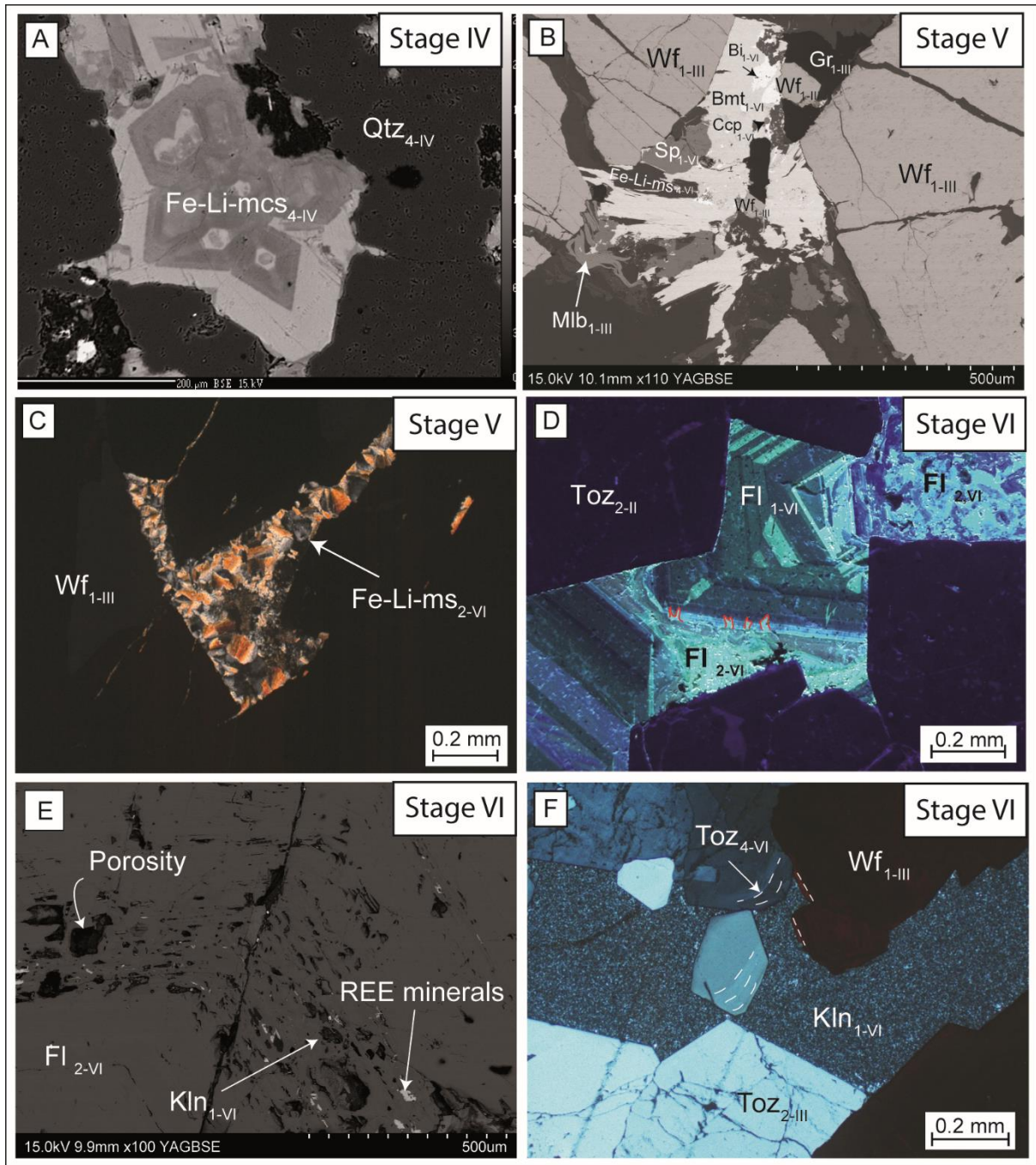


Figure 25. Mica textures and associations from stages IV to VI (type III and IV veins). (A) Zoning and rhythmic banding in Fe-Li-mcs_{4-IV} from stage IV (type-IV vein) (SEM). (B) Sulphide assemblages from Stage VII (SEM). (C) Fe-Li-ms_{2-VI} filling the cavities of a type-III vein from stage V (crossed polars). (D) Two generations of fluorite (FI_{1-VI} and FI_{2-VI}) from Stage VI (CL). (E) Zoning in FI_{2-VI} marked by yttrium content, porosity and abundance of REE minerals (SEM). (F) Overgrowth and zoning textures in Toz_{4-VI} and Wf_{1-III} in contact with kaolinite (Kln_{1-VI}) (crossed polars). Abbreviations according to the IMA: Bi = native bismuth; Bmt = Bismuthinite; Ccp = chalcopyrite; Fe-Li-mcs = Fe-Li-micas; Fe-Li-ms = Fe-Li-muscovite; FI = fluorite; Gr = graphite; Kln = kaolinite; Mlb = molybdenite; Qtz = quartz; Sp = sphalerite; Toz = topaz; Wf = Wolframite.

These have partially recrystallized, although the original grain boundaries have been preserved. In the accumulation zones, newly-formed oscillatory zoned Fe-Li-micas (Fe-Li-mcs_{4-IV}; Figure 25A) containing sporadic zircon and columbo-tantalite inclusions are found. The Qtz_{4-IV} growth appears to have been sequential, starting from a corroded quartz surface with the growth of small (0.5-1 mm) subhedral crystals of Qtz_{4a-IV} (Fig.8B), which are subsequently overgrown by a band of “massive” Qtz_{4b-IV} (several mm to cm wide), in turn followed by a band of laminated Qtz_{4c-IV} (several mm to cm, with 100 to 200 µm wide laminae). Small (~ 100 µm) euhedral crystals of pure albite (Ab_{2-IV}) and unzoned Fe-Li-mcs_{4-IV} also alternate with the Qtz_{4c-IV} laminations. In addition to the inherited Qtz_{2-III} and sequentially grown Qtz_{4abc-IV}, millimetric anhedral to subhedral quartz crystals (Qtz_{4d-IV}) overprint the quartz banding (Figure 26A). The Qtz_{4d-IV} is sometimes zoned by Fe-Li-micas (Fe-Li-mcs_{4-IV}) as well as by very small rounded topaz (Toz_{3-IV}) and K-feldspar (Kfs_{2-IV}) crystals, the latter being mostly found in the outermost (most recent) growth zones.

Stage V is characterized by Li-muscovite (Li-ms_{2-V}) as veinlets or infilling cavities (Figure 25C), forming microspherulites, associated with rare quartz (Qtz_{5-V}), sphalerite (Sp_{1-V}) and chalcopyrite (Ccp_{1-V}) (Figure 25B), all occurring in all vein cavities. The sphalerite has been affected by chalcopyrite disease (Barton, 1978). In contrast to stages I to IV, stage V is not associated with any vein emplacement.

Stage VI corresponds to a pervasive dissolution stage, which affects topaz (Toz_{2-III}) in type III veins in particular, and to an overprinting of stage V minerals, also observed in the type IV veins. Stage VI minerals consist of a succession of fluorite (Fl_{1-VI}), kaolinite (Kln_{1-VI}) and a late porous fluorite (Fl_{2-VI}) that contains numerous inclusions of 10- to 200-µm-sized REE minerals (xenotime (Xnt), monazite (Mnz) and unidentified REE-bearing fluorocarbonates, the latter most abundant in the Fl_{2-VI} cores) (Figure 25E). The REE-mineral-hosting fluorite (Fl_{2-VI}) exhibits oscillatory zoning marked by elevated yttrium concentrations (up to 3.8 wt.% Y).

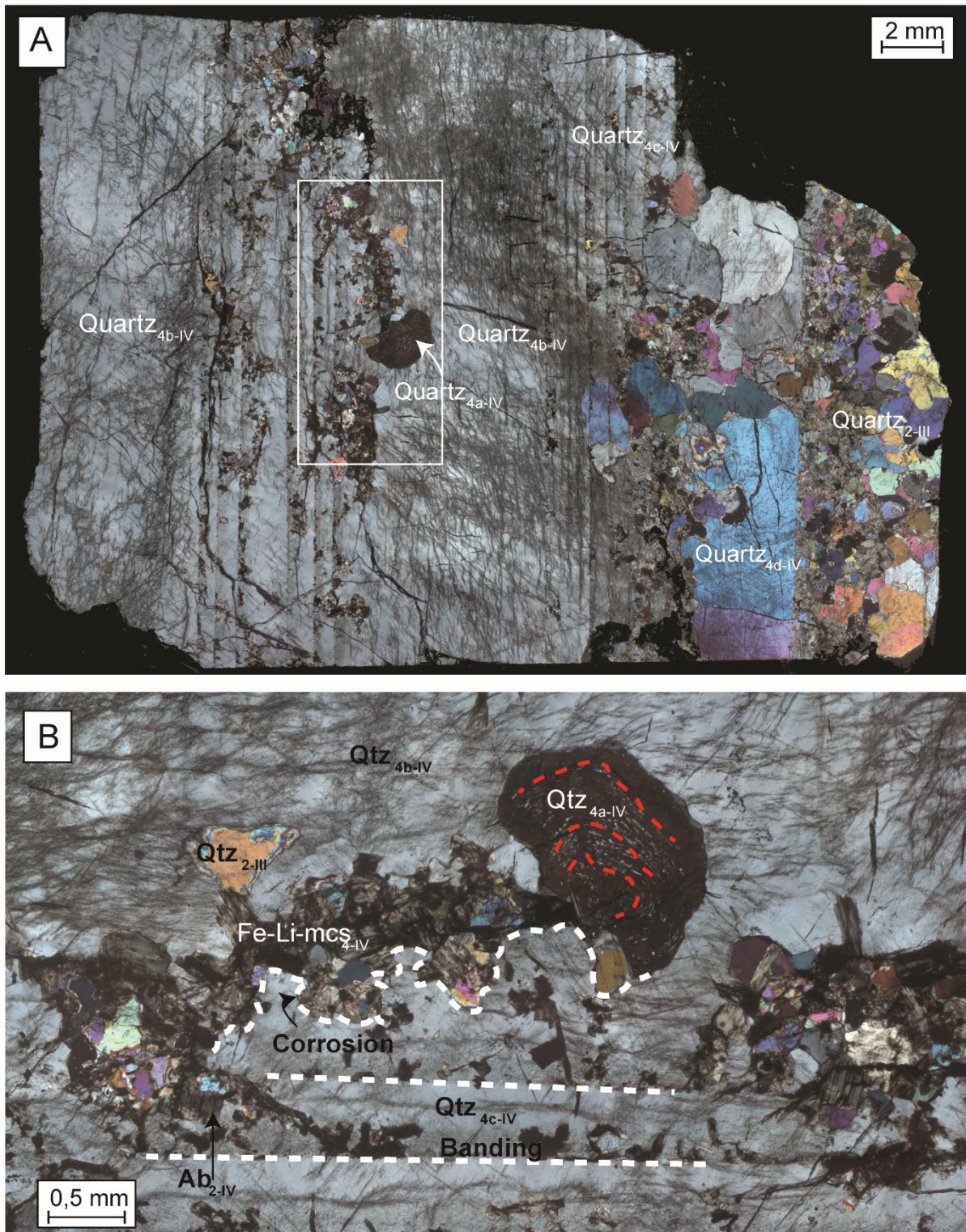


Figure 26. Stage IV banded quartz-Fe-Li-mica veins. (A) Banding in type-V veins highlighted by Qtz3B,IV, Fe-Li-mcs4-IV and albite (Ab2-IV) (thick section observed under crossed polars). (B) Close-up view of the banding and corroded quartz surface composed of an accumulation of quartz and Li-micas. See text for explanation.

Sporadic stannite can be found on Ccp_{1-V} rims in contact with fluorite (Fl_{1-VI}) or disseminated within Kln_{1-VI}. Dissolution cavities within Toz_{2-III}, when filled by Kln_{1-VI}, are marked by a rim of euhedral and zoned Toz_{4-VI} crystals of more than 100 µm in size (Figure 25F). EPMA analysis shows that this oscillatory zoning in Toz_{4-VI} is controlled by a variation in the F content, from 9.3 wt.% to 11.3 wt.%. The calculated mean structural formula for Toz_{4-VI} is Al_{1.16}Si_{1.13}O₄(F_{1.32},OH_{0.68}). At the contact with Kln_{1-VI}, Wf_{1-III} also presents an overgrowth rim, which has a similar composition to the core (Figure 25F). However, when filled by fluorite, no overgrowths are observed in the Wf_{1-III} dissolution cavities. The Fl_{2-VI}, however, has clearly corroded and replaced the euhedral and zoned Fl_{1-VI} (Figure 25D). Of note, a similar sequence of late fluorites has been described in the neighbouring Xihuashan W deposit (Wang *et al.*, 2003).

Stage VII corresponds to an assemblage of bismuthinite (Bmt_{1-VII}), which contains native bismuth (Bi_{1-VII}) inclusions, and pyrite with no evidence of dissolution. This assemblage is observed in both type III and type IV veins.

In conclusion, the paragenetic sequence records the succession of three major element assemblages: W-Sn-Li-Mo in stage III; Nb-Ta-Zr-P-REE in stage IV; and S-F-Y-P-REE in stages V to VII (Figure 23).

4.2. Composition of Li-micas

A total of 351 EPMA and 67 LA-ICP-MS analyses were performed on the eight generations of Li-micas from veins as well as from the granites and the greisen. EPMA analyses were systematically performed on each sample. The spot size used in the LA-ICP-MS analyses (44-120µm) was too large to allow analysis of the secondary micas observed in the cleavage and precise positioning in micro-zoned micas (only homogeneous cores and rims were analysed). Representative analyses are given in Table 1 and Table 2. Given the difference in spot size between the two methods, major and trace elements analyses could not be performed at the same location. As a result, the lithium LA-ICP-MS analyses are not

presented in the major element diagrams but were instead estimated from the EPMA major element data following a regression described in Tischendorff *et al.*, (1997).

4.2.1. Li_2O estimations

When plotted in the Monier and Robert (1986) Al-R^{2+} -Si diagram (Figure 27A), all micas are seen to be Li-micas (i.e. zinnwaldite, trilithionite), as is consistent with their elevated fluorine contents (up to 3.7 wt.% F in EPMA analyses) (Tischendorff *et al.*, 1997). Two groups of micas can be distinguished in Figure 27, one close to zinnwaldite in composition and the other richer in silica and trending towards a more aluminous composition. As Li contents cannot be directly measured by EPMA, the calculation of complete structural formulas for the Maoping micas is problematic.

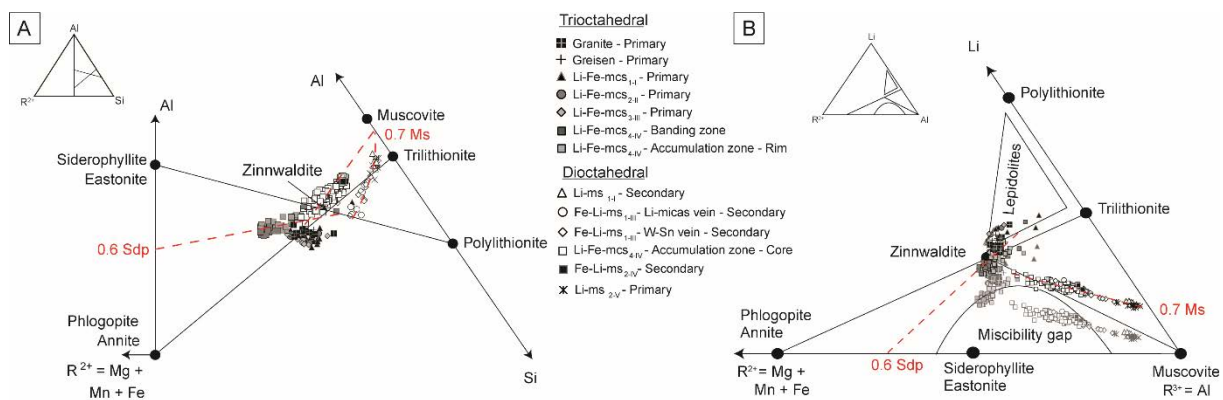


Figure 27. (A) Compositions of micas plotted on a Monier and Robert (1986) phase diagram as a function of R^{3+} (Al), R^{2+} (Fe+Mg+Mn) and Si (apfu). (B) Compositions of micas plotted on a Monier and Robert (1986) diagram as a function of R^{3+} (Al), R^{2+} (Fe+Mg+Mn) and Li (apfu). The dashed red lines indicate the extensions of the trends towards the end-members. Trends were drawn to ensure consistency between the diagrams. Uncorrected data are shown in semi-transparency. See text for an explanation of the estimations and corrections of the Li content.

Using a large database of separate mica analyses, Tischendorff *et al.* (1997) provided a series of empirical regressions that yield calculated Li_2O contents (also noted Li_2O^*) as a function of either SiO_2 , F, Rb_2O and MgO for the trioctahedral micas or F and Rb_2O for the dioctahedral micas. In a SiO_2 vs. F plot (Figure 28A), the Maoping micas display two trends, allowing their separation into two groups. A negative trend is observed when F contents are lower than ~4 wt.% whereas a positive slope is observed when F contents are greater than ~4 wt.%. These two groups can similarly be distinguished in the $\text{FeO}+\text{MgO}+\text{MnO}$ vs. F plot (Figure 28B), with

a positive trend observed for FeO+MgO+MnO contents below ~13 wt.% and a negative trend observed when FeO+MgO+MnO contents are greater than ~13 wt.%. The two groups thus correspond to di- and trioctahedral-like structures, respectively.

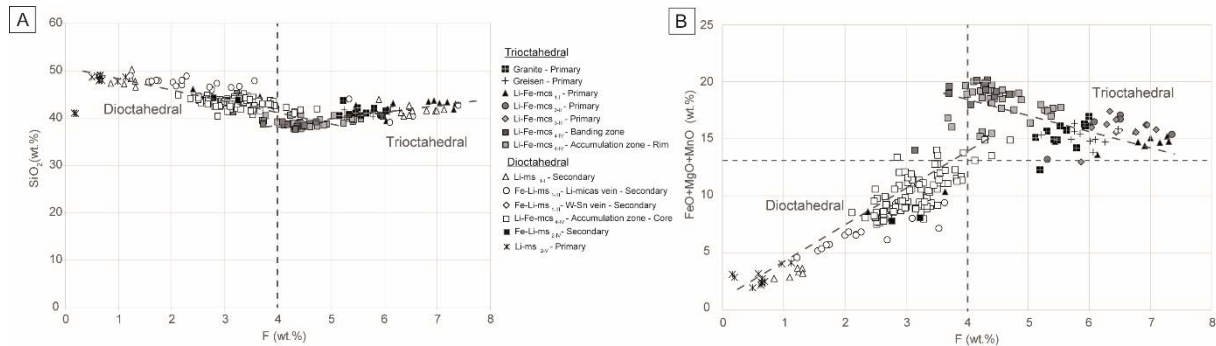


Figure 28. Discriminant composition diagrams for trioctahedral and dioctahedral micas from the Maoping W-Sn deposit. (A) F-SiO₂ covariation. (B) F and (FeO+MgO+MnO) diagram. See EPMA data in Table 1.

Li₂O contents were first estimated using SiO₂ regression for the dominantly trioctahedral micas ($\text{Li}_2\text{O} = 0.298 \times \text{SiO}_2 - 9.658$) and F regression for the dominantly dioctahedral micas ($\text{Li}_2\text{O} = 0.395 \times (\text{F}^{1.326})$). However, for each regression proposed by Tischendorf *et al.* (1997), when plotted in the Monier and Robert (1986) R²⁺-Al-Li diagram, most compositions fall inside the miscibility gap defined experimentally by Monier and Robert (1986) (Figure 27B). Because Li₂O* therefore appears to be systematically underestimated using this method, a correction needs to be applied to Li₂O* in order to correctly estimate the Li₂O contents. In Figure 9, the Fe-Li (trioctahedral) micas trend towards an intermediate composition between annite and siderophyllite (~0.6 siderophyllite mole fraction), while the dominantly dioctahedral micas trend towards an intermediate composition between muscovite and trilithionite (~0.7 muscovite mole fraction). With these constraints, the best fit in the R²⁺-Al-Li diagram is achieved by adding 1.5 wt.% to all Li₂O* values in order to compensate for the bias (Figure 27B). The underestimation might be due to the mainly magmatic database used by Tischendorf *et al.*, (1997), which would therefore require adjustment for hydrothermal systems.

PART 1 – Chapter 2

Table 1. Selected representative major oxide contents from EPMA analyses and calculated structural formulas for micas from each stage, greisen and granite. Li₂O contents were calculated following Tischendorf et al., (1997). Values preceded by the symbol '<' indicate element concentrations below the limit of detection.

EPMA	Feldspar vein (I)		Fe-Li-micas vein (II)		W-Sn vein (III)		Banded quartz vein (IV)				Late stage (V)		Granite	Greisen
wt%	Primary	Secondary	Primary	Secondary	Primary	Secondary	Banding zone	Accumulation - Rim	Accumulation - Core	Secondary	Secondary	Primary	Primary	
	Fe-Li-mcs _{1-I}	Li-ms _{1-I}	Fe-Li-mcs _{2-II}	Li-phg _{1-III}	Fe-Li-mcs _{3-III}	Li-phg _{1-III}	Fe-Li-mcs _{4-IV}	Fe-Li-mcs _{4-IV}	Fe-Li-mcs _{4-IV}	Li-phg _{2-IV}	Li-ms _{2-V}			
SiO ₂	43.13	47.27	42.69	46.04	40.72	48.01	39.18	39.9	44.62	44.28	48.09	41.16	41.71	
TiO ₂	bdl (<0.04)	bdl (<0.04)	0.04	bdl (<0.04)	0.04	bdl (<0.04)	0.04	0.08	bdl (<0.04)	bdl (<0.04)	bdl (<0.04)	bdl (<0.04)	0.06	
Al ₂ O ₃	21.97	33.16	22.2	27.10	23.2	28.84	23.75	23.72	29.01	30.08	33.28	23.21	22.56	
FeO	10.43	1.59	11.28	6.42	12.85	4.56	17.12	15.31	7.93	6.79	1.49	11.23	11.5	
MnO	4.32	1.08	3.73	1.89	3.76	1.03	1.73	2.17	1.1	0.98	0.11	3.69	4.35	
MgO	bdl (<0.04)	bdl (<0.04)	0.36	0.42	0.06	0.95	bdl (<0.04)	bdl (<0.04)	bdl (<0.04)	bdl (<0.04)	0.88	0.05	bdl (<0.05)	
CaO	bdl (<0.07)	bdl (<0.07)	bdl (<0.07)	bdl (<0.07)	bdl (<0.07)	bdl (<0.07)	bdl (<0.07)	bdl (<0.07)	bdl (<0.07)	bdl (<0.07)	0.1	bdl (<0.07)	bdl (<0.07)	
Na ₂ O	0.08	0.20	0.02	0.06	0.24	0.05	0.05	0.14	0.23	0.26	0.06	0.12	0.19	
K ₂ O	10.43	10.25	10.45	10.99	9.58	10.31	10.02	10.07	10.55	10.57	10.22	10.77	9.93	
Rb ₂ O	0.7	0.39	0.74	0.42	1.07	0.42	0.94	0.88	0.57	0.56	0.21	0.97	0.86	
Li ₂ O *	4.31	1.68	4.18	2.11	3.61	1.95	3.17	3.37	2.28	2.11	1.60	3.74	3.90	
F	7.32	1.10	7.38	2.76	6.45	2.20	4.71	5.29	3.33	2.79	0.69	5.44	6.49	
O=F	6.66	0.55	3.69	1.38	3.22	1.10	2.36	2.64	1.66	1.39	0.35	2.72	3.24	
H ₂ O	2.60	4.26	2.43	3.68	2.57	3.94	3.06	2.84	3.57	3.69	4.41	2.78	2.59	
Total	100.13	100.45	100.25	100.01	99.57	100.72	100.46	100.04	100.85	100.13	100.72	99.92	99.54	
Atoms per 12 [O, OH, F]														
Si	2.98	3.13	3.07	3.18	2.98	3.22	2.82	2.92	3.07	3.05	3.16	3.00	3.03	
^{IV} Al	1.02	0.87	0.93	0.82	1.02	0.78	1.18	1.08	0.93	0.95	0.84	1.00	0.97	
Sum Tet.	4.00	4.00	4.00	4.00	4.00	4.00	4.00	4.00	4.00	4.00	4.00	4.00	4.00	
Ti	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	
^{VI} Al	0.78	1.73	0.95	1.39	0.98	1.51	0.83	0.97	1.42	1.49	1.73	0.96	0.96	
Fe	0.60	0.09	0.68	0.37	0.79	0.26	1.03	0.94	0.46	0.39	0.08	0.72	0.70	
Mn	0.25	0.06	0.23	0.11	0.23	0.06	0.11	0.13	0.06	0.06	0.01	0.24	0.27	
Mn #	0.29	0.40	0.24	0.21	0.22	0.14	0.10	0.12	0.11	0.13	0.05	0.25	0.28	
Mg	0.00	0.00	0.04	0.04	0.01	0.10	0.00	0.00	0.00	0.00	0.09	0.00	0.00	
Mg #	0.00	0.00	0.04	0.08	0.01	0.24	0.00	0.00	0.00	0.00	0.50	0.00	0.00	
Ca	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	
Li	1.20	0.45	1.21	0.59	1.06	0.53	0.91	0.99	0.63	0.59	0.42	1.09	1.14	
Sum Oct.	2.83	2.33	2.87	2.39	2.83	2.38	2.77	2.91	2.57	2.47	2.32	2.78	2.78	
K	0.92	0.87	0.96	0.97	0.89	0.88	0.92	0.94	0.93	0.93	0.86	1.00	0.92	
Rb	0.03	0.02	0.03	0.02	0.05	0.02	0.04	0.04	0.03	0.02	0.01	0.05	0.04	
Na	0.01	0.03	0.00	0.01	0.03	0.01	0.01	0.02	0.03	0.03	0.01	0.02	0.03	
Sum A	0.96	0.92	0.99	1.00	0.97	0.91	0.97	1.00	0.99	0.98	0.88	1.07	0.99	
F	0.80	0.12	0.84	0.30	0.75	0.23	0.54	0.61	0.36	0.30	0.07	0.65	0.75	
OH	1.20	1.88	1.26	1.70	1.25	1.77	1.46	1.39	1.64	1.70	1.93	1.35	1.25	

4.2.2. Crystal chemistry: substitution vectors

Lithium is incorporated into the mica structure through various substitution mechanisms involving both the octahedral and tetrahedral sites (Foster *et al.*, 1960). Of the many possible substitution trends (Roda Robles *et al.*, 2006), four have been identified in the Maoping Li-micas, two for trioctahedral structures and two for dioctahedral structures (Figure 29).

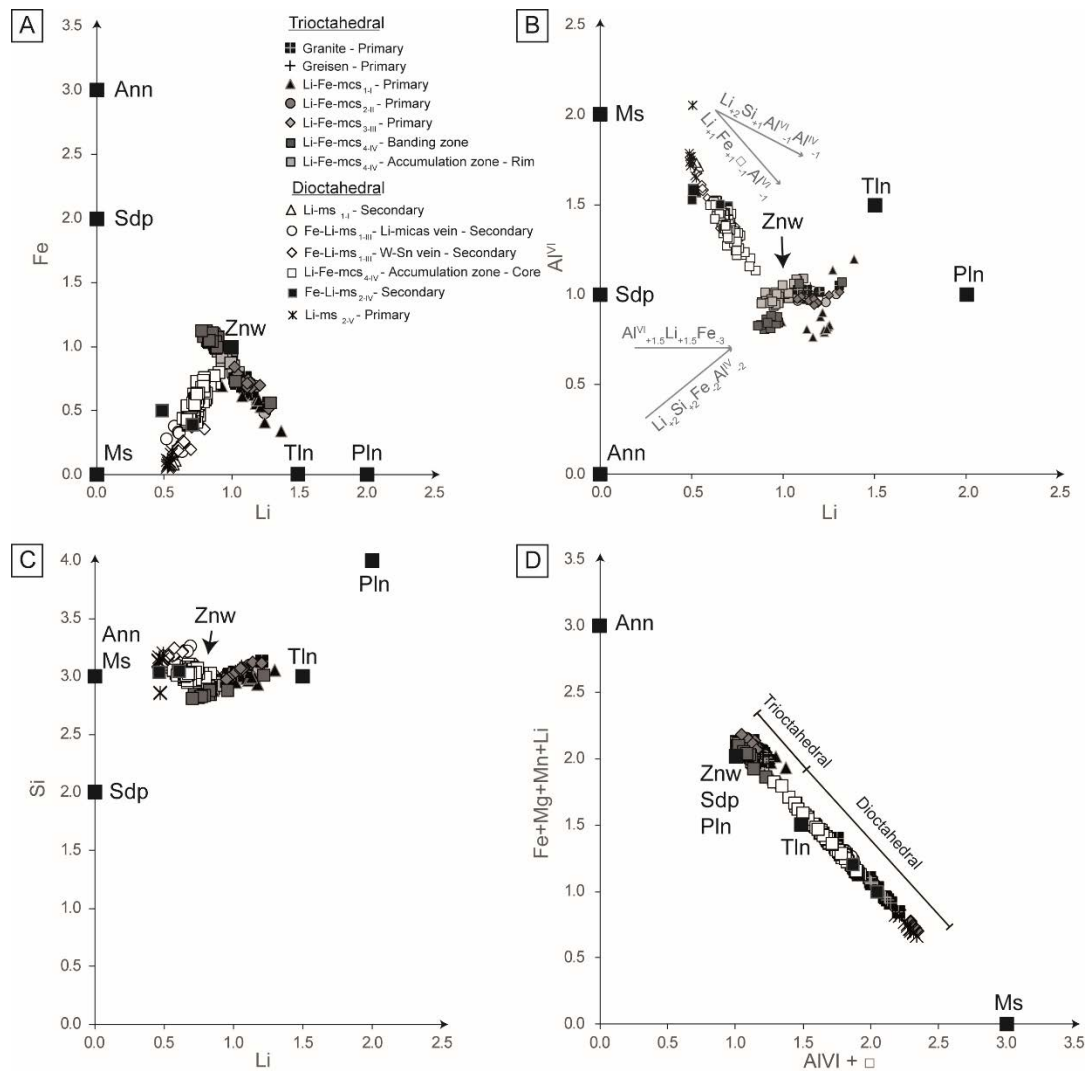


Figure 29. Substitution vectors for the trioctahedral and dioctahedral micas of the Maoping W-Sn deposit according to Fe, Li, AlVI, Si, Mg, Mn and vacancy (□) content in tetrahedral and octahedral sites. The data plot along four substitution vectors drawn on B, which are applicable to each diagram. Abbreviations according to the IMA: Ann = annite; Ms = muscovite; Pln = polyolithionite; Sdp = siderophyllite; Tln = trilithionite; Znw = zinnwaldite.

The trioctahedral mica compositions can all be explained by the linear combination of two vectors: $Al_{+1.5}Li_{+1.5}Fe_{-3}$ (annite towards trilithionite) in the octahedral site; and

$\text{Li}^{\text{VI}}_{+2}\text{Si}^{\text{IV}}_{+2}\text{Fe}^{\text{VI}}_{-2}\text{Al}^{\text{IV}}_{-2}$ (siderophyllite towards polyolithionite) in both tetrahedral and octahedral sites. As seen in Figure 29A and B, the resulting substitution vectors vary according to the mica generation. In particular, and unlike the other trioctahedral micas, the Li-Fe-mcs₁₋₁ (type I veins) appear to be mainly controlled by the annite-trilithionite vector. The dominantly dioctahedral compositions can also all be explained by the linear combination of two vectors: $\text{Li}_{+1}\text{Fe}_{+1}\square_{-1}\text{Al}_{-1}$ (muscovite towards zinnwaldite) in the octahedral site; and, as seen in particular in Figure 29C, $\text{Li}^{\text{VI}}_{+2}\text{Si}^{\text{IV}}_{+1}\text{Al}^{\text{VI}}_{-1}\text{Al}^{\text{IV}}_{-1}$ (muscovite towards polyolithionite) in both tetrahedral and octahedral sites. A linear alteration vector $\text{Fe}_{+1}\text{Mg}_{+1}\text{Mn}_{+1}\text{Li}_{+1}\text{Al}^{\text{VI}}_{-1}\square_{-1}$ can be applied to every generation of mica occurring in the deposit emplacement, as shown in Figure 29D. Except for the Li-ms_{2-v}, all dominantly trioctahedral micas in this study are primary whereas the dominantly dioctahedral micas were formed by alteration (secondary), mostly in the cleavage.

4.2.3. Compositional trends

In the R^{2+} -Al-Li diagram (Monier and Robert 1986) (Figure 27), three similar trends from zinnwaldite towards lepidolitic compositions (most of the micas on these trends being close to zinnwaldite in composition) are seen to characterize (i) the granite (and greisen) micas, (ii) the micas from type II and type III veins, and (iii) the micas from the type IV veins. As observed in Figure 29B, the type I vein micas exhibit a behavior that is distinct from all other generations. In the type II and III veins, the large Li-Fe-micas are zoned at the 100 μm scale, with compositions evolving from zinnwaldite (1) to lepidolite (2) from core to rim (Figure 30 and Figure 31).

In the type IV veins, the evolutions begin at zinnwaldite (1) (the small crystals along the banding to the oscillatory zoned crystals on the corrosion surface). Then, the zoning starts with Fe-rich Fe-Li-muscovite (3) and evolves (through numerous oscillations) towards Fe-depleted Fe-Li-muscovite, ending (after a break) at close to zinnwaldite compositions (1) (Figure 30 and Figure 31).

The secondary Fe-Li-muscovites and Li-muscovites from all stages follow the same trend as the primary Fe-Li-muscovite in the type IV veins, except that the end compositions are close to Li-muscovite (4), which is the composition of the late Li-ms_{2-V} (Figure 30).

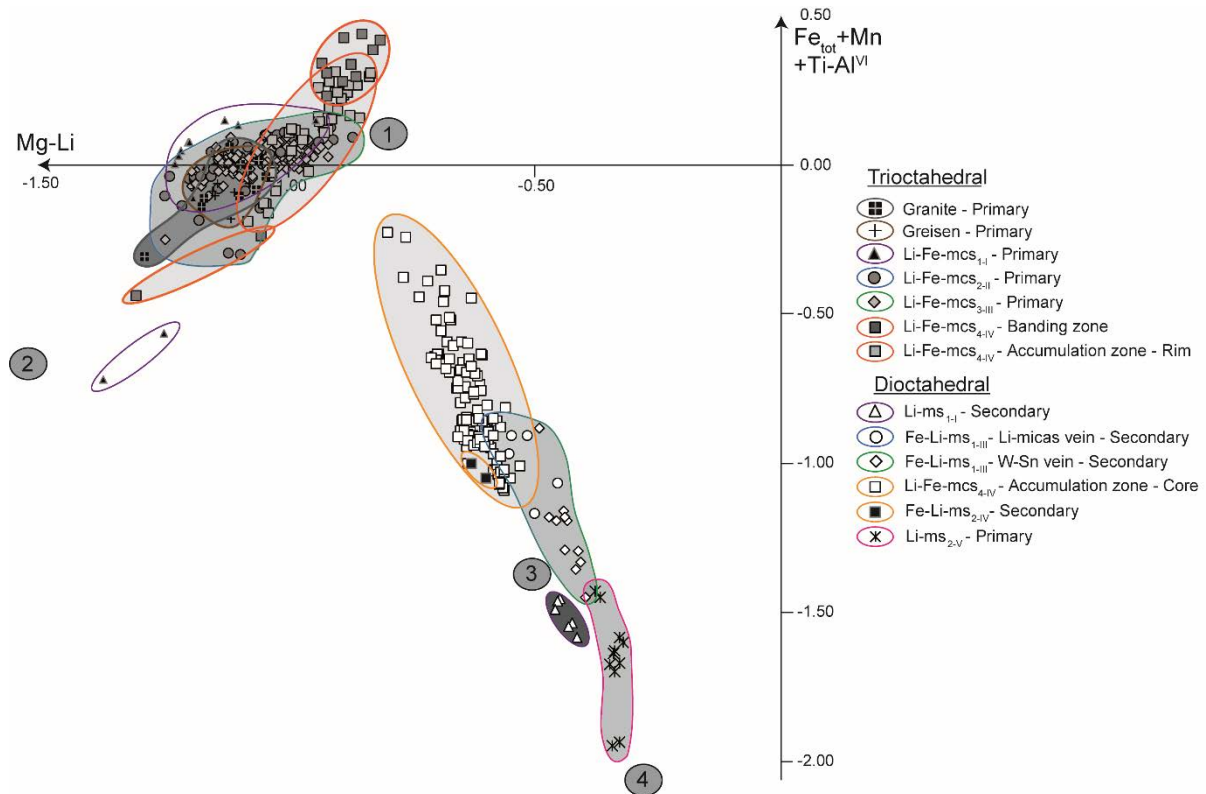


Figure 30. Compositions of micas presented in the Tischendorf et al., (1970) empirical diagram of Fe+Mn+Ti-AlVI vs Mg-Li. Each generation of micas displays an evolution trend that can be described by using up to four end-member compositions (1) to (4). Further explanations in the text.

4.2.4. Li-micas chemistry: trace elements

Trace element contents from LA-ICPMS analyses of the main types of micas in veins, granite and greisen are shown in Table 2 and Figure 32.

Most of the data was acquired from the trioctahedral Fe-Li-micas. Because of the large spot size (44 to 120 μm), it was not possible to analyze the Li-Fe-ms and Li-ms in the cleavages of the trioctahedral Fe-Li-mcs_{4-IV} located in the banding. Moreover, the only dioctahedral micas analyzed were the Fe-Li-micas_{4-IV} cores of the zoned micas in the banded quartz of the type IV veins. The other dioctahedral micas displayed were all located within the cleavage or small cavities.

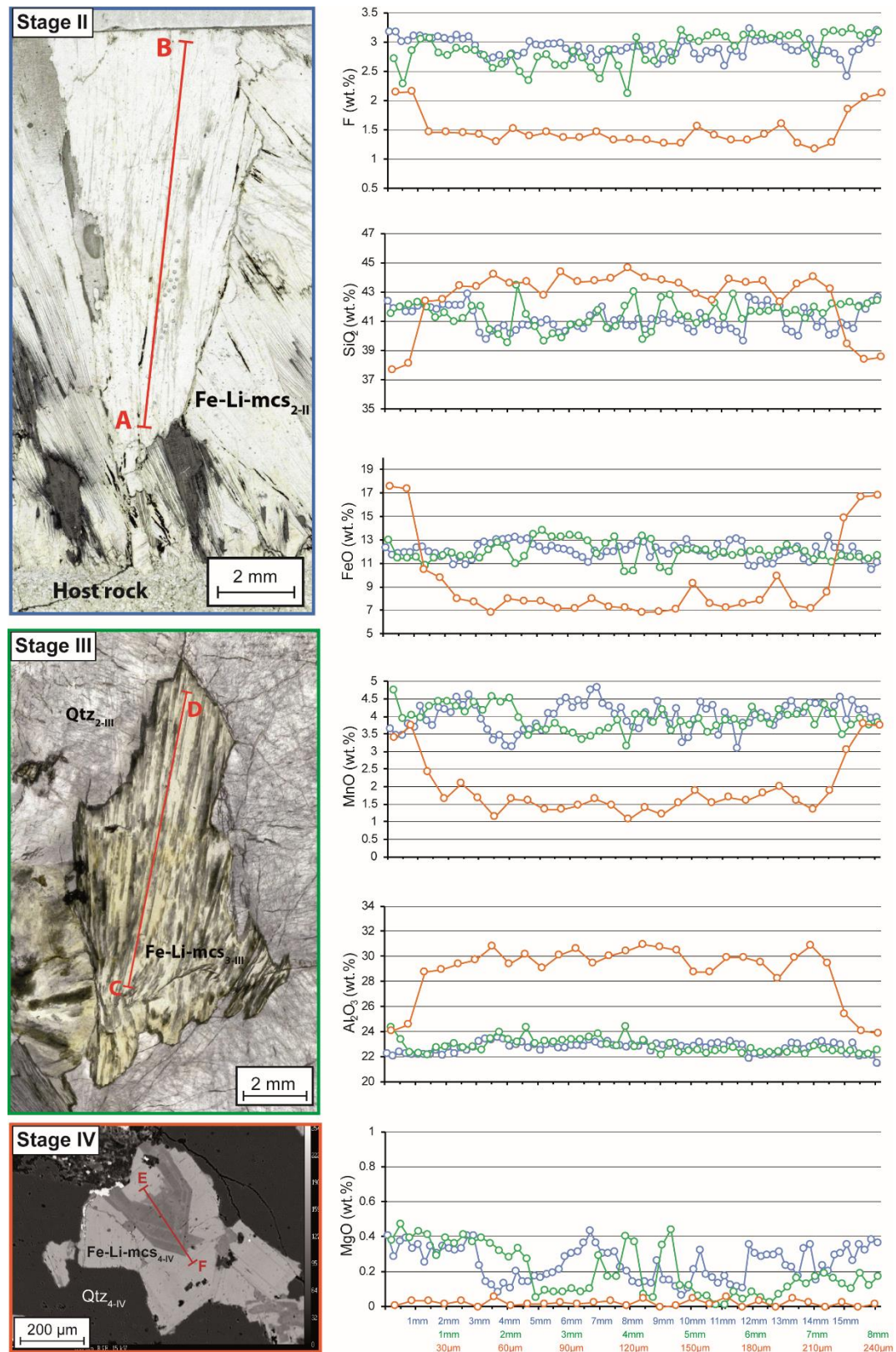


Figure 31. Compositional profiles of representative micas of stages II, III and IV. For stages II and III, the transmitted natural light microscopy image, and for stage IV the back-scattered-electron image, are provided together with profiles of F, SiO₂, FeO, MnO, Al₂O₃ and MgO. Concentrations are based on EPMA analyses ($\sigma_F=0.2\%wt$, $\sigma_{SiO_2}=0.6\%wt$, $\sigma_{FeO}=0.8\%wt$, $\sigma_{MnO}=0.2\%wt$, $\sigma_{Al_2O_3}=1.1\%wt$, $\sigma_{MgO}=0.5\%wt$).

The Maoping micas contain detectable amounts of Mg (82 to 2800 ppm), Ti (65 to 520 ppm), Nb (9 to 150 ppm), Sn (171 to 840 ppm), Cs (87 to 660 ppm), Ba (<1 to 110 ppm), Ta (6 to 200 ppm) and W (14 to 80 ppm). Zr, U, Y and REE were systematically below the detection limit (i.e. < ~1 ppm).

The trace element contents allowed identification of three broad compositional groups for the trioctahedral micas (Figure 32), mainly on the basis of their Nb, W, Sn and Ta contents: (i) the Nb-W-Ta-rich stage IV micas; (ii) the relatively Nb-W-Ta-poor micas from all I to III veins and the greisen; and (iii) the granite micas, with intermediate Nb-W-Ta contents. The stage I micas have the lowest total rare metal (W, Sn, Ta, Nb) contents and the stage III micas (W-Sn veins) exhibit the highest rare metal contents. In the binary diagrams in Figure 32B, however, the granite and stage IV micas tend to share similar compositions, except when lithium is involved, highlighting the differences with the relatively Nb-Ta-W-poor micas. In contrast with the latter, the granite and stage IV micas form a very wide positively-sloping W-Sn trend (Figure 32A). Moreover, whereas all micas form positive Ta-Nb (Figure 32B) and W-Nb trends, the granite and stage IV micas are characterized by very different Ta/Nb and W/Nb ratios. Although less well-defined, Sn and Nb also appear to be correlated. The stage IV di-octahedral micas differ significantly from the stage IV tri-octahedral micas, being poorer in Nb and Ta, but distinctly enriched in W and, moreover, Sn.

Table 2. Selected representative LA-ICP-MS analyses of trace elements in micas from each vein, greisen and granite. Y, Zr, Nd and U were also analyzed but were below the detection limit.

LA-ICPMS	Feldspar vein (I)		Fe-Li-micas vein (II)		W-Sn vein (III)		Banded quartz vein (IV)		Granite		Greisen	
(ppm)		σ		σ		σ		σ		σ		σ
Li ₂ O (wt.%)	5.9	0.1	3.7	0.1	3.8	0.04	3.1	0.1	5.0	0.05	3.9	0.05
Mg	309.0	4.2	2408.0	41.0	1380.0	13.0	152.7	2.6	227.7	2.1	2492.0	27.0
Ti	125.4	5.4	207.2	5.9	298.4	4.6	282.2	7.4	339.6	4.3	349.9	7.3
Nb	11.1	0.3	29.66	0.80	15.0	0.2	126.4	2.4	66.3	0.8	22.1	0.4
Mo	0.4	0.2	1.0	0.3	0.2	0.1	0.6	0.1	0.5	0.1	0.3	0.1
Sn	203.5	6.9	357.8	6.8	376.8	4.5	512.9	9.7	476.6	6.0	343.9	4.9
Cs	468.2	6.8	348.0	13.0	344.7	4.1	209.9	5.1	483.4	6.2	358.6	8.1
Ba	21.1	0.6	57.5	1.6	73.3	0.8	0.2	0.1	1.1	0.1	88.6	1.3
Ta	13.6	0.4	53.2	1.8	8.3	0.1	91.6	2.4	137.7	1.9	21.8	0.3
W	19.9	0.7	35.6	3.0	25.4	0.5	75.2	2.6	45.9	0.8	32.1	0.7

Covariations between W, Sn and Nb contents and Li contents reveal largely the same groups of Maoping tri-octahedral micas (W-Li is shown in Figure 32C as an example). The

micas from the granite form negative W-Li, Sn-Li, Nb-Li and Ta-Li (not shown) trends, whereas stage IV micas show no correlation. Weak but distinct positive correlations in W-Li, Nb-Li and Ta-Li (not shown) are observed for all micas from the greisen and veins II and III. The stage I micas systematically form a distinct group that lies along a prolongation of the trends defined by the granite micas. The greisen, stage II and stage III micas, however, seem to display independent trends, as was also seen in their major element compositional variations (Figure 30).

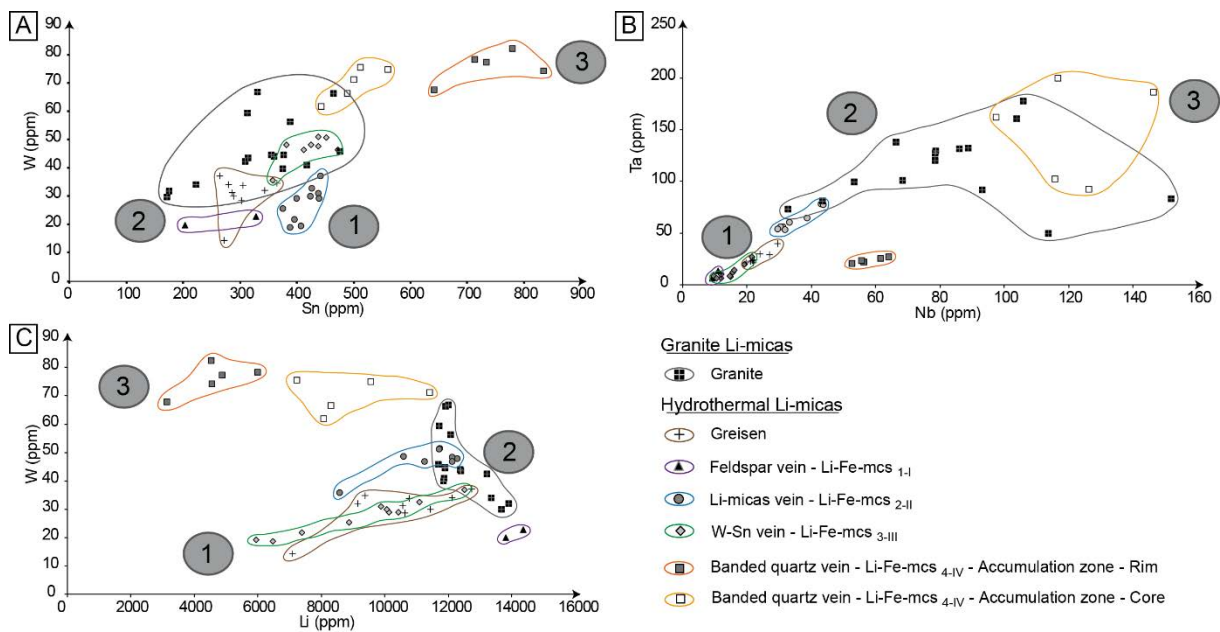


Figure 32. Trace element binary diagrams for seven generations of Li-micas in the Maoping deposit (data from LA-ICPMS analysis). (A) W-Sn, B- Ta-Nb, C- W-Li. For each generation, compositional trends are observed between up to four end-member compositions (1 to 4), which can easily be related to the four mica end-member compositions in Fig. 12. Further explanations in the text.

5. Discussion

5.1. Magmatic-hydrothermal Li-mica trends

The zinnwaldite to lepidolite (polylithionite-rich) solid solution observed in the peraluminous granite micas (Figure 30), along with, in particular, increase in the Mn/(Mn+Fe+Mg) ratio (Mn#) from 0.23 to 0.27 (Table 1) with increasing Li content can be interpreted as a magmatic fractionation trend along the liquid line of descent. The observed rare metal (W-Sn-Nb-Ta) depletion that accompanies this trend (Figure 32) can be interpreted

to result from an early fluid-melt separation in the granite system. Moreover, W-Sn and Ta (which correlates with Nb in this case) are known to partition into a chloride-rich magmatic fluid (e.g., Audétat et al., 1998; Marignac et al. 2003; Zhu et al., 2015). Given this interpretation, and in order to explain the extreme rare metal depletion of the Fe-Li-micas_{1-I}, which are also strongly fractionated (mean Mn# = 0.30 and high Li contents), the stage I feldspar veins can be considered to have resulted from residual crystallization of the granite magma (Figure 33).

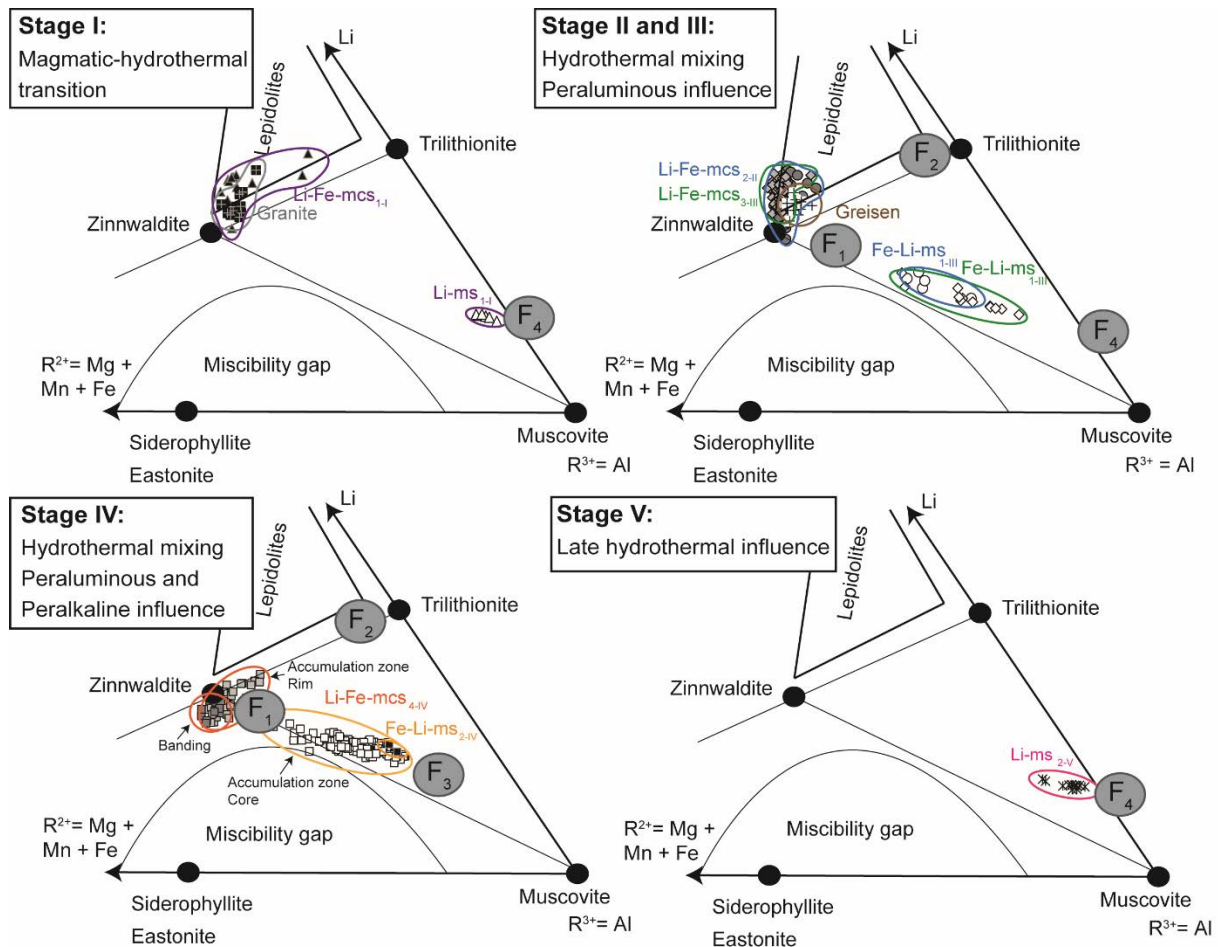


Figure 33. Synthesis of the evolution of Li-micas in the Monier and Robert (1986) phase diagram as a function of R_{3+} (Al), R_{2+} (Fe+Mg+Mn) and Li (apfu). The observed trends are interpreted to record mixing trends between four fluid end-member compositions (F1 to F4) reflecting the involvement of both peraluminous and peralkaline magmatic sources and external fluids. Explanations in the text.

5.2. Hydrothermal Li-mica trends: insights into fluid typology and mixing

Although associated with an increase in rare metal contents (Figure 32), the trends in the zinnwaldite to trilithionite evolutions of vein-hosted trioctahedral micas over time (i.e., stages II, III, IV) cannot be interpreted as fractionation trends during precipitation from a single

hydrothermal fluid. Two longitudinal profiles, made along the cleavage from the base to the top of the plumose crystals, were analyzed in apparently non-zoned mica crystals (SEM and microscopy observations) from stages II (Fe-Li-Mcs_{2-II}) and III (Fe-Li-Mcs_{3-III}) (Figure 31). Although clearly showing the large-scale ($\geq 500 \mu\text{m}$) variations that correspond to the zoning, the profiles do not display the systematic trends (e.g., increasing Fe with decreasing Mg) that would be expected in the case of continuous fractionation from a single fluid either within the source or resulting from mica crystallization in a closed system. As seen in Figure 31, there are significant variations (alternating increases and decreases) in element contents from core to rim (i.e. over time) in the Fe-Li-Mcs_{2-II} crystal, but no significant trend can be distinguished at the crystal scale.

The observed Fe-rich to Fe-poor oscillatory bulk evolution of the dioctahedral core of the stage IV Fe-Li-mcs_{4-IV} is similarly incompatible with a fractionation process, especially as no effects (such as an increasing Fe/Mg ratio) are observed at the scale of the growth zone crystallization in a closed system (Figure 31).

The observed trends must therefore represent mica formation in compositionally evolving fluids (Figure 30) that result from mixing between discrete hydrothermal end-members. In order to fully explain the complex trends displayed in Figure 31, a minimum of four end-members are required: an “F1”-fluid, corresponding to the zinnwaldite of composition (1); an “F2-fluid”, recorded by the trilithionite micas of composition (2); an “F3-fluid”, expressed in the crystallization of the Fe-rich dioctahedral micas cores in stage IV banded quartz, with composition (3); and an “F4-fluid”, corresponding to the more trilithionitic dioctahedral compositions, with composition (4) (Figure 30 and Figure 33). The observed trends for primary micas correspond to either “F1”-“F2” or “F1”-“F3” mixing trends.

Considering the details of the paragenetic mica succession (Figure 23), it would thus appear that bulk fluid evolution at Maoping was characterized by successive interplay of the “F1” end-member, first with “F2” and then with “F3”, and the repetitive involvement of the “F4” end-member.

5.3. Interpretation of magma and fluid evolution

The changes in the mineral and metal assemblages through time, as defined by the paragenetic sequence (Figure 23), may correlate with the fluid evolution described above.

The greisen and stages II and III correspond to a W-Sn-Li-(Mo) association (Figure 32B), which, being typical of peraluminous granite-related mineralization (Cerny *et al.*, 2005), would suggest the involvement of a peraluminous magma at this time. The “F₁-F₂” trend in Figure 33 implies the existence of two fluids during these stages, both of which may be related to the peraluminous granite. The “F₁” end-member, as seen in Figure 33 and Figure 32C, is in fact characterized by an elevated rare-metal content, as shown by the zinnwalditic greisen and stage II and stage III micas, whereas the “F₂” component, being poorer in rare metals (Figure 32C), likely represents a fluid better interpreted as an external fluid. Mixing of a magmatic and a meteoric fluid could explain cassiterite and wolframite deposition, as described for instance around the Mole granite in Australia (Audétat *et al.*, 1998). In the Maoping case, “F₁” could be either a true magmatic-hydrothermal fluid or a fluid equilibrated under high temperature subsolidus conditions with a peraluminous rare metal granite. “F₂” on the other hand, could be either a metamorphic fluid (derived for instance, from metamorphic contact aureoles) or a meteoric fluid that has undergone variable degree of exchange with the crust. Unfortunately, it is impossible here to distinguish diluted magmatic or metamorphic fluids from meteoric fluids that may have percolated within the crust.

Stage IV zinnwalditic trioctahedral micas, with their elevated W-Nb-Sn-Ta contents, may also be considered as having been deposited by a more-evolved “F₁” end-member. However, the micas in the stage IV banded quartz are characterized by a rare metal content that is higher than that of the peraluminous granite micas (Figure 32). A micro-paragenesis displaying Nb-F-REE associations and micas with Nb enrichment are associated with this stage (Figure 32B) and would be more akin to a new peralkaline magmatic source involved in the fluid composition of an “F₃” fluid (Cerny *et al.*, 2005). Given the elevated Ta content of the primary dioctahedral micas from stage IV, a mixed influence from both peraluminous and

peralkaline magmas seems most probable. In Figure 15, the “F₂” and “F₃” fluids are both seen to mix with an “F₁” component. In the banded quartz, this component is represented by the small protolithionitic platelets that characterize the banding. This in turn suggests that the stage IV quartz primarily grew from this “F₁” fluid, with occasional influxes of fluids influenced by both peraluminous and peralkaline magmas. Indeed, the absence of significant W-Sn mineralization in stage IV might be explained by the prevalence of the “F₃” fluid contribution during this part of the evolution of the Maoping system. As demonstrated by the stage VI assemblage (F-P-REE), the “F₃” fluid may have also dominated the end of the depositional history. However, unfortunately no mica proxies exist to support this, unless the Li-ms_{2-V} can be considered as a precursor, as is suggested by their positions in Figure 33 (it was not possible to perform any trace element analyses on these because of their radial and zoned morphology).

Finally, *the “F₄” end-member*, which is associated with the alteration of primary Fe-Li-micas into Li-muscovite, may be interpreted by comparing it with similar types of alteration found in the rare metal granite environment. For instance, at Beauvoir in the French Massif Central, Li-muscovite overgrowth onto magmatic lepidolite was suggested to have been related to high temperature sub-solidus ingress of meteoric fluids into the cooling granite cupola (Cuney *et al.*, 1992). In the Maoping veins, the late Li-muscovites may similarly be interpreted as recording the involvement of a meteoric fluid, the “F₄” end-member.

5.4. Temporal rather than spatial zoning

As mentioned in Section 2.2, a zonal distribution has previously been described at Maoping by Feng *et al.* (2011), with a vein mineralogy that changes gradually from the greisen to the surface, from quartz-W-Mo-(Sn) veins at depth to W-Sn sulphide veins and eventually to an upper fluorite-muscovite veinlet zone. In light of the detailed paragenetic sequence exposed previously, this zoning may essentially be temporal in nature, recording the successive emplacement of granitic magmas, only one of which is apparently observed at the present level of exposure. This granite, which in the previous sections was shown to be associated with the earliest feldspar veins (stage I) and which is overprinted by mineralized

quartz veins and the related greisen, cannot therefore be the peraluminous granite that influenced the “F₂” end-member. As shown in Figure 33 and Figure 32C, at least two more successive magma pulses are required to provide the peraluminous-influenced fluids involved in the greisen to stage III and stage IV micas, respectively. In addition, the presence of a concealed peralkaline (A1-type) intrusion at depth is also required for the “F₃” end-member. Whereas the peraluminous granites were likely associated with the early Yanshanian magmatic cycle, this peralkaline granite would be representative of the Late Yanshanian cycle (Hu and Zhou 2012).

6. Conclusion

By correlating the geochemistry of Li-micas with the detailed paragenesis of the Maoping deposit, we are able to obtain a number of constraints on the magmatic and hydrothermal processes at the origin of the W-Sn deposits in the Nanling region, the richest W-Sn province worldwide. Textures and compositions of Li-micas along successive paragenetic stages allow to identify : (i) magmatic Fe-Li-micas from the granite and the feldspar veins, which are thought to demonstrate an early fluid-melt separation; (ii) hydrothermal Fe-Li-micas coeval with the greisenisation, which precipitated from the mixing of fluids that were influenced to varying degrees by the underlying peraluminous magma source, (iii) hydrothermal zoned Li-micas with compositions fluctuating between zinnwaldite and Fe-Li-muscovite and influenced by a new peralkaline magma; and (iv) secondary Fe-Li-muscovites and late Li-muscovites that reflect a late meteoritic influence at each stage. Coupled detailed paragenesis and Li-mica composition are therefore powerful indicators of the magmatic-hydrothermal evolution of the Maoping deposit, highlighting the various fluid types and magma inputs involved in a single rare metal deposit.

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Chapter 3: The ore-forming magmatic-hydrothermal system of the Piaotang W-Sn deposit (Jiangxi, China) as seen from Li-mica geochemistry

Article published in American Mineralogist 103 (2018) 39-54.

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Abstract

Many studies have proved the usefulness of Li-mica and chlorite geochemistry as indicators of the chemical and thermal evolution of magmatic systems. This study highlights the suitability of Li-micas as tracers of hydrothermal mineralizing events in world-class W-Sn deposits associated with Jurassic (190–150 Ma) granites in China through the complex magmatic–hydrothermal evolution of the Piaotang deposit (South Jiangxi). A paragenetic sequence has been established for the Piaotang deposit comprising (i) a first “silicate-oxide” stage that hosts abundant W-Sn mineralization (wolframite and cassiterite), (ii) a “calcic” stage with scheelite and wolframite, (iii) a “base metal sulphides” stage with cassiterite and wolframite, and (iv) a late “sulphide” stage, involving for the first time a polyphase emplacement of the mineralization. Li-micas from the underlying granite, greisen, and the different stages represented in the veins, were studied. The chemistry of the micas (characterized by

intermediate compositions between phlogopite-zinnwaldite-muscovite poles) demonstrates the presence of end-members representing three different fluids that were involved in the emplacement of the Piaotang deposit. These end-members can be linked to previous fluid inclusion studies conducted on this deposit. The three fluids are identified to be magmatic, meteoric (as previously reported in the literature), and also metamorphic, and are shown to have mixed throughout the different stages. Moreover, it appears that the magmatic fluids could not have been derived from the Piaotang biotite granite but rather must have originated from a more evolved rare metal granite that is presently unidentified. These fluids were responsible for the greisenization.

Finally, chlorite geochemistry reveals the occurrence of a heating process (from 200°C in stage II to 300°C in stage III) during the post-mineralizing stages, which was responsible for the precipitation of new generations of ore-bearing minerals (cassiterite and wolframite) concomitant with a continuous gain of metals during the emplacement of the Piaotang deposit.

Keywords

Piaotang - W-Sn deposit – Yanshanian - Lithium-mica – Chlorite - Magmatic-hydrothermal

1. Introduction

Constraining the origin and evolution of mineralizing fluids in W-Sn-quartz vein-type deposits remains challenging. Current models refer to (i) metal-rich magmatic fluids exsolved from granitic magmas (e.g., Kamenetsky et al., 2004; Audétat et al., 1998; Thomas et al., 2005), (ii) “external” fluids (metamorphic or meteoric) that circulate around cooling peraluminous granitoids and leach metals from the peraluminous granitoid or the country rocks (e.g., Wilkinson 1990; Blamart 1991; Smith et al. 1996; Zhao and Jiang, 2004), or (iii) combinations of the two (e.g., Beuchat et al., 2004; Carruzzo et al., 2004; Marignac and Cathelineau 2009; Wei et al., 2012; Chicharro et al., 2016). In association with stable isotope

(O, H) studies of minerals (Beuchat et al., 2004; Carruzzo et al., 2004; Wei et al., 2012; Chicharro et al., 2016), fluid inclusion studies have contributed greatly to these models in recent years, by providing valuable information about the origins and physical–chemical evolution of the mineralizing fluids (e.g., Audétat et al. 1998; Beuchat et al., 2004; Carruzzo et al., 2004; Wei et al., 2012; Chicharro et al., 2016). However, fluid inclusion studies are restricted to just a few favourable mineral species (e.g., topaz, quartz, wolframite, and cassiterite) and even then, primary fluid inclusions are often hard to identify. Consequently, several stages of the deposit formation cannot be studied by this approach, and can only be investigated indirectly using detailed paragenetic successions and in-depth geochemical analysis. In this perspective, Li-mica and chlorites, which are frequently found in W–Sn vein-type deposits (Giuliani, 1985; Tischendorf et al., 1997; Costi et al., 2002; Johan et al., 2012; Nieva, 2013) show interesting potential. The majority of chlorite species are sensitive to temperature in hydrothermal and geothermal systems (Walshe, 1986), and thus provide a useful geothermometer in many geological settings and, in particular, in hydrothermal metallogenic systems. Moreover, Legros et al. (2016) showed that, in the case of the Maoping deposit (Jiangxi, China), the detailed chemistry of magmatic and hydrothermal Li-mica can be used to interpret the relative contributions of magmatic, meteoric and metamorphic fluids in the formation and evolution of W–Sn deposits. Based on this work, the present study focuses on the Piaotang W–Sn deposit, located in the southern Jiangxi Metallogenic Province (SE China), the most important W–Sn province in the world (USGS, 2016). The Piaotang W–Sn deposit exhibits several generations of magmatic and hydrothermal Li-mica, as well as two generations of chlorite, that crystallized during multiple stages of the ore-forming system. This study uses combined optical microscopy, SEM, EPMA and LA-ICPMS to establish for the first time a detailed paragenetic succession for the Piaotang deposit and to determine the major and trace element compositions of Li-mica and chlorite geothermometers, with the aim of providing new constraints on the physical–chemical evolution of fluids involved in the formation of such deposits.

2. Geological Setting and sampling

2.1 The southern Jiangxi Metallogenic Province

The southern Jiangxi W-Sn province, also referred as Nanling metallogenic belt, is located in the Cathaysia Block of the South China Craton (SCC) and is currently the main W repository in China (90% of the reserves: Wang et al. 2016). The SCC resulted from late Grenvillian suturing between two terranes: the Yangtze Block to the north and the Cathaysia Block to the south, during the Jiangnan orogeny (Charvet 2013 and references therein). The final age of the collision is still under debate, with estimates ranging between 900 and 800 Ma (e.g., Li et al. 2009, Hu and Zhou 2012, Yao et al. 2013). Mostly coinciding with the southern boundary of the orogen, the major Jiangshan-Shaoxi fault zone is considered the northern boundary of the Cathaysia block (Figure 34).

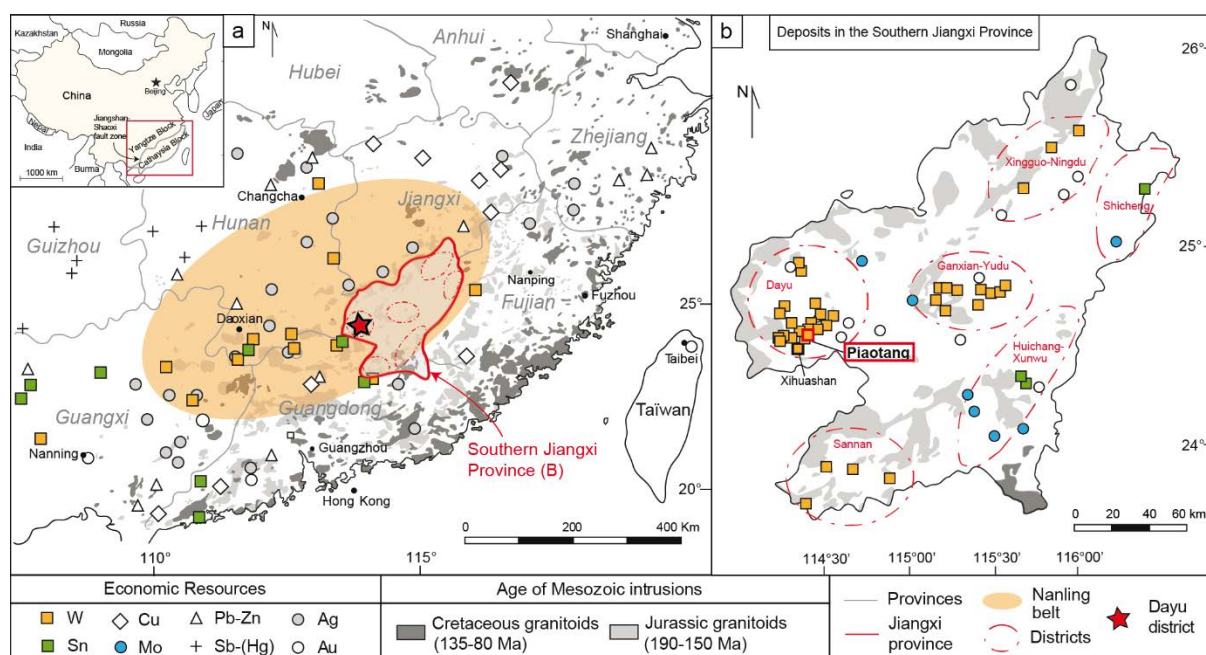


Figure 34. (a) Ore mining districts and metal deposits of the Cathaysia Block, (b) Detailed map of the southern part of the Jiangxi Province, SE China, showing the distribution of Jurassic and Cretaceous granitoids. The Piaotang deposit is located in the Dayu district in the south-western part of the Jiangxi province (Modified after Legros et al. (2016) and references therein).

After the Neoproterozoic collage of the Yangtze and Cathaysia terranes, the newly formed SCC was strongly reworked, becoming progressively metacratonized through a

sequence of intra-continental events starting in the late Neoproterozoic (the failed Nanhua Rift: Wang et al. 2006), and followed by the Middle Palaeozoic (“Caledonian”) Kwanghsian orogenesis, the Early Mesozoic Indosinian events and the Jurassic-Cretaceous Yanshanian events. Although the entire SCC was affected, the metacratonisation and the most pervasive magmatic activity during the Yanshanian were observed in the Cathaysia Block. Consequently, the Phanerozoic magmatism was responsible for producing one of the largest igneous provinces in the world (Zhang et al. 2012, Wang et al. 2013). A variety of granites were produced, including S-, I- and peralkaline-types. Metaluminous A-type granites remain the most frequently found. Nb-Ta-rich peraluminous rare metal granites (RMG) also form part of this magmatic diversity. Sporadic occurrences of RMG were first recorded in the Kwanghsian (Wang et al., 2011), whereas both the Indosinian (270–200 Ma) (with the world-class Yichun deposit) and the early Cretaceous (ca. 130 Ma) appear to have been the most productive episodes of RMG magmatism.

The Middle Palaeozoic Kwanghsian (Caledonian) orogeny, which produced a double-verging NE-trending belt, is commonly interpreted to have resulted from the inversion of the Nanhua Rift (e.g., Charvet et al., 2010, Charvet 2013). This first intra-cratonic event, which was restricted to the Cathaysia Block, culminated with crustal thickening and a HT metamorphic event at ca. 450 Ma, yielding a high-grade belt composed of migmatites and granulites (e.g., Liu et al. 2010), followed by basaltic underplating, dehydration melting in the lower crust, and the emplacement of large syn-kinematic S-type granite bodies (covering more than 20,000 km²: Li et al. 2011) at ca. 430–420 Ma (e.g., Wang et al. 2013, Xia et al. 2014).

At the end of the Triassic, a second major intracratonic event, which reworked the earlier Kwanghsian structures (with the same NE-trending direction but a NW vergence), recorded the suturing of the SCC with the North China craton (e.g., Wang et al. 2001). This Indosinian event, which affected both the Cathaysia Block and the Jiangnan orogen, was associated with HT metamorphism (metamorphic core complexes: Faure et al. 1996), and produced large volumes of magma (presently covering 14,300 km²), with the subsequent

emplacement of I-type granites (245–230 Ma) and S-type granites (220–210 Ma) (Shu et al. 2008 and references therein). The first cycle of Nb–Ta granites occurred in the Nanling Range (Mao et al. 2013).

The Yanshanian event (Late Jurassic–Cretaceous) was essentially marked by magmatism that led to the generation of the large igneous province (Li et al., 2014). However, the event was also associated with alternating transpressive and transtensional deformation along the same NE–SW lineaments that controlled the Kwangsian and Indosinian events (Li et al., 2014, Liu et al., 2012, Shu et al., 2009). The Yanshanian events are currently interpreted as reflecting a major change in the overall plate tectonic regime, with a shift from a Palaeo-Tethysian to a Pacific control, marked by the inception of subduction of the Paleo-Pacific (Izanagi) plate under the SCC (e.g. Zhou et al., 2002). The Yanshanian is subdivided into the Early (Jurassic) and Late (Cretaceous) Yanshanian. The Jurassic (190–150 Ma) is characterized by extensive magmatic activity, today represented by the outcropping of granitic rocks over an area of about 100,000 km². The peak of magmatic activity is dated at 160–150 Ma, with several pulses, and the granitic province exhibits all the characteristics of a felsic large igneous province. The majority of the Cathaysia W–Sn deposits were formed during the same 160–150 Ma interval, although this was recently challenged (ca. 130 Ma ages proposed by Wang et al. 2016). Coeval with this, the E–W trending Nanling Range, which is composed of three roughly parallel granitic belts and associated wolframite deposits (Zhao et al. 2017), was formed. The Cretaceous event (135–80 Ma) was characterized by granites (mainly intruded in the Cathaysian interior) and rhyolites (mainly erupted along the coastal area). Renewed rare-metal deposition occurred in the Nanling Range, characterized notably by large magmatic-related Sn and U mineralizations, while Au–Cu–Pb–Zn–Ag mineralizations were deposited along the southeastern coast (Hua et al., 2005).

2.2 The Piaotang W-Sn deposit

The polymetallic W-Sn-(Nb) Piaotang deposit is one of several giant tungsten deposits in the Nanling Range and produces 1,500 t WO₃ per year, ranking it eighth in the top ten reserves in China with 47,775 t WO₃ (MB Company database: www.metalbulletin.com). The deposit is composed of mineralised veins emplaced in Cambrian metasediments that lie above the concealed Piaotang biotite granite (Figure 35). Two sets of faults can be observed in the field: E-W-oriented faults intersecting NE-SW-oriented faults (Figure 35A). The granite was emplaced during the Jurassic (159.8 ± 0.3 Ma, U-Pb zircon dating; Zhang et al., 2017), coeval with the W mineralization (159.5 ± 1.5 Ma, U-Pb cassiterite dating; Zhang et al., 2017). A Kwangsi quartz diorite body (439 ± 2 Ma, U-Pb zircon dating; He et al. 2010) also occurs at depth.

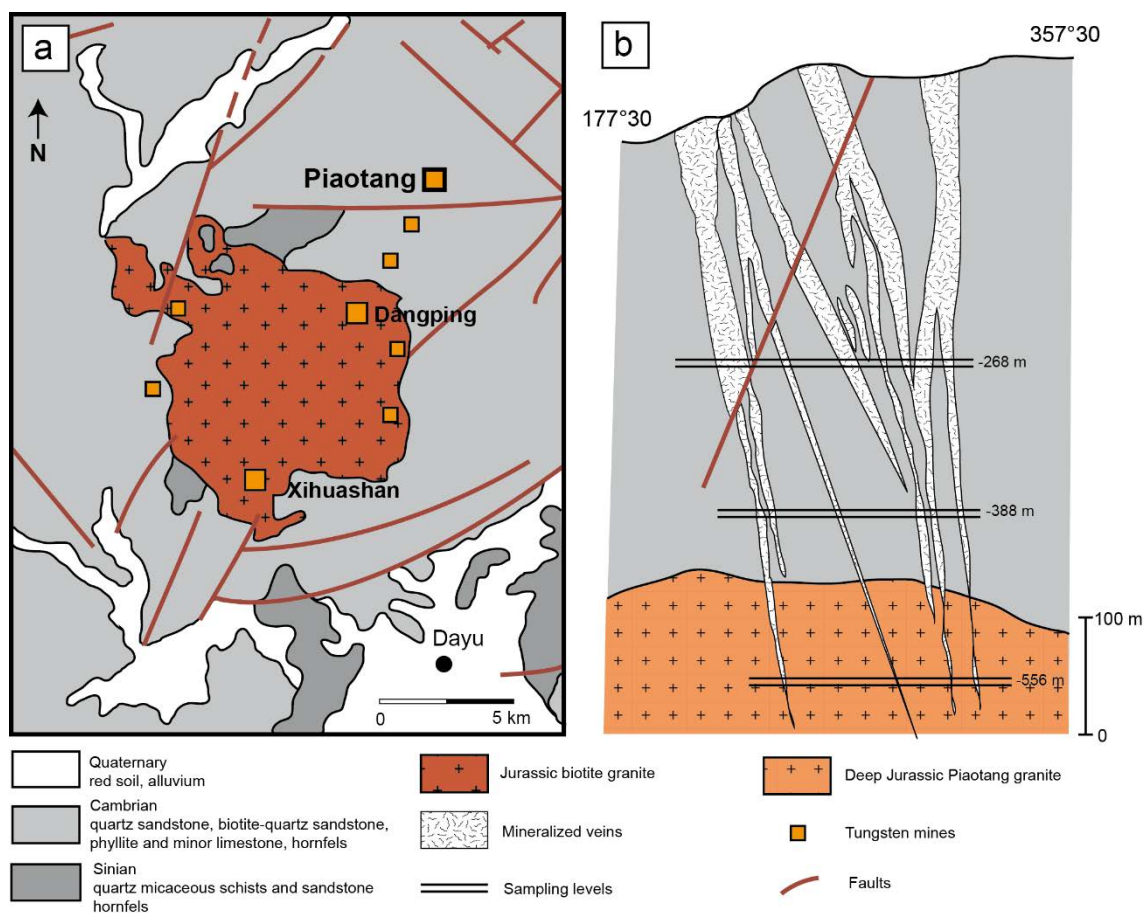


Figure 35. Simplified geological map (a) and cross-section with associated strikes (b) of the Piaotang W-Sn deposit, showing the relationships between the deep granite, Cambrian host rocks, and mineralized veins and the sampling levels ((a) modified after Tanelli 1982 and (b) modified after Ni et al., 2015).

The vein system: The vein system extends vertically over 300 m, down to the Piaotang biotite granite (Figure 35B). The vein system includes flat-lying and steep veins, associated with evidence for brittle tectonics. Field observations at different levels show (i) a first generation of, more or less lenticular, flat-lying (N80-90°E 30-40°N) quartz veins, displaying some evidence of faint extensional tectonics, (ii) a second generation of similar flat-lying veins (N0-30°E 30-40°W) corresponding to inverse faulting (as demonstrated by their intersections with the first generation veins), and (iii) a third generation of steep veins, with clear evidence for both faint inverse and sinistral strike-slip tectonics. Whereas the first generation of veins is scarce and the second generation is moderately frequent, the third generation of veins is ubiquitous. Only the third generation of veins shows mineralizations of economic interest. The thickness of the veins increases from the bottom to the top, from a few centimetres at the 556 level, to more than one meter at the 268 level.

Relationship with the granite: At the bottom of the system, a fine-grained biotite granite is observed. The interaction between this granite and the vein system appears to be complex.

- The granite exhibits a locally steep and sharp contact with the country rocks, striking 30°N. Outside the main body, several gently dipping dike-like expansions are observed, which themselves present complex digitations in the form of crackle breccias. No ductile deformation is observed along the contact between the granite and country rocks.

- Inside the granite body, a gently dipping vein system is observed. The veins show borders composed of feather-like K-feldspars megacrysts, and a filling of greisenized fine-grained rock that forms a cement around large quartz crystals (stockscheider).

- The granite overprints a generation of flat-lying quartz veins which likely represent the first generation of veins. Some dike-like expansions appear to be associated with inverse brittle faulting as they intersect earlier flat-lying quartz veins. These dikes are likely coeval with the second generation of veins.

Granite and greisen: The primary granite mineralogy consists of quartz, orthoclase, albite and biotite (see below, Figure 38a). The texture is microgranular with porphyric perthitic K-feldspar and quartz. No ductile or brittle deformation is observed: the granite is always macroscopically isotropic. The micas enclose a variety of accessory minerals—apatite, quartz, xenotime, rutile, monazite, fluorite and zircon—that are not observed in quartz or feldspars. W, Sn or Nb-Ta minerals have not been observed in the granite. The feldspars are altered to muscovite but also exhibit clay mineral alteration. This may be considered as incipient greisenization, even if a clear granite to greisen transition is not observed in the mine.

The greisen at the top of the granite body is composed exclusively of quartz, muscovite and fluorite (see below, Figure 38b). All minerals are corroded and very weakly deformed. Fluorite is observed exclusively as inclusions in muscovite.

3. Analytical methods

Thirty samples of the mineralized veins, granite and greisen (Figure 36) were collected in the Piaotang mine site at the 268, 388 and 556 levels (respective depths). The relationships between host-rocks and veins are easily observed in the mine. As discussed previously, the third generation of veins is the mineralization-bearing vein and thus only this vein is considered in the remainder of the manuscript.

All preparations and analyses described below were carried out at the GeoRessources laboratory (Nancy, France). Petrographic observations were performed on polished thin-sections using conventional transmitted and reflected light microscopy and a HITACHI FEG S4800 scanning electron microscope (SEM) equipped with an energy dispersive spectrometer (EDS), using a Si(Li) semi-conductor detector.

Electron microprobe analyses (EMPA) of Si, Al, Ti, Na, Mg, Mn, Fe, K, Ca, Rb and F were performed on chlorites and Li-bearing micas using a CAMECA SX100 instrument equipped with a wavelength dispersive spectrometer (WDS) and calibrated using natural and

synthetic oxides and silicates (albite, olivine, Al_2O_3 , orthose, andradite, MnTiO_3 , hematite, cassiterite, RbTiPO_5 , topaz, scheelite, LiTaO_3 and LiNbO_3). A current of 12nA and an accelerating voltage of 15kV was used with a counting time of 10s per element. Special attention was paid to fluorine determination by using a spectrometer dedicated to this element (average detection limit of 1300 ppm). The analyses have a spatial resolution of 1 to 2 μm . The reproducibility of standard analyses was 1% for each element analyzed. Total Fe is presented as FeO.

Major, minor and trace elements (^{24}Mg , ^{27}Al , ^{28}Si , ^{47}Ti , ^{55}Mn , ^{59}Co , ^{93}Nb , ^{95}Mo , ^{118}Sn , ^{137}Ba , ^{181}Ta , ^{182}W) were measured in Li-bearing micas using a LA-ICPMS instrumental setup described in Leisen et al. (2012) and Lach et al. (2013), composed of a GeoLas excimer laser (ArF, 193 nm, Microlas, Göttingen, Germany) (Günther et al., 1997) and an Agilent 7500c quadrupole ICPMS. The laser beam was focused onto the sample within a 24.5 cm^3 cylindrical ablation cell with a Schwarzschild reflective objective (magnification $\times 25$; numerical aperture 0.4) mounted on an optical microscope (Olympus BX41), equipped with a X–Y motorized stage and a CCD camera. Si (determined by EPMA) was chosen as an internal standard and precisely quantified by EPMA for all analysed micas. NIST SRM 610 (values from Jochum et al., 2011) was chosen as the external standard (accuracy was verified by analysing NIST SRM 612). The parameters used were a fluence of $8\text{J}/\text{cm}^2$ and a laser shot frequency of 5 Hz, $\text{He} = 0.5\text{ L}\cdot\text{min}^{-1}$ as a carrier gas mixed with $\text{Ar} = 0.7\text{ L}\cdot\text{min}^{-1}$ via a cyclone mixer prior to entering the ICP torch, spot sizes of 120 μm , and an ablation duration of 40 s. Quantifications were performed using Iolite software (Paton et al., 2011) and concentrations were calculated following Longerich et al. (1996).

The temperature of chlorite formation was calculated using the graphical geothermometer of Bourdelle and Cathelineau (2015) for each composition that plotted between the sudoite, corundophilite and Al-free chlorite end-members on the Si-R $^{2+}$ diagram of Wiewiora and Weiss (1990). This method assumes that quartz and water activities are equal to 1 and is valid for a temperature range of 50–350°C, which seems reasonable in the case of

quartz-bearing rocks formed under low-grade metamorphic conditions, such as those in the Piaotang deposit. All Fe was assumed to be ferrous in the calculations of the structural formulas.

4. Paragenetic sequence

A paragenetic succession was previously established by Tanelli (1982), providing a common chart for the Piaotang and Xihuashan deposits. According to Tanelli (1982), the succession can be divided into three stages: (i) a first “oxide stage” with quartz, K-feldspar, beryl, topaz, wolframite and cassiterite; (ii) a second “sulphide stage” with quartz, bismuthinite, sphalerite, galena, cassiterite and wolframite; and (iii) a third “carbonate stage” with quartz, chlorite, sericite, fluorite, calcite, pyrite and scheelite.

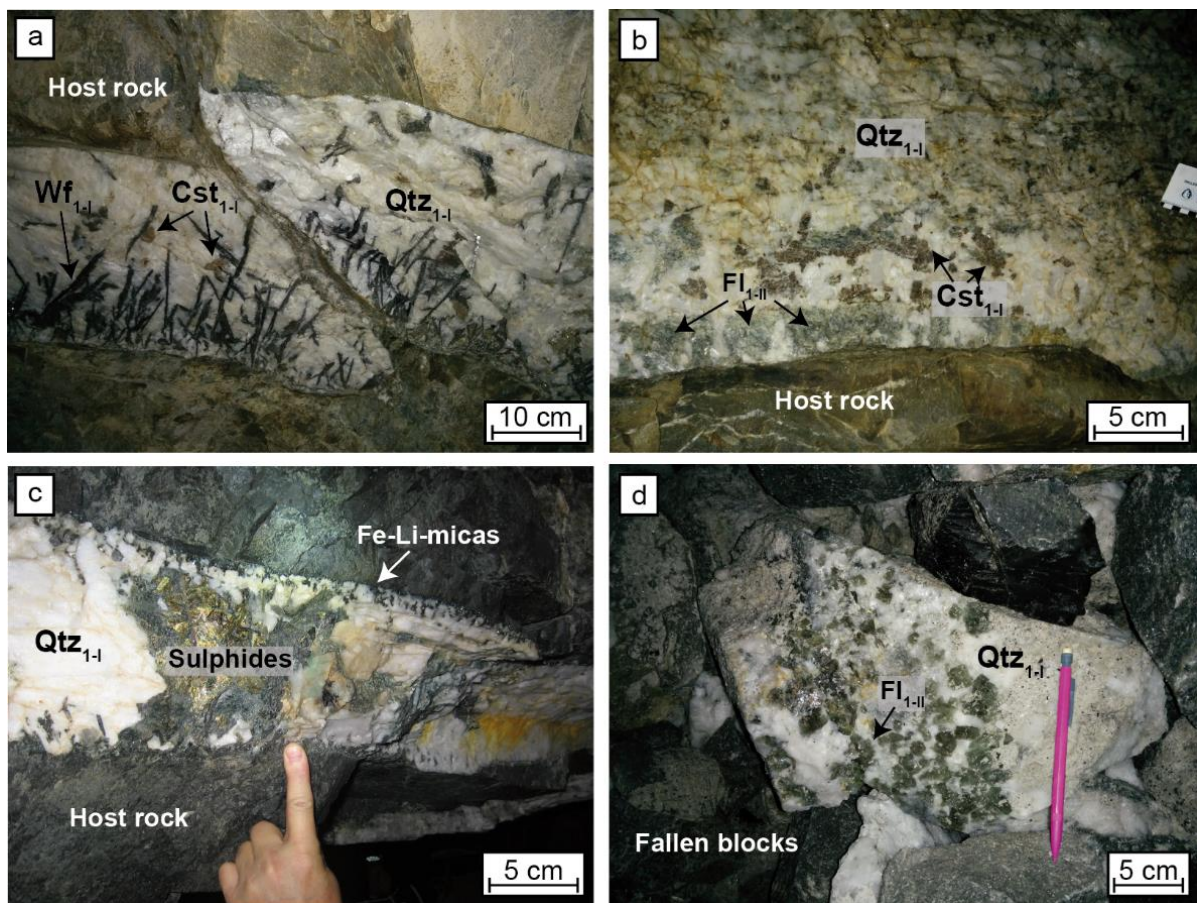


Figure 36. Mineralized veins as observed in the mine galleries at different levels of sampling. (a) Wolframite-cassiterite-quartz vein. (b) Fluorite-cassiterite-quartz vein. (c) Accumulated sulphides in the vein. (d) Fluorite accumulation in the vein.

In the present study, a new paragenetic sequence was established from field observations in the mine galleries, as well as from petrographic observations by optical microscope, SEM and EPMA analyses in the laboratory (Figure 37). Hereafter, minerals refer to (i) their recommended IMA mineral abbreviations, (ii) their generation number (1 to 4, from the oldest to the youngest), and (iii) the paragenetic stage to which they belong (I to III, from the oldest to the youngest). For example, Wf_{2-III} stands for the second generation of wolframite in the whole paragenetic sequence, occurring during stage III (sulphide stage).

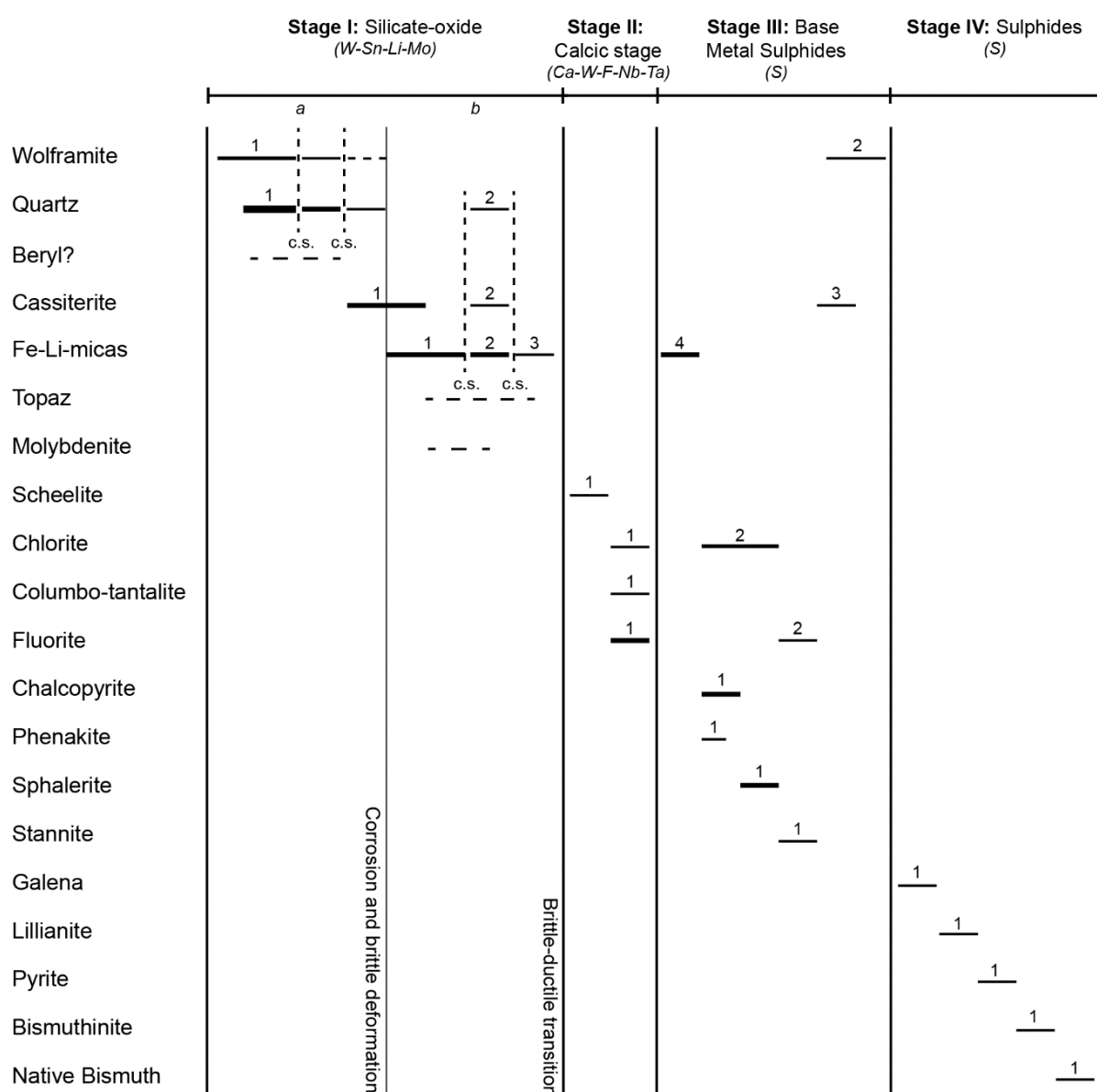


Figure 37. Paragenetic sequence of veins in the Piaotang W–Sn deposit based on petrographic observations of samples from mine galleries at levels 268, 388 and 556. See text for details of the different stages, mineral abbreviations and notations.

As seen in Figure 37, our detailed study has led to some modification of the Piaotang paragenetic succession. The main difference is seen in the “calcic stage”, now intercalated between the “oxide” and “sulphide” stage on the basis of crosscutting relationships between major minerals of the different stages. The succession obtained in this study is valid for the entire vertical extent of the mineralized system (all levels).

The silicate-oxide stage (Stage I) represents the main stage of crystallization of the ore-bearing minerals, wolframite and cassiterite. The Stage I minerals are Fe-Li-mica (Fe-Li-mca_{1,2,3-1}), cassiterite (Cst_{1,2-1}), wolframite (Wf₁₋₁), topaz (Toz₁₋₁), quartz (Qtz_{1,2-1}), molybdenite (Mlb₁₋₁), and possibly beryl (Br₁₋₁). Stage I can be subdivided into two sub-stages: the first (Ia) is the main ore stage and the second (Ib) is the main Fe-Li mica stage. There is however, considerable overlap: (i) the earliest Fe-Li-mca₁₋₁ are coeval with Cst₁₋₁, and (ii) Cst₂₋₁ is observed to have crystallized coevally with later Fe-Li-mca₂₋₁.

The wolframite Wf₁₋₁ shows maximum growth perpendicular to the walls, with crystals up to 15 cm in size found in the largest veins (Figure 36). Wf₁₋₁ is coeval with quartz growth (Qtz₁₋₁), which shares similar comb textures and size. However, the initial morphologies of both the quartz and wolframite were considerably altered by a succession of crack-seals (up to brecciation) and recrystallization phenomena, with the growth of small newly formed wolframite prisms on previous crystals (Figure 38c). Nevertheless, the wolframite composition is homogeneous, with an average structural formula of Fe_{0.5}Mn_{0.5}WO₄ (SEM). The main cassiterite (Cst₁₋₁) crystallization occurred after the main wolframite deposition, and cassiterite growth is often observed on wolframite crystals (Figure 38c). Cst₁₋₁ deposition was coeval with crack-sealing and Cst₁₋₁ is therefore frequently found at the vein selvages. The cassiterite composition is pure SnO₂ (SEM). The earliest Fe-Li-mca₁₋₁ are associated with Cst₁₋₁, and have often grown in dissolution cavities affecting Qtz₁₋₁ (Figure 38d). A second generation of Fe-Li mica (Fe-Li-mca₂₋₁) occurs as infilling of vein openings, most often at the vein selvage, where it separates early cassiterite from the wall-rocks. Small overgrowths of Cst₂₋₁ are observed at the mica-Cst₁₋₁ boundary. Crack-seals filled with Fe-Li-mca₂₋₁ are also observed within Fe-Li-

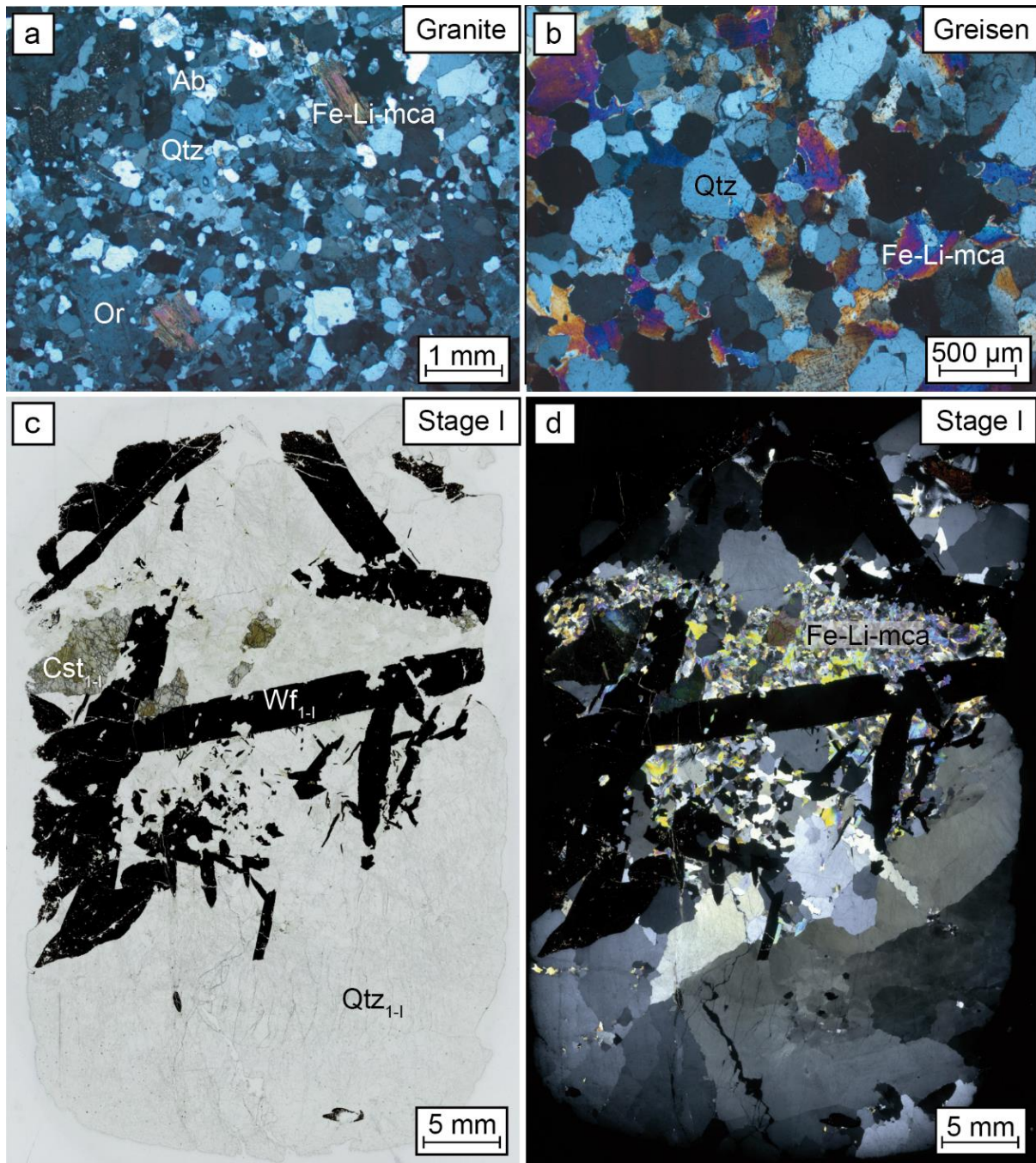


Figure 38. Microphotographs of the mineralogical textures and associations of the Piaotang deposit. (a) Micrograined-granite with porphyritic feldspar and biotite (crossed polars). (b) Slightly deformed quartz grains and corroded muscovite from the greisen (crossed polars). Thin-section in (c) natural light and (d) crossed polars of the main minerals that crystallized in stage I (Cst_{1-1} , Wf_{1-1} , Qtz_{1-1} and Fe-Li-mca), showing the multiple crack-seal episodes observed in quartz and wolframite. Abbreviations according to the IMA: Ab = albite; Cst = cassiterite; Mca = micas; Or = orthoclase; Qtz = quartz; Wf = wolframite.

mca_{1-1} aggregates and Cst_{1-1} , with overgrowths of Cst_{2-1} associated with Qtz_{2-1} . Cst_{2-1} is compositionally identical to Cst_{1-1} . A last episode of crack-seal is observed in Fe-Li- mca_{2-1} at

the vein boundary and is associated with the deposition of the third generation of Fe-Li micas (Fe-Li-mca_{3-I}).

Topaz (Toz_{1-I}) has been deposited in fractures affecting Qtz_{1-I}, with symplectic topaz-quartz local associations indicating partial dissolution of the quartz. The exact timing of the topaz deposition is ambiguous. Local observations indicate that the topaz post-dates the Cst_{1-I} deposition and has been altered by stage III micas, but no relationship between the topaz and the main micas was observed. Topaz was most probably emplaced during stage I as it experienced the same plastic deformation as the surrounding Qtz_{1-I} as well as molybdenite (see below).

Tanelli (1982) listed K-feldspar, beryl, tourmaline, helvite and biotite in the oxide stage. These minerals were not observed in the present study, with the possible exception of beryl. Some euhedral prismatic crystals with hexagonal sections are observed in association with Qtz_{1-I}, which, being totally pseudomorphosed by stage III Fe-Li-mca_{4-III}, could originally have been beryl (Brl_{1-I}).

An episode of ductile deformation separates stage I from stage II. This episode affected both Qtz_{1,2-I}, all the Fe-Li micas, the topaz and the molybdenite. This deformation consists of very low intensity kinking of the micas and molybdenite, but is pervasive in quartz (subgrain boundaries, undulose extinction). Such ductile-brittle transition separation (only observed microscopically) between early minerals and later stage minerals was also observed in the nearby Maoping deposit (Legros et al. 2016), where it may have been related to extensional tectonics.

Calcic stage II: Stage II is characterized by the partial transformation of wolframite into scheelite (Sch_{1-II}), followed by a first generation of chlorite (Chl_{1-II}) and fluorite (Fl_{1-II}) (Figure 39a and b). Small euhedral prismatic crystals of W-rich (up to 8.5 wt% WO₃) manganocolumbite (Clb_{1-II}), up to 5 µm in size, are systematically dispersed in the Chl_{1-II}. It is likely that the Clb_{1-II} is the result of mobilization of Nb and W during wolframite transformation into scheelite. However, no particular textures were observed in Wf_{1-I} using the SEM.

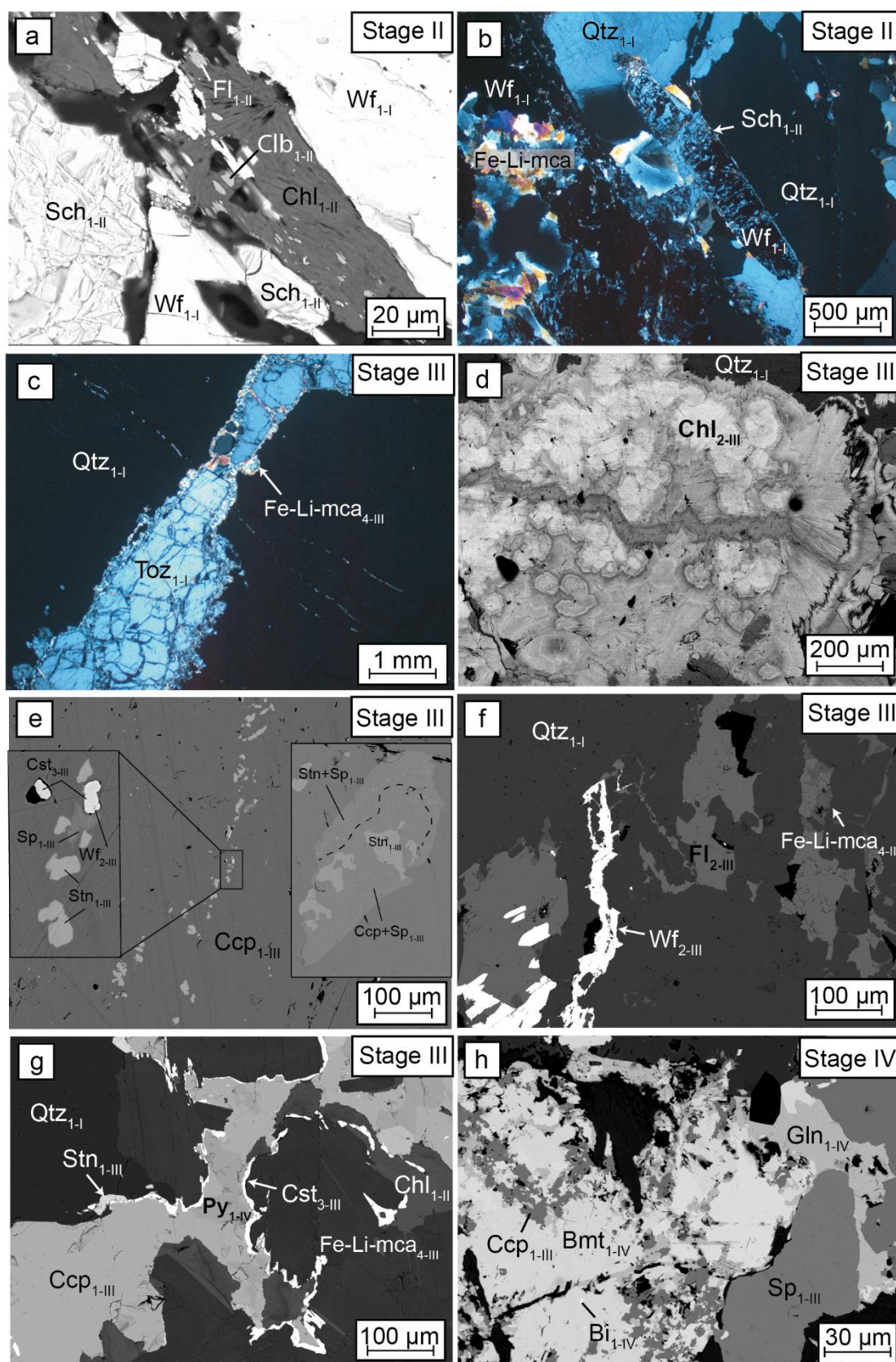


Figure 39. Microphotographs of the mineralogical textures and associations of the Piaotang deposit. (a) Alteration of Wf_{1-I} into Sch_{1-II} followed by the crystallization of associated Chl_{1-II} , Fl_{1-II} and Clb_{1-II} during stage II (SEM). (b) Textures of stage II retromorphosis of wolframite into scheelite associated with early stage I minerals (crossed polars). (c) Alteration of early stage I Toz_{1-I} by $Fe-Li-mca_{4-III}$ in stage III (crossed

polars). (d) Spherulites of chlorite that crystallized during the stage II (SEM). (e) Microcracks lining the Ccp_{1-IV} boundary with inclusions of Cst_{3-III}, Wf_{2-III}, Stn_{1-III}, and symplectic textures observed between Ccp_{1-III}, Sp_{1-III} and Stn_{1-III} (SEM). (f) Association of the second generation of Wf_{2-III} with Fl_{2-III} and Fe-Li-mca_{4-III} in stage III (SEM). (g) Association of a third generation of cassiterite Cst_{3-III} and Stn_{1-III} to stage IV sulphides (SEM) replacing second stage chlorite Chl_{1-II}. (h) Texture and association of Ccp_{1-III}, Sp_{1-III}, Gln_{1-IV}, Bmt_{1-IV}, Bi_{1-IV} in stage IV (SEM). Abbreviations according to the IMA: Bi = native bismuth; Bmt = bismuthinite; Ccp = chalcopyrite; Chl = chlorite; Clb = Colombo-tantalite; Cst = cassiterite; Fl = fluorite; Gln = galena; Mca = micas; Mlb = molybdenite; Py = pyrite; Qtz = quartz; Sch = scheelite; Sp = sphalerite; Stn = stannite Toz = topaz; Wf = wolframite.

Sulphide stage III: Although mainly marked at the macroscopic scale by sulphide deposition, this stage is characterized by a complex succession of phyllosilicates, sulphides and late wolframite and cassiterite. The Fe-Li-mica (Fe-Li-mca_{4-III}) developed first, mainly at the expense of a pervasively corroded Qtz_{1,2-I} or in the form of small veinlets overprinting earlier minerals as Chl_{1-II}. Fe-Li-mca_{4-III} is also observed in thin crack-seal re-openings at the vein boundaries (exhibiting shearing features) and in alteration of Toz_{1-I} (Figure 39c). Fe-Li-mca_{4-III} also appears as spherulites (up to 150 µm in diameter) in vuggy microcavities. Some overgrowths on earlier micas (mainly Fe-Li-mca_{1-I}) are associated with these spherulites. As seen in Figure 40, these overgrowths (Z1 to Z4) are zoned and associated with reworking of the early zinnwaldite/Li-Ms association, which is overprinted by micas similar to the first zone of the overgrowth. The zones consist of an alternation of Li-Fe-Ms and Li-Ms. In the Monier and Robert diagram (1986) (Figure 41c), these zones plot along the Fe-Li-mca_{4-III} trend. The end of the overgrowth is marked by distinctly Mg-rich (and Mn-poor) compositions, very similar to those of the Fe-Li-mca_{4-III} micas (Figure 40). Consequently, these overgrowths are considered to be coeval with the latter (Fe-Li-mca_{4b-III}). Sulphide deposition, coeval with a second generation of chlorite (Chl_{2-III}) (Figure 6d), follows chalcopyrite deposition (Ccp_{1-III}). Small euhedral prisms of a phenakite (Phk_{1-III}) are found as inclusions in the chalcopyrite. This phenakite could have trapped the beryllium leached from Brl_{1-I} when replaced by Fe-Li-mca_{4-III}. Chl_{2-III} deposition started with a Fe-chamosite composition and then evolved towards an Al-Fe clinocllore composition. Sphalerite (Sp_{1-III}) and stannite (Stn_{1-III}) followed the chalcopyrite deposition and appear as trails of more-or-less rounded inclusions (a few tens of µm in size). In detail, the sphalerite inclusions are zoned, with a core that is spotted with multiple

chalcopyrite micro-inclusions and a rim that exhibits symplectic stannite-sphalerite associations (Figure 39e). Chalcopyrite micro-inclusions in sphalerite are known as “chalcopyrite disease”, which is classically interpreted to result from a reaction between sphalerite and a permeating Cu-rich fluid (Barton et al., 1978, Bortnikov et al., 1991). In the present case, the texture could therefore be referred to as “sphalerite disease”, interpreted as the healing of cracks affecting chalcopyrite. In systematic spatial association with sphalerite and stannite in the chalcopyrite, small euhedral cassiterite crystals ($\text{Cst}_{3\text{-III}}$) are observed, which locally contain inclusions of Mn-wolframite ($\text{Fe}_{0.43}\text{Mn}_{0.57}\text{WO}_4$, $\text{Wf}_{2\text{-III}}$) (Figure 39f). $\text{Cst}_{3\text{-III}}$ is also present in the form of microcracks lining the chalcopyrite boundary. $\text{Wf}_{2\text{-III}}$ microcracks of hübneritic composition (from $\text{Fe}_{0.20}\text{Mn}_{0.80}\text{WO}_4$ to $\text{Fe}_{0.12}\text{Mn}_{0.88}\text{WO}_4$) are also observed. A late fluorite ($\text{Fl}_{2\text{-III}}$) is coeval with $\text{Cst}_{3\text{-III}}$ and $\text{Wf}_{2\text{-III}}$ (Figure 38f and g).

Sulfosalt stage IV: Overprinting all preceding mineral assemblages, a late generation of sulphured minerals has precipitated in the following sequence: galena (Bi-rich, up to 1 wt%), members of the lillianite series ($\text{Ag}_{0.8-1.1}\text{Pb}_{1.6-1.3}\text{Bi}_{2.6-2.7}\text{S}_6$), lead-rich bismuthinite (up to 5 wt%), and finally, native bismuth (Figure 39h).

5. Composition of micas

5.1 Major elements

All mica generations from the granite, greisen and the veins were studied by SEM and analysed by EPMA for major elements and LA-ICPMS for minor and trace elements. Representative EPMA analyses are presented in Table 3. A total of 170 EPMA and 98 LA-ICPMS analyses were performed.

The problem of Li content: The LA-ICPMS analyses (Table 4) showed that all Piaotang micas are Li-bearing micas. However, the micas appear to display complex internal patterns (see below) and the diameter of the LA-ICPMS spots (44 to 120 μm) was not small enough to measure most of the microstructures related to EPMA measurements (internal standardization

for LA-ICPMS). Moreover, being a highly volatile element, lithium remains difficult to quantify with precision by LA-ICPMS (up to 0.5 wt. % Li_2O). To avoid this “lithium issue”, the empirical regressions of Tischendorf et al. (1997) are used. The suitable regressions in our system would be SiO_2 for tri-octahedral micas and F for di-octahedral micas, and both were used in the present study. However, the results were inconsistent: in the Li-Al-R^{2+} diagram of Foster (1960), the Piaotang micas plot within the immiscibility gap determined by Monier and Robert (1986), meaning that our calculated LiO_2 contents (Li_2O^*) are underestimated. This problem was also encountered by Legros et al. (2016) in a study of the Maoping deposit and was resolved by adjusting the Li_2O^* contents (by adding a constant to the Li_2O^* values estimates from the Tischendorf et al. (1997) regressions). However, this method would be very complicated to apply in the case of the Piaotang deposit, where more trends are exhibited (Figure 41). Consequently, the lithium content of the micas has been removed from the diagrams.

Micas from granite: With an average Fe# ($\text{Fe}/(\text{Fe}+\text{Mg}+\text{Mn})$ ratio) of 0.55 and an average $\text{R}^{2+}/(\text{R}^{2+}+\text{Al})$ of 0.53, the granite micas plot between annite-phlogopite and eastonite-siderophyllite compositions (Figure 41a) and may be classified as lithian biotite.

Fe-Li-mca_{1,2-j}: The two first generations of micas appear as intergrowths of tri-octahedral and di-octahedral components. Two converging trends are displayed in the Monier and Robert (1986) diagram (Figure 41b): (i) a zinnwaldite-phlogopite trend (referred to as zinnwaldite in the following) and (ii) a trend towards Li-muscovite (about 0.5 trilithionite), referred to as Fe-Li muscovite in the following (Figure 41b). In detail, the micas consist of a succession of alternating compositions between Fe-Li muscovite (Li-Fe-ms) and zinnwaldite (Znw). Both Li-Fe-ms and Znw show a significant manganese content even though the zinnwaldite components always exhibit higher Mn contents (up to 1.9 wt. % MnO). In addition, the earliest Fe-Li-mca₁₋₁ are characterized by the development of a large overgrowth, on a core formed by the complex intergrowths described above (Figure 40). This overgrowth is zoned, showing a succession of Fe-Li-ms toward Li-ms trends with distinct Fe#, similar to the

composition of the core (Figure 40). In addition, the outer zones are characterized by very low Fe# and Mn contents.

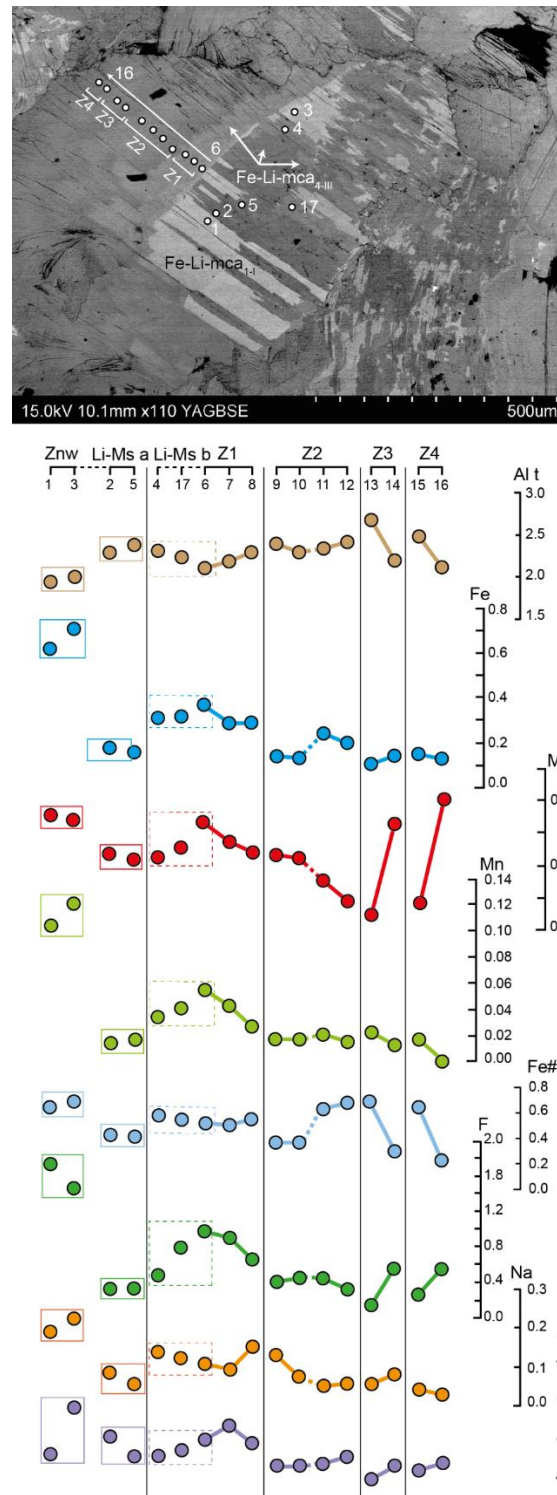


Figure 40. Detailed study of an altered Fe-Li-mca_{1-I} spherulite into Fe-Li-mca_{4-III}. The chemical profiles show the oscillatory changes in composition during alteration. All elements are presented as apfu values, calculated from EPMA data.

End of stage I micas: The Fe-Li-mca_{3-I} are either Znw or Li-Fe-ms, and exhibit very similar compositions to the earliest Fe-Li micas (Figure 41b).

Stage III micas: The Fe-Li-mca_{4-III} micas are all Fe-Li-ms, which, in the Al-Si-R²⁺ diagram, are displaced from their equivalent from stage I (Figure 41c). However, they exhibit distinctive low Fe# (0.2–0.4) and MnO (less than 1.0 wt. %) contents.

Greisen: The greisen micas are Fe-Li-ms, which, in the Monier and Robert (1986) diagram, are very similar to the Fe-Li-mca_{1-I} and Fe-Li-mca_{3-I} Fe-Li muscovites.

It is noteworthy that these data exhibit similar trends to the Maoping Li-micas analysed by Legros et al. (2016) in the same district (Figure 41d).

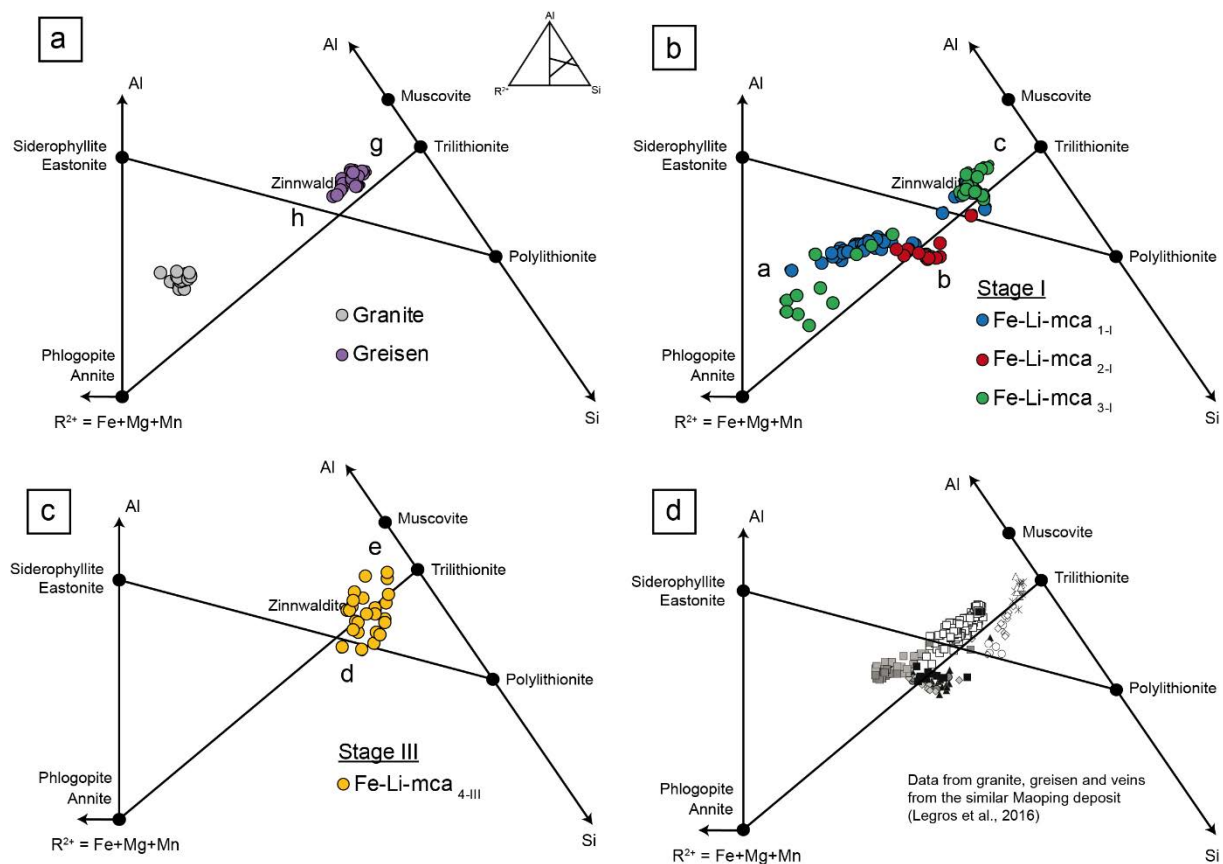


Figure 41. Compositions of micas plotted on a Monier and Robert (1986) phase diagram as a function of R³⁺ (Al), R²⁺ (Fe+Mg+Mn) and Si (apfu). Each generation of micas from the veins, granite and greisen displays an evolution trend that can be described using end-member compositions from (a) to (h) and be compared to similar published data from the Maoping deposit (Legros et al., 2016). See discussion section for explanations.

PART 1 – Chapter 3

Table 3. Selected major oxide contents from EPMA analyses and calculated structural formulae for micas from each stage, granite and greisen. Li₂O contents were calculated using Tischendorf et al. (1997). Values preceded by the symbol '<' indicate element concentrations below the limit of detection.

EPMA (wt%)	Fe-Li-micas _{1-I} (N=54)	Fe-Li-micas _{2-I} (N=13)	Fe-Li-micas _{3-I} (N=26)	Fe-Li-micas _{4-III} (N=26)	Granite (N=30)	Greisen (N=20)
SiO ₂	35.42	44.44	45.36	47.46	35.54	43.79
TiO ₂	<0.05	0.06	<0.05	0.27	1.85	0.07
Al ₂ O ₃	21.16	21.61	28.79	26.61	19.38	27.75
FeO	24.58	8.46	7.13	4.84	24.70	9.16
MnO	1.25	1.57	0.26	0.72	1.23	1.47
MgO	1.18	3.26	0.66	2.61	2.20	0.08
CaO	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07
Na ₂ O	0.06	0.09	0.16	0.07	0.10	0.13
K ₂ O	9.59	10.77	10.65	9.83	8.89	8.93
Rb ₂ O	0.67	0.62	0.33	0.48	0.15	0.40
Li ₂ O *	2.07	4.68	2.87	2.54	2.11	2.40
F	2.24	6.91	1.82	4.07	0.73	2.02
O=F	1.12	3.45	0.91	2.03	0.36	1.01
H ₂ O	3.49	3.09	4.09	3.73	3.77	3.92
Total	100.12	100.65	100.83	100.34	100.14	99.63
Atoms per 12 [O, OH, F]						
Si	2.64	2.8	3.01	3.03	2.70	2.99
^{IV} Al	1.36	1.2	0.99	0.97	1.30	1.01
Sum Tet.	4.00	4.00	4.00	4.00	4.00	4.00
Ti	0.00	0.00	0.00	0.01	0.11	0.00
^{VI} Al	0.50	0.43	1.26	1.04	0.43	1.22
Fe	1.53	0.45	0.40	0.26	1.57	0.52
Mn	0.08	0.08	0.01	0.04	0.08	0.08
Mg	0.13	0.31	0.07	0.25	0.25	0.01
Ca	0.00	0.00	0.00	0.00	0.00	0.00
Li	0.62	1.20	0.77	0.65	0.65	0.66
Sum Oct.	2.86	2.47	2.51	2.25	3.09	2.49
K	0.91	0.87	0.90	0.80	0.86	0.78
Rb	0.03	0.03	0.01	0.02	0.01	0.02
Na	0.01	0.01	0.02	0.01	0.01	0.02
Sum A	0.95	0.91	0.93	0.83	0.88	0.82
F	0.26	0.69	0.19	0.41	0.09	0.22
OH	1.74	1.31	1.81	1.59	1.81	1.78

5.2 Trace elements

Micas from granite: These micas are characterized by elevated Nb and Ta contents ($\text{Nb}+\text{Ta} > 100$ ppm; Figure 42a), with variable Nb# ($\text{Nb}/(\text{Nb}+\text{Ta})$) ranging from 0.91 to 0.67. They have the lowest W (<10 ppm) and Sn (<200 ppm) contents of all micas analysed in this study. A vertical positive correlation between Nb# and Mn content is observed in these micas (Figure 42b).

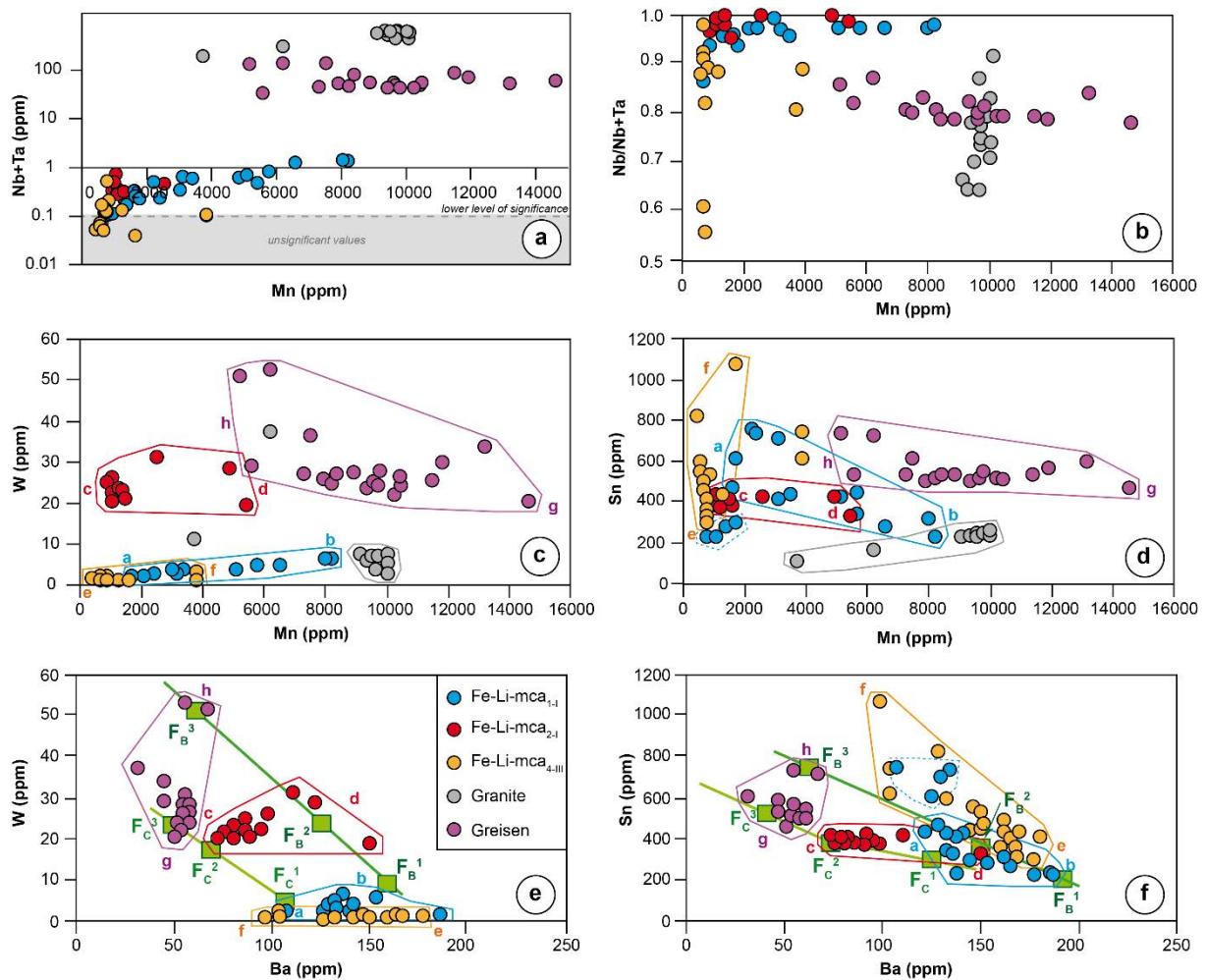


Figure 42. Trace element binary diagrams for three generations of micas from the veins, the granite and the greisen in the Piaotang deposit (data from LA-ICPMS analysis). The chosen binary diagrams aim to unravel the behavior of different metals observed in trace amounts in micas and separate the evolution of each generation. “F_x” refer to fluids identified in the discussion section (see text for further explanation). The granite mica data have been removed from (e) and (f) binary diagrams for better visibility.

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Table 4. LA-ICPMS analyses of trace elements in micas from three generations in veins, granite and greisen. Zr, U, Y, and REE were also analyzed but were systematically below 1 ppm.

LA-ICPMS	Spot	Mg (ppm)	±	Ti (ppm)	±	Mn (ppm)	±	Co (ppm)	±	Nb (ppm)	±	Mo (ppm)	±	Sn (ppm)	±	Ba (ppm)	±	Ta (ppm)	±	W (ppm)	±
Fe-Li-mca _{1,4}	1	7500	52	781.3	8.1	4835	28	3.04	0.11	0.622	0.027	0.033	0.041	436.6	3.3	121.8	1.5	0.0061	0.0043	28.75	0.43
	2	12003	82	853.2	7.9	5390	32	3.82	0.11	0.476	0.021	0.085	0.039	335.1	2.8	149.7	1.6	0.0094	0.0042	19.53	0.59
	3	16040	100	1039	11	6554	80	2.94	0.11	1.217	0.04	0.109	0.049	281.9	2	153.4	2.1	0.0379	0.0065	5.02	0.17
	4	16800	120	936.1	8.7	8199	46	2.528	0.088	1.319	0.041	0.132	0.043	226.8	2.1	136.5	1.4	0.0363	0.0066	6.72	0.21
	5	17880	110	966.7	8.6	8033	46	2.9	0.092	1.372	0.036	0.107	0.046	319.4	2.7	135.9	1.5	0.0453	0.0076	6.46	0.19
	6	16170	110	690.6	7.8	5741	49	1.879	0.085	0.796	0.027	0.111	0.043	339.2	2.7	131.6	1.3	0.0256	0.0051	4.94	0.19
	7	10104	98	609.1	5.3	2191	16	1.199	0.065	0.489	0.023	0.046	0.038	748.1	5.9	107.3	1.4	0.0149	0.0044	2.56	0.13
	8	11531	86	450.2	5.6	2398	15	0.878	0.058	0.237	0.016	0.026	0.035	738.2	6.1	133.7	1.7	0.0062	0.0036	2.71	0.14
	9	15816	91	593.4	5.5	5085	27	2.248	0.078	0.681	0.027	0.082	0.039	423	9	140.9	1.8	0.0205	0.0046	4.19	0.15
	10	13241	82	448.7	5.7	3012	28	1.142	0.064	0.348	0.02	0.029	0.035	409.4	4.3	137	1.7	0.0048	0.0036	3.48	0.14
	11	12963	77	329.6	4.8	1612	13	0.839	0.055	0.313	0.021	0.02	0.045	470.2	3.1	127.8	1.7	0.0146	0.0057	1.71	0.1
	12	8628	49	272.7	4.3	1622.9	9.8	0.937	0.064	0.245	0.018	0	0.036	610.3	4.2	125.4	1.5	0.0161	0.0057	1.95	0.11
	13	16169	98	199.4	3.4	830.9	5.9	0.623	0.047	0.099	0.011	0.011	0.038	217	1.6	187	2.2	0.0074	0.0042	1.125	0.083
	14	17290	130	209.7	3.7	933	8.5	0.633	0.047	0.106	0.011	0.055	0.043	243.3	2.2	185.5	2.3	0.0086	0.004	1.18	0.082
	15	13490	84	614.4	9.6	3102	23	1.017	0.06	0.618	0.028	0	0.037	705.5	5.7	130.7	1.5	0.0217	0.0051	2.86	0.14
	16	10470	100	509.8	5.6	3396	20	1.272	0.066	0.573	0.026	0.033	0.037	435.6	3	133.1	1.7	0.03	0.0059	3.47	0.13
	17	10697	94	248.9	4.1	1761	14	0.844	0.051	0.212	0.016	0.013	0.035	295.9	2.8	144.1	1.7	0.0146	0.005	1.708	0.091
	18	16150	140	225.6	4.3	1369	17	0.663	0.046	0.165	0.013	0.058	0.036	274.2	3.5	164.1	2	0.0068	0.0039	1.439	0.088
	19	15870	140	188.3	3.3	754.3	7.3	0.625	0.04	0.113	0.012	0	0.029	230.2	2.5	177.2	2.1	0.0118	0.0042	1.018	0.068
	20	17480	200	199.6	3.4	719	10	0.597	0.044	0.101	0.011	0.1	0.044	319	3.2	162	2	0.015	0.0042	1.079	0.068
Fe-Li-mca _{2,4}	1	11283	58	367.7	4.1	1059.2	6.3	3.93	0.12	0.28	0.015	0.013	0.025	388.5	2.8	76	1.1	0.008	0.0033	21.49	0.34
	2	10467	61	454.5	4.4	1000.9	6.4	3.68	0.094	0.317	0.016	0	0.019	389.6	3.7	80.4	1.1	0.01	0.003	21.87	0.37
	3	12117	86	352.7	5.5	1075.3	8.7	4.438	0.098	0.347	0.017	0.01	0.023	376.9	4.1	77.5	1.2	0.011	0.003	21.58	0.35
	4	13124	86	342.3	4.1	916.7	8.7	4.06	0.12	0.316	0.018	0.013	0.026	425.2	4.5	73.4	1.1	0.0123	0.0034	20.95	0.34
	5	11990	100	417.2	4.3	1077.9	8.8	4.1	0.12	0.351	0.019	0.039	0.027	392.3	4.9	80.4	1.4	0.0071	0.0031	20.82	0.41
	6	9731	72	406.9	5.1	1252.6	9.9	3.621	0.095	0.258	0.016	0.013	0.024	411.6	4.6	80.4	1.2	0.0036	0.0025	21.23	0.35
	7	10308	86	392.8	5	1051.6	9.6	3.76	0.11	0.276	0.017	0.012	0.024	384.2	4.3	85.7	1.3	0.0077	0.0029	22.68	0.41
	8	8050	69	413.3	4.7	1322	11	3.71	0.098	0.2	0.012	0	0.019	389	4.9	79.2	1.2	0.0016	0.002	21.34	0.36
	9	8506	79	346.3	4.6	1405	11	3.57	0.12	0.22	0.017	0	0.023	399.5	5.1	80.6	1.4	0.0015	0.0025	22.66	0.4
	10	5877	36	464.4	4.6	2538	18	2.549	0.083	0.468	0.024	0.069	0.033	422.3	4.5	110.1	1.4	0.0045	0.0031	31.28	0.45
	11	9310	48	524	5.8	1342.5	8.1	3.73	0.1	0.275	0.019	0.031	0.031	373	2.7	88.9	1.3	0.0081	0.0042	22.38	0.35
	12	9874	72	361.7	4.3	891	11	3.562	0.098	0.511	0.024	0	0.025	385.6	2.4	87	1.1	0.0175	0.0039	25	0.39
	13	11140	59	363.9	4	1037	12	3.82	0.099	0.714	0.025	0.023	0.028	388	2.9	98.1	1.2	0.0187	0.0037	25.82	0.35
	14	8043	53	411.6	5.4	1301.7	6.9	3.466	0.085	0.202	0.013	0.018	0.024	384.4	2.6	81.5	1	0.0057	0.0025	23.28	0.32
	15	12620	180	428.8	4.5	972.2	9	3.863	0.089	0.493	0.02	0.018	0.024	435	5.2	91	1	0.0162	0.0031	22.57	0.36
	16	10185	75	531.5	4.3	1528	14	3.746	0.097	0.262	0.015	0.018	0.028	387.8	3.2	88.8	1.1	0.0128	0.0033	21.09	0.33
	17	9480	130	426.4	4.1	1091.3	9.9	3.782	0.09	0.276	0.013	0.016	0.021	394.8	2.9	87.79	0.94	0.0098	0.0029	24.56	0.36
	18	9048	56	513.4	5.5	1276.1	6.6	3.498	0.094	0.316	0.015	0	0.022	398.6	2.8	95.2	1	0.0094	0.0031	22.45	0.33

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Table 4. Continued.

LA-ICPMS	Spot	Mg (ppm)	±	Ti (ppm)	±	Mn (ppm)	±	Co (ppm)	±	Nb (ppm)	±	Mo (ppm)	±	Sn (ppm)	±	Ba (ppm)	±	Ta (ppm)	±	W (ppm)	±
Fe-Li-mca _{4-III}	1	21990	110	193	3.5	534.5	2.9	0.589	0.052	0.057	0.011	0.013	0.043	459.5	3.9	151	1.9	0.0019	0.0038	1.025	0.08
	2	19844	82	204.4	3.4	593.4	2.8	0.68	0.049	0.067	0.011	0.021	0.043	493.2	3.5	160.5	1.6	0.0034	0.004	1.101	0.089
	3	20853	90	200.8	3.4	554.4	2.9	0.659	0.052	0.0628	0.0098	0.151	0.068	478.5	3.9	152.2	1.6	0.002	0.0039	1.016	0.091
	4	21350	140	190.3	3.2	673.8	4.5	0.792	0.053	0.11	0.011	0.002	0.037	429.6	6.1	169.7	1.8	0.0096	0.0046	1.26	0.096
	5	18460	100	195.5	3.2	696.2	4.2	0.617	0.043	0.121	0.013	0.026	0.039	415.5	3.4	179	1.8	0.0028	0.0041	1.269	0.091
	6	18220	110	202.1	3.3	719.6	4.5	0.698	0.05	0.144	0.013	0.29	0.099	414.2	5.3	164.6	1.8	0.015	0.0049	1.96	0.25
	7	21200	130	186.9	3.2	398.8	4.4	0.716	0.049	0.0461	0.0082	0.24	0.26	820.2	5.9	128	1.4	0.0082	0.004	1.45	0.16
	8	39430	660	229.6	3.8	1640	65	1.824	0.089	0.0343	0.0075	0.014	0.039	1063	11	97.8	1.2	0.0057	0.0034	0.885	0.095
	9	20290	130	192	3.3	542.3	3.6	0.646	0.046	0.0639	0.0091	0	0.035	532.4	3.7	149.6	1.7	0.0072	0.0039	1.003	0.078
	10	15390	130	217.2	3.4	816	5.5	0.787	0.053	0.191	0.015	0.026	0.042	534.6	4.2	148.9	1.7	0.0233	0.0053	1.76	0.11
	11	27270	190	188.3	3	1230.9	9.1	2.083	0.085	0.119	0.011	0.098	0.048	440	4	160.7	1.8	0.0162	0.0043	1.377	0.093
	12	69690	640	187	3.3	3826	35	10.83	0.23	0.091	0.011	0.074	0.047	742.9	7.1	103.3	1.4	0.0117	0.0038	2.8	0.62
	13	18980	190	183.6	2.9	704.2	6.6	0.808	0.055	0.126	0.011	0.05	0.037	305.7	2.8	175.7	2	0.0176	0.0042	1.091	0.081
	14	19860	150	188.9	3.2	538	4.3	0.645	0.045	0.055	0.0093	0.008	0.034	566.9	4.6	144.6	2	0.0079	0.0035	1.373	0.084
	15	67500	500	180.1	3.3	3826	30	10.91	0.21	0.097	0.011	0.084	0.053	619.5	5.3	102.8	1.6	0.0132	0.004	1.21	0.11
	16	20820	330	191.1	3.2	740	15	0.992	0.069	0.104	0.01	0.019	0.033	321.9	7.4	168.3	1.8	0.0227	0.0041	1.202	0.081
	17	20510	170	195.5	3.1	651.4	5.8	0.712	0.043	0.0591	0.0088	0.047	0.039	367	3.5	158.3	1.6	0	0.0037	1.149	0.071
	18	20080	150	195.2	2.7	517.8	3.9	0.713	0.051	0.0229	0.0069	0	0.028	596.4	5.4	133	1.5	0	0.0034	1.009	0.075
	19	20600	160	203.8	3.1	600.7	4.8	0.689	0.045	0.1039	0.0072	0.027	0.03	444.7	4.5	145.8	1.5	0.0664	0.0042	1.093	0.075
	20	17900	180	198.9	3.3	764.9	9.3	0.793	0.046	0.293	0.012	0.001	0.032	364	3.1	167.3	1.8	0.2328	0.0055	1.127	0.07
Granite	1	13250	410	3400	770	3730	840	4.4	1	154	37	0.79	0.26	98	21	36.9	2.6	36.8	8.8	11	2.4
	2	12850	450	6300	1100	6200	1000	7.2	1.3	263	46	1.22	0.36	165	26	103	14	39.5	7.3	37.7	7.3
	3	11100	170	9200	150	9730	130	11.67	0.6	441.8	7	1.34	0.35	229	4.2	26.4	1.6	159.3	3	5.4	0.5
	4	11950	250	9100	160	9730	160	11.08	0.4	435.1	5.8	1.88	0.35	243.1	3.7	122.3	3.4	129.7	2.7	5.07	0.44
	5	12030	240	8910	150	9650	140	11.01	0.48	425.3	6.7	1.9	0.36	246	3.1	1006	27	126.6	2.4	5.27	0.37
	6	11310	250	8490	160	9410	120	10.74	0.56	415.3	6.1	1.3	0.25	241.9	4.7	643	13	120.6	3	6.23	0.48
	7	12130	250	8230	130	10020	140	11.78	0.54	447.9	7.6	1.46	0.4	249	4.5	69.1	2.6	158.8	3.3	6.33	0.37
	8	11320	190	9450	180	9870	120	10.98	0.49	442.2	8.1	1.6	0.38	243.1	4.4	15.77	0.99	119.2	2.6	5.23	0.34
	9	11050	160	8190	120	9330	120	10.68	0.43	410.8	6.7	0.9	0.26	238.4	4.2	215.9	4.3	225.8	3.9	6.15	0.39
	10	11410	220	8040	140	9700	130	10.88	0.47	420.5	7.3	0.76	0.28	239.6	3.7	129.2	3	235.1	4	6.94	0.5
	11	11460	260	9420	110	10000	100	10.88	0.49	459.6	5.6	1.34	0.3	239.5	3.9	11.88	0.86	163.5	3.2	6.21	0.45
	12	10850	150	9230	150	9800	140	11.76	0.57	442.4	6.7	1.12	0.34	231.3	4.6	6.75	0.7	152.7	3.9	5.18	0.41
	13	11510	220	9470	180	9650	140	11	0.43	460.1	7.5	1.1	0.3	236.7	4	79.9	4.1	155	3.9	4	0.33
	14	11810	240	9930	140	10030	120	11.74	0.61	428.6	6.1	1.36	0.28	244.7	4	34.1	1.5	91.6	1.7	4.47	0.41
	15	12350	310	10910	150	10070	100	11.75	0.55	412.7	5.2	1.76	0.37	252.2	3.8	100.6	3.1	39.35	0.76	2.28	0.22
	16	12230	300	10980	160	9670	110	11.17	0.54	384.2	5.2	2.06	0.4	250.4	4.1	1134	22	59.1	1.4	4.33	0.32
	17	11680	200	9250	130	10100	120	11.63	0.58	442.7	5.9	1.19	0.31	241.8	3.7	139.8	7.1	156.3	3	4.8	0.36
	18	11310	120	8920	110	10000	100	11.1	0.64	431	5.2	0.93	0.29	240.3	3.9	33.9	1.6	178.1	2.3	7.77	0.44
	19	11400	170	9070	120	9500	120	11.04	0.44	432.6	6.4	1.28	0.27	236.7	3.1	110	2.7	185.8	3.2	6.46	0.44
	20	11130	240	8120	160	9080	120	10.4	0.55	382.4	6.6	0.59	0.24	236.4	4.5	200.9	7.9	191.2	3.8	7.77	0.48

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Table 4. Continued.

LA-ICPMS	Spot	Mg (ppm)	±	Ti (ppm)	±	Mn (ppm)	±	Co (ppm)	±	Nb (ppm)	±	Mo (ppm)	±	Sn (ppm)	±	Ba (ppm)	±	Ta (ppm)	±	W (ppm)	±
Greisen	1	366.9	7.4	394	16	10420	150	2.03	0.33	39.53	0.88	0.08	0.33	516.8	8.3	54	2.1	10.24	0.39	26.7	1.1
	2	464	9.4	624	22	11900	140	2.28	0.28	55	1.2	0.29	0.35	566.3	8.1	55.3	1.9	15.13	0.39	30.2	1.1
	3	573	10	1205	28	11480	130	2.34	0.34	69.2	1.4	0	0.3	535.8	7.4	55.7	1.9	17.8	0.48	25.6	1.3
	4	254.6	5.3	197.4	8.7	13180	140	1.7	0.28	44.97	0.98	0	0.27	595	11	45.3	1.9	8.85	0.28	33.9	1.2
	5	410	13	476	29	10460	200	1.2	0.43	43	2	0.09	0.57	505	12	59.3	4.8	11.58	0.56	24.3	1.8
	6	425	14	379	23	10230	190	0.93	0.36	34.7	1.4	0.19	0.42	516	11	53.8	2.9	9.21	0.5	22.6	1.6
	7	927	13	517	22	7891	95	1.62	0.28	44.41	0.95	0	0.25	492.3	7.9	56.3	2.4	9.16	0.28	26.1	1.1
	8	513	12	515	21	9610	150	2.11	0.38	43.84	0.89	0.24	0.43	518.4	9.9	56.7	2.9	11.95	0.42	25.5	1.5
	9	423	10	460	19	8860	120	1.8	0.3	43.8	1	0.13	0.32	532	10	57	2.9	11.86	0.41	27.7	1.3
	10	422.1	9.2	405	17	9660	120	2.03	0.34	39.4	0.81	0	0.37	517.7	6.8	57.6	3.1	9.9	0.3	24.1	1.2
	11	466.7	9.7	374	17	8230	100	1.95	0.37	37.78	0.96	0	0.26	511.9	8.5	56.5	2.6	8.98	0.37	24.9	1.1
	12	1430	23	225	15	5572	65	1.08	0.24	27.62	0.77	0	0.27	532.7	8.2	46	2.9	6.17	0.28	28.9	1.4
	13	806	16	689	24	8390	100	2.1	0.32	63.8	1.2	0	0.29	530.6	8.3	52.9	2.7	17.42	0.42	27.3	1.1
	14	393.9	7.8	247	13	9780	130	1.85	0.31	35.56	0.72	0	0.26	539.9	8.5	58.2	2.4	8.17	0.33	28	1.2
	15	2364	35	998	27	7515	88	1.74	0.25	111.6	1.5	0.03	0.27	606	10	31.7	2.1	28.94	0.66	36.5	1.4
	16	2949	32	840	24	5148	53	1.74	0.29	111.9	1.9	0.48	0.29	722.6	8.1	66.8	2.4	19.24	0.57	51	1.7
	17	2801	43	958	30	6186	76	1.69	0.23	118.4	2.2	0.1	0.22	727	12	55.7	2.3	18.44	0.48	52.8	1.4
	18	505.7	9.2	277	16	7290	110	1.58	0.27	35.75	0.97	0.31	0.32	532	10	60.4	2.5	8.82	0.37	27.1	1.3
	19	517	11	313	15	9410	200	1.99	0.39	35.69	0.86	0.13	0.22	504	13	52.5	3.2	7.87	0.3	24	1.1
	20	616	16	966	29	14590	270	2.46	0.33	47.2	1.4	0.16	0.25	461	13	50.4	3	13.47	0.54	20.4	1.1

Vein micas: As seen in Figure 42a and Figure 42c, the first (Fe-Li-mca_{1-I}) and fourth (Fe-Li-mca_{4-III}) generations of micas display positive correlations between Mn and W or Nb+Ta, the Fe-Li-mca_{4-III} being the most depleted. A group of Fe-Li-mca_{1-I} analyses are displaced from the overall trend and most likely represent the Fe-Li-mca_{4-III} overgrowth (Z1 to Z4, Figure 40) and, indeed, these micas plot with the Fe-Li-mca_{4-III} micas in all diagrams. In contrast, the Fe-Li-mca_{2-I} micas plot as a cluster in all diagrams (Figure 42a, c and d). Compared to other generations of micas, the Fe-Li-mca_{2-I} micas are the most W-enriched (20 to 30 ppm), whereas the Fe-Li-mca_{1-I} micas are distinguished by higher Nb+Ta (up to 1.4 ppm). The Fe-Li-mca_{4-III} micas are characterized by elevated Sn contents (up to 820 ppm) and very low W and Nb+Ta values. Due to the LA-ICPMS spot size, Fe-Li-mca_{3-I} micas could not be analysed for trace element contents.

Greisen micas: Compared to the micas analysed in veins, the greisen micas are enriched in Nb and Ta (Figure 42a) and exhibit constant (close to the granite average) Nb# (Figure 42b). In Figure 41a and b, the greisen micas also share some similarities with the stage I micas.

6. Composition of chlorites

A total of 33 EMPA analyses were performed on chlorites (Chl_{1-II} and Chl_{2-III}) that crystallized in the veins in stages II and III. Analyses were carried out on homogeneous Chl_{1-II} and 100-µm size spherulites of Chl_{2-III}, perpendicular to their Fe-Mg zonation (Table 5, Figure 43). All of these chlorites were found to be tri/dioctahedral Fe-chlorites with compositions that lie between ripidolite-brunsvigite and pycnochlorite fields. In the Al^{IV} versus Al^{VI} plot, both generations of chlorite are displaced from the Tschermak line, which is always the case for low-temperature chlorites (Figure 43c; Cathelineau and Nieva, 1985). The Chl_{2-III} chlorites present FeO contents of 17 to 35%, with Fe/(Fe+Mg) ratios of 0.3 to 0.8 (Table 5). From the core to the margin of the spherulites, major elements, especially FeO and MgO concentrations, show oscillatory variations (Figure 43a and b), but no general trend can be distinguished.

PART 1 – Chapter 3

Table 5. Electron microprobe analyses of chlorites from the Piaotang mineralized veins sampled at the level -388 m. Values preceded by the symbol '<' indicate element concentrations below the limit of detection.

EPMA (wt.%)	Chlorite _{1-II}	Chlorite _{2-III}																				
	representative data	388-13-2	388-13-3	388-13-4	388-13-5	388-13-10	388-11-19	388-11-20	388-11-21	388-11-22	388-11-23	388-11-24	388-11-25	388-11-26	388-11-27	388-11-28	388-11-29	388-11-30	388-11-31	388-11-32	388-11-33	388-11-34
Al ₂ O ₃	18.16	20.97	20.27	21.04	21.47	21.23	20.04	19.79	20.04	20.74	20.61	20.88	19.62	20.9	21.45	17.48	20.82	20.54	19.9	21	20.38	21.04
SiO ₂	29.35	23.47	27.21	25.16	26.23	23.89	26.65	26.91	27.21	27.42	26.52	26.67	25.52	27.08	24.73	31.99	26.24	26.73	26.32	26.75	27.91	27.75
TiO ₂	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Na ₂ O	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07
MgO	15.3	5.89	15.32	8.6	13.4	5.05	13.39	10.19	17.27	15.42	12.24	11.43	10.36	15.11	10.72	22.33	12.77	12.48	9	14.6	17.72	16.3
MnO	0.72	2.61	1.19	0.66	3.79	2.32	0.55	3.75	0.46	0.64	1.8	1.87	1.74	1.25	1.2	0.47	0.79	0.54	2.64	1.26	0.86	0.83
FeO	23.09	32.93	21.8	31.1	21.44	33.97	24.84	26.68	19.22	21.92	28.21	28.07	28.57	23.5	28.21	16.92	26.59	26.46	28.88	22.64	19.43	21.71
K ₂ O	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	0.07	<0.07	<0.07	<0.07	<0.07	<0.07	0.18	<0.07	<0.07	<0.07
CaO	<0.07	0.11	<0.07	0.1	0.14	<0.07	<0.07	<0.07	0.1	<0.07	<0.07	<0.07	0.07	0.08	0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07	<0.07
Total	86.62	86.01	85.86	86.66	86.47	86.51	85.62	87.4	84.34	86.26	89.44	88.97	86.03	87.98	86.44	89.28	87.25	86.79	86.96	86.36	86.34	87.67
Atoms per 14 O																						
Si	3.08	2.68	2.88	2.77	2.79	2.71	2.87	2.91	2.88	2.87	2.79	2.82	2.82	2.82	2.70	3.14	2.80	2.86	2.88	2.83	2.89	2.86
Al ^{IV}	2.24	1.32	1.12	1.23	1.21	1.29	1.13	1.09	1.12	1.12	1.21	1.18	1.18	1.18	1.30	0.86	1.20	1.14	1.123	1.17	1.11	1.14
Al ^{VI}	0.92	1.51	1.40	1.51	1.48	1.55	1.41	1.43	1.38	1.44	1.35	1.42	1.37	1.38	1.46	1.15	1.41	1.44	1.45	1.44	1.37	1.41
Mg	1.32	1.00	2.41	1.41	2.12	0.85	2.15	1.64	2.73	2.41	1.92	1.80	1.71	2.34	1.75	3.26	2.03	1.99	1.47	2.30	2.73	2.50
Fe ²⁺	2.39	3.14	1.93	2.87	1.913	3.23	2.23	2.41	1.70	1.92	2.48	2.48	2.64	2.04	2.58	1.39	2.37	2.36	2.65	2.00	1.68	1.87
Mn	2.02	0.25	0.11	0.06	0.34	0.22	0.05	0.34	0.04	0.06	0.16	0.17	0.16	0.11	0.11	0.04	0.07	0.05	0.24	0.11	0.07	0.07
Ti	0.06	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Na	0	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.01	0.00	0.01	0.00	0.01	0.01	0.00	0.01
K	0	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.02	0.00	0.00	0.00
Ca	0	0.01	0.00	0.01	0.02	0.00	0.01	0.01	0.01	0.01	0.01	0.00	0.01	0.01	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00
Σcat Oct	5.74	5.64	5.74	5.79	5.51	5.64	5.79	5.48	5.81	5.77	5.76	5.71	5.72	5.77	5.79	5.81	5.82	5.80	5.57	5.74	5.79	5.79
R ₂ ⁺	4.42	4.14	4.34	4.28	4.03	4.08	4.38	4.05	4.43	4.33	4.41	4.28	4.34	4.39	4.32	4.65	4.40	4.35	4.12	4.30	4.41	4.37
R ₃ ⁺	2.29	3.00	2.60	2.78	2.93	2.99	2.59	2.75	2.54	2.61	2.67	2.72	2.68	2.65	2.85	2.05	2.67	2.62	2.74	2.70	2.54	2.60
Fe/(Fe+Mg)	0.46	0.76	0.44	0.67	0.47	0.79	0.51	0.60	0.38	0.44	0.56	0.58	0.61	0.46	0.60	0.30	0.54	0.54	0.64	0.46	0.38	0.43
K ₂ O+Na ₂ O+CaO	0.1	0.12	0.06	0.1	0.14	0.03	0.13	0.06	0.1	0.08	0.06	0.05	0.2	0.13	0.13	0.08	0.04	0.03	0.22	0.08	0.03	0.04

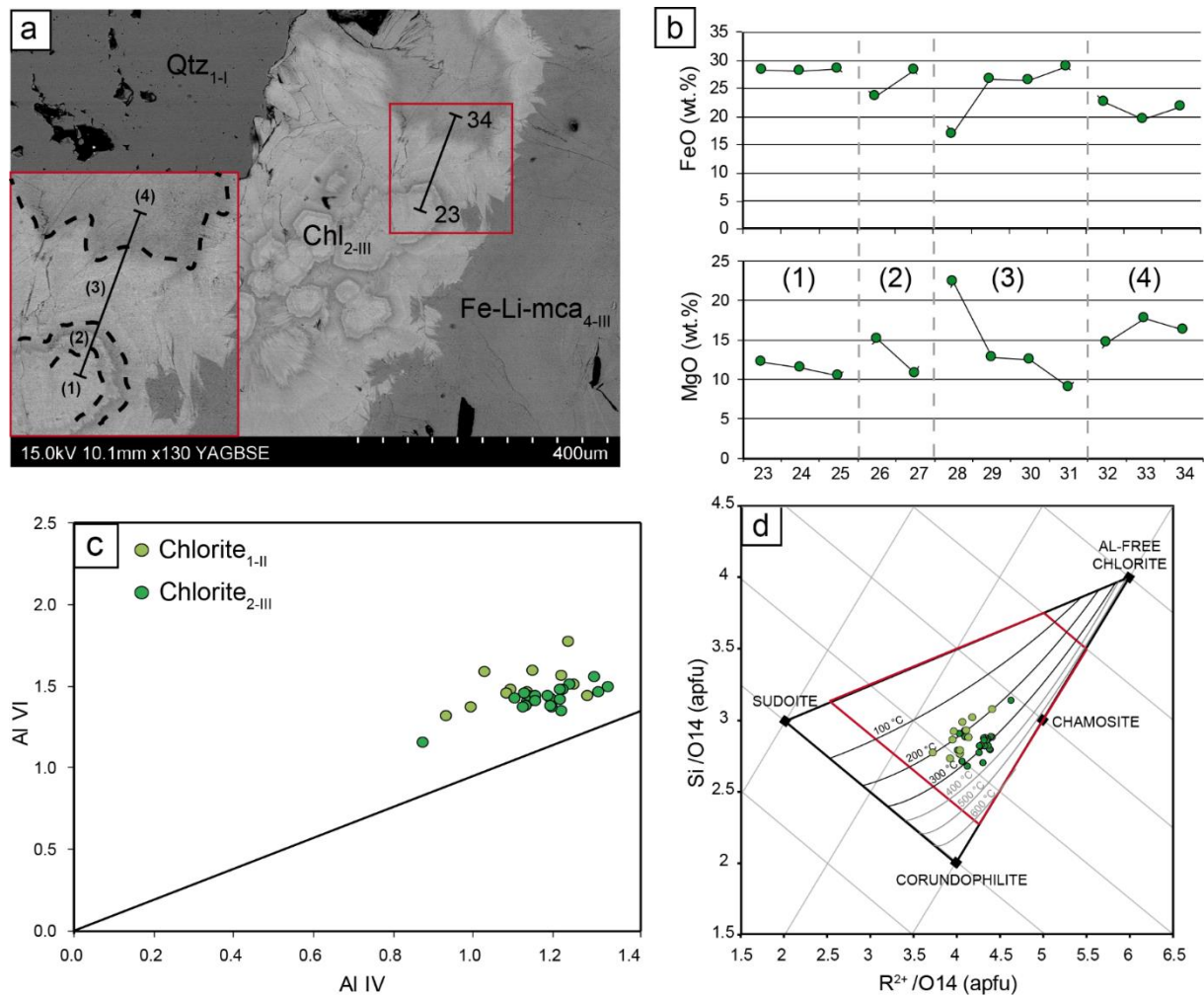


Figure 43. (a) Zoning in stage II chlorites and an example of the spatial distribution of analytical points across the zoning (SEM). (b) Compositional profiles corresponding to chemical variations in FeO and MgO along the transect shown in (a) (EMPA data). (c) Octahedral Al (apfu) vs. tetrahedral Al (apfu) in stage II and stage III chlorites (EMPA data). The line corresponds to the Tschermak line. (d) Distribution of temperature determined by chlorite graphical thermometry (Cathelineau and Bourdelle, 2015) and plotted in a T-R²⁺-Si diagram.

Temperatures were calculated using the graphical geothermometer of Bourdelle and Cathelineau (2015) and plotted in the Si-R²⁺ diagram of Wiewiora and Weiss (1990) (Figure 43d). This thermometer is based on the chlorite-quartz equilibrium. It does not require prior knowledge of the valence state of iron and is calibrated for low-T chlorites ($T < 350^{\circ}\text{C}$ and pressures below 4 kbar). The temperatures obtained range from 180 to 260°C for Chl_{1-II} (average temperature of 200°C) and 200 to 380°C for Chl_{2-III} (average temperature of 300°C) and correspond to temperatures reached during the second and third stages of crystallization

of the veins. No correlation between the calculated temperatures and the petrographic zoning in the Chl_{2-III} or chemical composition was observed.

7. Discussion

7.1 Major elements in hydrothermal micas: evidence for fluid mixing

Fe-Li-mca_{1,2-I} micas: In the Al-Si-R²⁺ diagram (Figure 41), the early Fe-Li-mca_{1,2-I} display two trends: a “phlogopite-zinnwaldite” trend between two poles **a** and **b**, and a “zinnwaldite-muscovite” trend between the same **b** pole and a muscovite **c** pole (Figure 41b).

The systematic alternation between zinnwaldite and muscovite compositions during the growth of Fe-Li-mca_{1,2-I} indicates the coexistence, at this time in the Piaotang hydrothermal system, of three distinct hydrothermal fluids responsible for the **a-b** and **b-c** trends. The **b-c** trend may be interpreted as a mixing trend between two fluids, one richer in Fe and Mg and the other, richer in Al, that co-precipitate in either Fe-Li-mca_{1-I} or Fe-Li-mca_{2-I}. By contrast, the zinnwalditic **a-b** trend can be interpreted in terms of closed system crystallization (correlated Fe and Mg contents). The **a**-pole is in fact associated with a primitive fluid end-member (F_A), whereas the **b**-pole is associated with the evolution of F_A towards a F_B composition by fractional crystallization (closed system). Moreover, the **b-c** trend might be interpreted to be the result of an opening of the system, with the arrival of a new F_C fluid end-member (associated with the **c**-pole), and a mixing of F_C with the residual F_B fluid. This trend is also observed in the greisen micas (**g-h** trend). According to this interpretation, the complex structure of the Fe-Li-mca_{1,2-I} would result in a combination of alternating influxes of F_A and F_C, with repeated periods of closure of the system, allowing the evolution of F_A into F_B. This is consistent with the continuous presence of tectonic activity recorded during the hydrothermal process and the repetitive occurrence of crack-seal phenomena. These early micas share similar compositions and processes with early micas observed in the mineralizing stage of the Maoping deposit (also from the Dayu district) by Legros et al. (2016).

Fe-Li-mca_{3-I}: The late stage I micas, either zinnwaldite, or Li-Fe-ms, display similar trends to the other Piaotang micas (Figure 41b). The same pattern of a double trend converging towards the same mica composition is observed. Fe-Li-mca_{3-I} are therefore likely to result from the same combination of closed system crystallization and mixing as the Fe-Li-mca_{1,2-I} micas.

Fe-Li-mca_{4-III}: The Fe-Li-mca_{4-III} display a single trend between a zinnwalditic **d** pole and a muscovite **e** pole (Figure 41c), consistent with a scheme of fluid mixing between the two end-members. Direct evidence for mixing is found in zoned overgrowths at the microscopic scale, where compositional changes could not be caused by a fractional crystallization (closed system) process (i.e., either Fe and Mg co-vary or Fe decreases when Mg increases) (Figure 40). The consistency between these **d** and **e** poles and the previous **b** and **c** poles will be addressed in the following sections. These post-mineralization micas are similar in composition to micas observed in the post-mineralizing stages of the Maoping deposit (also from the Dayu district) (Legros et al. 2016).

7.2 Conditions of interpretation of trace element compositions

The chemical composition of a given hydrothermal mineral is a record of the fluid composition, generally controlled by the partition coefficients of the incorporated elements between the mineral and the fluid. These are in turn controlled by crystal chemistry constraints and intensive parameters (P, T, pH, fO₂, etc.). Crystal chemistry constraints are clear for major elements, and may also be significant for trace elements. For instance, incorporation of Li (likewise Nb and Ta) in trioctahedral micas depends on Al, due to the Li₊₁(Fe, Mg)₊₁Al₋₁ mechanism of incorporation. In the same way, Sn and W are incorporated by exchanges involving Ti (and also Fe, Mg in the case of W) (Bos, 1990). Generally speaking, crystal chemistry constraints are not well established for most trace elements in micas. Nevertheless, it may be suggested that, at the 1000 ppm level (and below), the ability of sites and the major elements needed for the substitution mechanisms are always sufficient to allow element

incorporation. In this respect, the presence, or not, of a given trace element in a mica, is only governed by the presence of the element in the hydrothermal fluid. The abundance of the element in the mica is however controlled by the fluid-to-mica partition coefficient of the element, and under a given set of intensive parameters, significant differences between the partition coefficients of distinct trace elements might exist.

Experimental data related to mica-fluid partition coefficients are scarce and very limited for hydrothermal conditions, experiments usually being conducted at high temperature (over 500°C). For instance, concerning F entry into micas, Zhu and Sverjensky (1991) conclude that the higher the temperature, the lower the amount of fluorine that partitions into minerals. In contrast, following the experimental results of Munoz and Ludington (1977), the $F/(F+OH)$ ratio of muscovite and phlogopite correlates positively with temperature (but only significantly at $T > 500^{\circ}\text{C}$). According to Munoz and Ludington (1977), when muscovite equilibrates with biotite, it is systematically poorer in fluorine with no dependence upon octahedral occupancy (i.e., the Al content). Nevertheless, both Li and Mg should increase F partitioning in muscovite. Experiments on Rb and Cs partitioning between phlogopite and fluid performed at 800°C and 0.2 to 4 GPa by Melzer and Wunder (2001) show that while Rb partitions in the mineral, Cs partitions into the fluid. Thus, the Rb/Cs ratio in phlogopite is not representative of the fluid composition. However, from experiments on phlogopite at 650°C and 0.2 GPa, Bos (1990) concluded that the Rb, Zn, Pb and Cu contents of the mica are only controlled by their concentrations in the fluid. From these limited data, it may be estimated that the main control on the incorporation of elements into a mica structure, is their abundance in the hydrothermal fluids, and that their ratios in the mineral should therefore be close to their ratios in the fluids. The large compositional differences between the different Piaotang mica generations may thus be safely interpreted in terms of differences in the hydrothermal fluid compositions.

7.3 Trace elements: characterization of fluid end-members

The data obtained from the Fe-Li-mca_{1-I} and Fe-Li-mca_{2-I} micas represent combinations of both the **a-b** and **b-c** trends, depending on the local proportion of zinnwaldite and Fe-Li-ms components at the spot site. Considering the mica structures, it seems that the Fe-Li-ms components are statistically more abundant in the Fe-Li-mca_{1-I} micas, which should consequently yield more information on the **b-c** trend. Conversely, in the Fe-Li-mca_{2-I} structure, the zinnwaldite and Fe-Li-ms components are more equally distributed and are more likely representative of the **b** pole. The data in Figure 41b are quite consistent with these suggestions: the Fe-Li-mca_{1-I} micas lie along trends suggestive of mixing, whereas the Fe-Li-mca_{2-I} data plot in a more homogeneous cluster, likely representing an average composition.

Given the trace element data for these micas, it is possible to simplify our interpretation with a two-fluid model. Indeed, all hydrothermal mica generations (Fe-Li-mca_{1-I}, Fe-Li-mca_{2-I}, Fe-Li-mca_{4-III} and greisen) are characterized by trends in the trace element diagrams (Figure 42). According to major element interpretations, these trends may be considered as reflecting mixing events and therefore may define four pairs of potential end-members (**a** and **b** for Fe-Li-mca_{1-I}, **c** and **d** for Fe-Li-mca_{2-I}, **e** and **f** for Fe-Li-mca_{4-III}, and **g** and **h** for greisen micas) (Figure 42e and f). Owing to the fact that Mn is more abundant in zinnwaldite and other Fe-rich micas, and in reference to the W-Mn diagram (Figure 42c), **a** and **c** correspond to the F_B end-member while **b** and **d**, reflect the F_C end-member. The W-Ba and Sn-Ba diagrams allows us to refine our interpretation of the trace element data. It appears (Figure 42e and f) that two series of end-members (**g**, **a**, **f** and **h**, **d**, **b**, respectively) display well-defined trends, connecting the earliest vein micas to the greisen micas. In contrast, the later Fe-Li-mca_{4-III} micas form their own trend and result from the F_E-F_F mixing process. Following this logic, the greisen micas appear to result from the interaction of two series of fluids associated with the stage I micas (F_B and F_C). These fluids evolve from a metal-poor to a metal-rich composition with a constant fractionation vs mixing model (see section above).

7.4 Relationships with the ore-forming process

Given that both the F_B and F_C fluids carry significant concentrations of metal, both are potentially able to contribute to the ore-forming process. However, micas precipitating from the mixing ore-depositing fluid record a lower saturation value than micas from the pre-mixing stages. Thus, micas from the ore-stage are expected to be depleted in rare metals. This may explain, for instance, why the Fe-Li-mca_{1-I} micas are depleted in W (coeval with wolframite) and the Fe-Li-mca_{2-I} micas are depleted in Sn (coeval with cassiterite). Correlatively, the relative enrichment in Nb in the Fe-Li-mca_{1-I} micas could result from the incompatible character of Nb, which was also not detected at a significant level in wolframite or in cassiterite, whereas other vein micas would be very Nb-poor. Therefore, the Fe-Li-mca_{2-I} micas and the greisen micas have recorded a period where the conditions for ore-deposition were not encountered. Moreover, greisen micas are considered to be the result of the biotite granite transformation, which includes alteration of the initial Nb-Ta-rich biotite into an enriched Fe-Li muscovite in the greisen with inherited rare metals. This interpretation is supported by the constant Nb# in greisen micas, fixed at the average value of the biotite precursors. In their study of the Dajishan deposit, Wu et al. (2017) observed that the muscovite in RMG granites was enriched in Nb, Ta, Rb and Cs relative to the vein-hosted muscovite, whereas both displayed similar Sn and W contents (similar to the Piaotang case). In Wu et al. (2017), muscovites in the granite were interpreted to have formed by the same fluids that formed the veins but modified through reaction with the HFSE-enriched granite in the same way as in the Piaotang deposit. The lower Nb and Ta contents in the greisen micas relative to the granite micas reflect the leaching of these rare metals out from the granite body.

The late Fe-Li-mca_{4-III} micas, which were deposited at the very beginning of stage III, characterized by renewed Sn deposition (abundant Stn_{1-III} and minor Cst_{3-III}), are enriched in Sn and depleted in W, and may thus be considered as having recorded a Sn-rich fluid, which would, in this case, be identified as the F_F end-member (Figure 42b and f).

7.5 Comparison with published fluid inclusion studies

Fluid inclusion (FI) studies were performed at the Piaotang deposit on quartz (Wang et al. 2013b; Ni et al. 2015), cassiterite (Zeng et al. 2002; Wang et al. 2013b) and wolframite (by infrared microscopy; Ni et al. 2015). The corresponding data are summarized in Figure 44. For each mineral presented in Figure 44, the primary FI were plotted. Secondary inclusions were only analysed in quartz by Ni et al. (2015) and are referred to as “post stage I”. The FI display a trend of decreasing temperature from wolframite (400 to 300°C) to cassiterite (350 to 300°C) and finally to post-ore fluids trapped in quartz (300 to 150°C). Fluid mixing is attested to by salinity variations in wolframite (between 4.6 and 8.9 wt. % eq. NaCl) and quartz (0 to 11 wt. % eq. NaCl), whereas cassiterite trapped the most saline fluids (8.5 to 9.5 wt. % eq. NaCl).

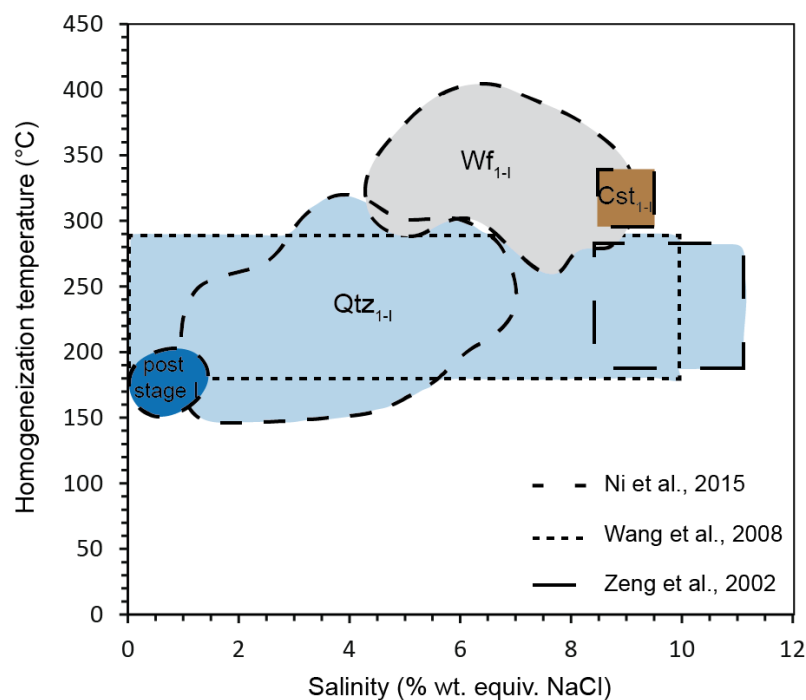


Figure 44. Fluid inclusion data plotted on a binary salinity vs. homogenization temperature diagram after Ni et al., (2015), Wang et al., (2008) and Zeng et al., (2002).

The preceding interpretations of mica compositions are consistent with these results. Moreover, the decrease in temperature revealed by the FI studies would explain why the Fe-Li-mca_{2-I} micas are not associated with wolframite deposition in that the temperature decrease could have inhibited wolframite precipitation.

7.6 Origin of the fluids

Stage I fluids: As discussed in the preceding sections, the F_B fluid, or, more precisely, its F_A parent, are likely to be representative of the ore-forming fluids at stage I. The interpretative model detailed in the previous section implies that each F_B^x should be associated with an F_A^x fluid (Figure 42) involving a renewal of the source. According to the major and trace element composition data, (except for their W (and Sn) content) the F_A fluids are characterized by high contents of F, Fe+Mg (Fe# of 0.75) and Mn. Such characteristics could be interpreted as reflecting a magmatic-hydrothermal origin, involving a rare metal granite (RMG) with a high F content. However, such fluids are unlikely to be Mg-rich as they are associated with evolved granites. Moreover, the enrichment in F should be associated with Al enrichment. In any case, this RMG could not be the Piaotang granite. Interaction of an evolved granite with an external fluid, which (based on the Fe and Mg contents) could be of metamorphic origin, would be more likely. However, no volatile component was observed in the FI studies (Zeng et al. 2002; Wang et al. 2013b, Ni et al. 2015).

The F_C fluid, poorer in F, represents the diluting fluid recorded in the FI studies. Wang et al. (2009) characterize this fluid as a meteoric air-saturated fluid, interpreted to have recorded downward infiltration of meteoric water through faults and fractures. This shallow water could have infiltrated through tectonically-activated regional drains. However, the trace element content of this F_C fluid, notably in terms of rare metals, suggests that this end-member equally interacted with the same granite as the F_A fluids.

Stage III fluids: The stage III F_E and F_F fluid end-members are interpreted in the same terms as the stage I end-members, with F_E being the Sn-rich ore-forming fluid (Figure 42f) and F_F , the external, diluting end-member.

7.7 Geothermometry implications

Fluid inclusion analyses have only been performed on stage I wolframite, quartz and cassiterite, as presented previously (Zeng et al., 2002, Wang et al., 2009, Ni et al., 2015). Chlorite geothermometry allows us to constrain post-mineralization temperatures at the Piaotang deposit. At the end of stage I, a brittle to ductile (based on microscopic observations only) environment is recorded (Figure 37). According to Stöckhert et al. (1999), the temperature of the brittle–plastic transition in quartz is between 310 ± 30 and 350 ± 50 °C. Cassiterite was the last of the three minerals to crystallize (Figure 37) and recorded homogenization temperatures of between 300 and 350°C (Zeng et al., 2002). Moreover, as fluids were circulating, a hydrostatic gradient should be considered in our system. Given this, even if a wide gradient of 30°C/km to 90°C/km is considered, the cassiterite crystallizing fluids should have been at higher temperatures (at least 400°C). Thus, the fluids must have been cooling at the end of stage I, supporting the idea of involvement of a meteoric fluid (Fc).

Stage II and III temperatures have not been constrained in the literature. However, scheelite (Sch_{1-II}) development at the expense of wolframite is usually interpreted as reflecting a temperature decrease (Wood and Samson 2000). Moreover, Chl_{1-II} compositions applied to the Bourdelle and Cathelineau (2015) geothermometer indicate that the Piaotang system cooled to a temperature of close to 200°C during stage II. Moreover, the Chl_{2-III} geothermometer indicates that the hydrothermal fluids were reheated to a temperature of at least 300°C during stage III. The increase in temperature at this stage could explain the crystallization of new generations of cassiterite and wolframite.

8. Implications

A new paragenesis of the Piaotang deposit has been established here, composed of four stages of emplacement, three of which involve W-Sn minerals: (i) the “silicate-oxide” stage, which hosts the main mineralization; (ii) a “calcic” stage with scheelite and columbo-tantalite;

(iii) a “base metal sulphides” stage with wolframite and cassiterite; and finally (iv) a late “sulphide” stage. This detailed paragenesis highlights for the first time the deposition of W-Sn minerals during three successive stages with a complementary Nb-Ta occurrence during the “calcic” stage.

Fe-Li-mica and chlorite are common in W-Sn deposits, which are usually emplaced during multiple magmatic and hydrothermal events that affect granites and other country rocks. In this work we have presented a detailed study of Fe-Li-mica and chlorites that can be correlated with fluid inclusion studies in order to observe diverse magmatic and hydrothermal influences. The correlation between the W-Sn content of Fe-Li-micas and its concordance with the crystallization of wolframite and cassiterite confirms that Li-micas are good tracers of mineralization in this kind of deposit.

Moreover, by correlating the geochemistry of micas and chlorite with the findings of previous fluid inclusion studies, it appears that two mixing end-members (magmatic and meteoric), clearly identified in both approaches, are associated with the deposition of wolframite and cassiterite during the first stage. However, it should be noted that this study also highlights involvement of a third fluid (external and possibly metamorphic) during deposition of the mineralization and that the circulation of fluids during stage I is responsible for the greisenization. More importantly, the now buried Piaotang biotite granite cannot be the source of the mineralizing magmatic fluids and that the source should instead be linked to a more evolved RMG granite that has not yet been identified.

Finally, our study of Fe-Li-mica and chlorite in the post-mineralizing stages has allowed, for the first time, the different processes involved after the “silicate-oxide” stage to be constrained. A new source of heat was induced into the system during stage III, enabling the crystallization of new generations of minerals of economic interest (cassiterite and wolframite). A fluid inclusion study of the post-mineralizing stages could help to identify the fluid at the origin of this heat advection.

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Part 2 – Fluid inclusions

Preamble

This second part aims to use fluid inclusions to complement the study of fluids by the intermediate of lithium-micas described in the first part. Few fluid inclusion studies have been carried out within the Nanling Range. Many approaches as microthermometry, Raman spectrometry, stable isotopes (O, H, C, S) and noble gas isotopes in transparent and opaque minerals have been performed. These studies point out that fluid inclusions observed in the main quartz of the veins does not reflect the fluid inclusions of the main ore mineral which are cassiterite and wolframite. However, interpretations from fluid inclusions observed in transparent and opaque minerals agree on the implication a magmatic fluid exsolved from the peraluminous magma and transporting the metals, mixing with a meteoric fluid infiltrated within the crust. This mixing is believed to be the trigger process leading to ore mineral precipitation. However, the Li-micas studies in part 1 lead on both Maoping and Piaotang deposits suggest the implication of four fluid end-members. This Chapter 4 combines all fluid inclusion data obtained for the Maoping and Piaotang deposits. The aim of this chapter is to demonstrate the complexity of emplacement of these deposits through the implication of multiple successive fluids determined by an integrated study of microthermometry, Raman spectrometry, stable isotopes (O, H) and LA-ICPMS data.

Chapter 4: Multiple fluids involved in granite-related W-Sn deposits from the world-class Jiangxi Province (China)

Article submitted to Chemical Geology, ECROFI special issue

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Abstract

Chinese granite-related W-Sn deposits represent the main resource of W and Sn in the world. These deposits are believed to be the result of various magmatic and hydrothermal processes that are still debated. Although W-Sn deposits worldwide have been widely documented over the years, studies of mineralizing fluids in Chinese deposits need to be complemented by integrated and trace element analysis of fluid inclusions. The Maoping and Piaotang W-Sn deposits (South Jiangxi, China), genetically associated with the emplacement of Jurassic granites, provide new insights into the ore processes in the richest W-Sn province in the world through microthermometry, Raman spectrometry, LA-ICPMS of fluid inclusions

and stable (O and H) isotope geochemistry. Four fluid end-members have been identified. Three of them are involved in the main W-Sn stage: (i) a magmatic fluid (exsolved from a rather primitive magma) associated with the crystallization of gangue minerals and unmixing processes, (ii) a low- δD magmatic fluid (exsolved from a highly differentiated magma) involved in the ore mineral deposition and, (iii) a meteoric fluid mixing with the two latter end-members. The fourth and last end-member was identified in both deposits and is representative of a new hydrothermal pulse, also mixing with the meteoric fluid. This end-member is associated with higher temperatures and salinities, registered in late mineralization stages where other minor ore minerals crystallize. Trace amounts of gases (mainly CO₂) were observed in the two magmatic end-members. This study raises evidences of: (i) a non-coeval crystallization of the gangue and ore minerals, (ii) magma differentiation and fluid mixing as processes involved in ore deposition, (iii) the first evidence of multiple fluids involved at different timing in Chinese W-Sn deposits and, (iv) a common history for both deposits, implying regional processes associated with one of the largest granitic provinces emplaced in the Jurassic-Cretaceous period.

Keywords

W-Sn – deposits - fluid inclusions – Maoping – Piaotang - China

1. Introduction

In granite-related W-Sn deposits, the main resource of W and Sn in the world, magmatic and hydrothermal activity are thought to have promoted the formation of ore minerals in and around peraluminous granitic plutons (Burnham, 1979; Candela, 1997; Audétat et al., 2000; Mao et al., 2007; Xi et al., 2007; Pirajno, 2009; Zhou et al., 2010). However, the nature and the origin of the ore-forming fluids appear to be variable and are still debated (Marignac and Cathelineau, 2009). Most models involve (i) high-salinity magmatic-hydrothermal fluids

exsolved from the granitic magmas (Audétat et al., 2000; Kamenetsky et al., 2004; Thomas et al., 2005) and/or (ii) “external” hydrothermal fluids of deep-seated or surficial origin, with variable volatile and salt content and variably equilibrated with the granites and their metamorphic hosts (Weisbrod 1988; Wilkinson 1990; Blamart 1991; Noronha et al., 1992; Smith et al., 1996; Bebout et al., 1999; Polyá et al., 2000; Burnard and Polyá 2004; Cai et al., 2007).

In the present study, two W-Sn deposits (Maoping and Piaotang) have been targeted as representatives of the World’s richest W and Sn province: the Nanling Range, China (USGS, 2017). The Nanling Range is a metallogenic belt where multiple and diverse economic deposits (W, Sn, Cu, Pb-Zn, etc.) are associated with one of the largest granitic province (100,000 km²), emplaced during Jurassic-Cretaceous times (Zaw et al., 2007).

Several fluid inclusion (FI) studies have been already carried out in W-Sn deposits from the Nanling Range using microthermometry in transparent and opaque minerals, Raman spectrometry, stable isotopes (O, H, S, C) and noble gas isotopes (He, Ar) (Giuliani et al., 1988; Xuexin et al., 1990; Zeng et al., 2002; Xi et al., 2008; Cao et al., 2009; Wang et al., 2009; Feng et al., 2012; Gong et al., 2015; Hu et al., 2012; Wang et al., 2012; Wei et al., 2012; Zhang et al., 2012; Ni et al., 2015; Xiong et al., 2017). All studies point to a mixing between a magmatic fluid exsolved from a peraluminous granitic magma and a meteoric fluid, as responsible for ore minerals precipitation.

However, detailed petrographic and mineralogical studies coupled with Fe-Li-micas geochemistry in the Maoping and Piaotang deposits suggest that at least, three to four types of fluids showing variable degrees of mixing were involved (Legros et al. 2016 and in press). The idea that multiple types of fluids could be involved, in contrast with single-fluid or two-fluids models mentioned above, has motivated for the first time a detailed characterization of the fluids by microthermometry (PVTx properties of FIs), Raman spectrometry (volatile content of the FIs), stable isotope analysis (H and O isotopic composition of FIs and host minerals respectively) and LA-ICPMS (major and trace element composition of FIs). Both ore minerals

(wolframite, cassiterite) and gangue minerals (quartz, topaz, fluorite) were studied in order to investigate the evolution of the fluids from the pre-ore to the post-ore stages.

2. Geological settings, previous fluid inclusion studies and sampling

2.1 The southern Jiangxi metallogenic province (Nanling Range)

The southern Jiangxi province represents 90% of the China's tungsten resources (Zeng et al. 2007) with a total of 429 deposits and 1.7 Mt WO₃. This province is located within the Nanling Range, in the Cathaysia Block from the South China Craton (SCC).

The SCC results from the suture between the Yangtze block to the north and the Cathaysia block to the south, during the late proterozoic Jiangnan (or Sibao) orogen (Figure 45; Charvet 2013 and references therein). The Sibao orogen, dated between 1040 and 900 Ma by U-Pb (zircon) and ⁴⁰Ar/³⁹Ar (muscovite) methods, is the result of the diachronous closure of the ocean between the Cathaysia and Yangtze blocks during the assembly of Rodinia (Li et al. 2007). The boundary between the two blocks, bordering the belt to the SE, is generally considered to be marked by the Jiangshan-Shaoxi fault zone that was evidenced by a Moho's depth drop of several kilometres in this zone (Wang et al., 2013 and references therein).

From the late Neoproterozoic onwards, the South China Craton was strongly reworked and progressively metacratonized through multiple intracontinental episodes, starting with the major aborted Nanhua rifting (Wang et al., 2006). This episode was followed by the double-verging NE-trending Kwangsian orogeny (Ordovician-Devonian), as the result of the Nanhua rift inversion (Charvet et al., 2010 and 2013), and multiple metamorphic events which main peak reached 720 °C and 10.3 kbar at ca. 450 Ma.

Then, the Indosinian (Permian-Triassic) orogeny overprinted the Proterozoic deformations of the Jiangnan belt in the Cathaysia and was marked by extensive granitic

magmatism spanning over 60 Ma (265-205 Ma; Mao et al., 2012 and references therein) and covering 14,300 km² (Sun et al., 2012).

Finally, the Yanshanian orogeny (Jurassic-Cretaceous) generated the most extensive and intense magmatic activity (mainly composed of granites and rhyolites) (Li et al., 2014), associated with three major metallogenic periods: magmatic-related Cu, Pb-Zn and polymetallic mineralization (Jiangxi and southeastern Hunan) between 180 and 170 Ma; rare-metal mineralization related to S-type granitoids (W-Sn, Nb, Ta) from 160 to 139 Ma; and magmatic-related Sn and U and Au-Cu-Pb-Zn-Ag deposits (in the Nanling Range and southeast coast zone respectively; Hua et al., 2005) between 125 and 98 Ma. W-Sn deposits are almost exclusively distributed in the outer contact zone of S-type Jurassic granites as in the W-Sn Maoping and Piaotang deposits and other numerous large W-Sn deposits located in the Dayu district of the southern Jiangxi Province and dated between 160 and 150 Ma like the Xihuashan deposit (Giuliani et al., 1988; Guo et al., 2012; Figure 45).

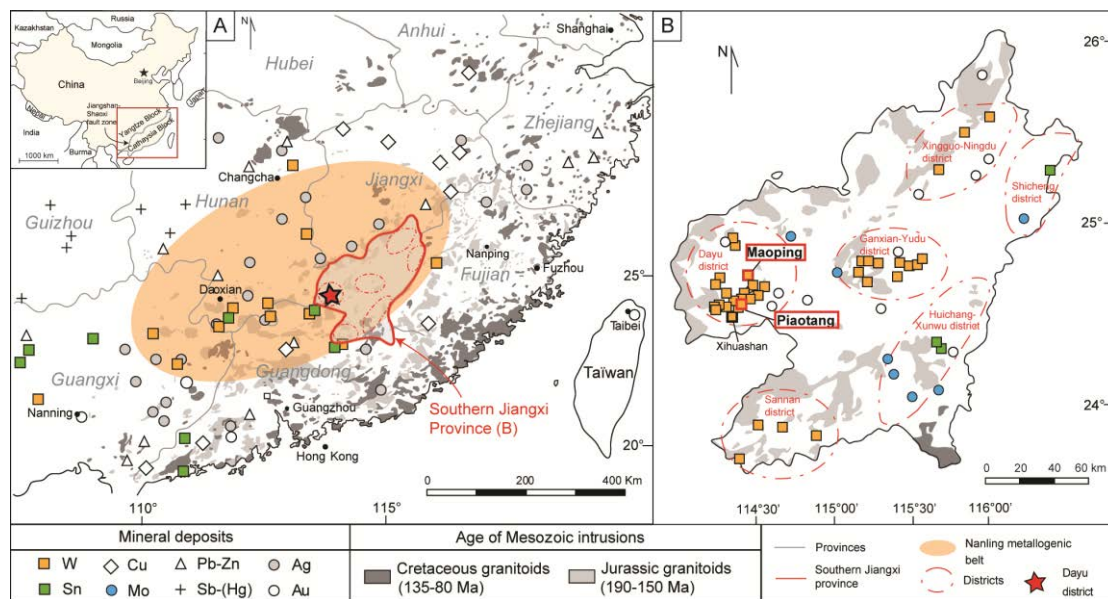


Figure 45. Geological and geographical context of the Maoping and Piaotang deposits (modified after Legros et al., 2016). (A) Location of mineral deposits and Cretaceous and Jurassic granitoids of the Cathaysia Block. Most W-Sn deposits from the Cathaysia Block are located in the Nanling metallogenic belt, here indicated in orange, and associated with Jurassic and Cretaceous granitoids. The southern Jiangxi Province is outlined in red (B) Location of mining districts, associated minerals deposits and Cretaceous and Jurassic granitoids from the southern part of the Southern Jiangxi Province. The Southern Jiangxi Province is the richest part of the Nanling metallogenic belt in term of W-Sn resources. The Maoping and Piaotang deposits are located in the Dayu district in the western part of the Southern Jiangxi Province.

2.2. Previous fluid inclusion studies in Sn-W deposits from the Nanling Range

Several vein-type granite-related Sn-W deposits from the Nanling Range were the subject of FI studies and more precisely within the southern Jiangxi and Hunan provinces: Xihuashan (Wei et al., 2012), Yaogangxian (Hu et al., 2012), Dajishan, Dangping, Piaotang, Pangushan (Ni et al., 2015), Taoxikeng (Wang et al., 2012), Shimenshi (Gong et al., 2015; Wei et al., 2017), Baxiannao (Feng et al., 2012), Shizhuyuan and Yejiwei (Xuexin et al., 1990). FI studies have been performed on various transparent and opaque minerals within the vein systems such as beryl, quartz, wolframite, cassiterite, fluorite, and tourmaline. Fluids are dominantly aqueous with some occurrences of CO₂-rich FIs in quartz. Coexisting vapor-dominated CO₂-rich FIs with liquid-dominated aqueous FIs is commonly observed in quartz and was interpreted as boiling and/or immiscibility (Wei et al., 2012; Giuliani et al., 1988; Ni et al., 2015). Yet, only aqueous inclusions are observed in wolframite or cassiterite, unraveling differences of fluid conditions between ore and gangue minerals (Piaotang, Dangping, Dajishan, Pangushan, Ni et al., 2015; Xihuashan, Wei et al., 2012). Microthermometric analyses show homogenization temperatures between 150 and 300 °C in gangue minerals, significantly lower than in wolframite (250 and 400°C). Similarly, gangue minerals FIs have lower salinity (0.5 to 9.0 wt.% equiv. NaCl) than in wolframite (3.0 to 14.0 wt.% equiv. NaCl) (Xihuashan, Giuliani et al., 1988; Shizhuyuan, Yejiwie, Xuexin et al., 1990; Piaotang, Zeng et al., 2002, Wang et al., 2009; Dajishan, Xi et al., 2008; Yaogangxian, Cao et al., 2009; Baxiannao, Feng et al., 2012; Taoxikeng, Wang et al., 2012; Dangping, Pangushan, Ni et al., 2015; Shimenshi, Wei et al., 2017). Within single deposits, FIs in wolframite usually display a continuum between a high-salinity, high-temperature end-member and a low-salinity, low-temperature end-member, interpreted as a mixing trend. The combination of mixing and cooling processes are considered to be decisive for ore mineral precipitation (Wei et al., 2012; Ni et al., 2015). Estimated PT conditions of FI entrapment in Xihuashan, Dajishan and Taoxikeng deposits, assuming hydrostatic pressure conditions, range between 40 and 130 MPa and 320 to 400 °C (Wei et al., 2012; Xi et al., 2008; Wang et al., 2012). Pressure estimates

define depths of emplacement between 1 and 4 km (Xuexin et al., 1990; Wang et al., 2012). Stable isotope analyses (O-H-S) have been performed on quartz-hosted FIs of the Xihuashan, Taoxikeng, Shimenshi and Baxiannao deposits. $\delta^{18}\text{O}$ values of quartz range from -8.8 to 8.4 ‰, δD of FIs in quartz range from -43 to 108 ‰ and $\delta^{34}\text{S}$ of late sulphides range from -1.6 to 0.1 ‰ (Feng et al., 2012; Liu et al., 2002; Wang et al., 2012; Wei et al., 2012; Gong et al., 2015; Wei et al., 2017). These isotopic compositions of the gangue minerals crystallizing within the ore stage (mainly quartz), are interpreted as reflecting a mixing between a magmatic fluid and a meteoric fluid. Helium and argon isotopes (R/Ra between 0.2 and 0.8 and $^{40}\text{Ar}/^{36}\text{Ar}$ between 350 and 590) compositions of wolframite- and sulfide-hosted inclusions from the Piaotang and Yaogangxian deposits are consistent with these interpretations (Wang et al., 2009; Hu et al., 2012).

Collectively, previous FI studies of vein-type granite-related Sn-W in the Nanling Range invoke either two fluids (magmatic dominant and meteoric minor) mixing or a single fluid (magmatic) cooling, leading to the precipitation of ore minerals.

2.3. Sampling

2.3.1 Maoping deposit

The Maoping deposit is estimated to 63 kt WO_3 and 15 kt Sn with average grades of 0.93% and 0.3% respectively (Feng et al., 2011) and is considered as one of the biggest W-Sn deposits of the Nanling Range.

The Maoping deposit is associated with a buried peraluminous granite (ca. 300 m deep) which contact with the surrounding Cambrian quartzite and schists can only be observed on drill cores. The granite was emplaced during the Jurassic at 151.8 ± 2.9 Ma (SHRIMP U-Pb zircon; Feng et al., 2011). Two types of mineralization, associated with this granite, are described: wolframite-cassiterite-quartz veins and disseminated wolframite in the greisen.

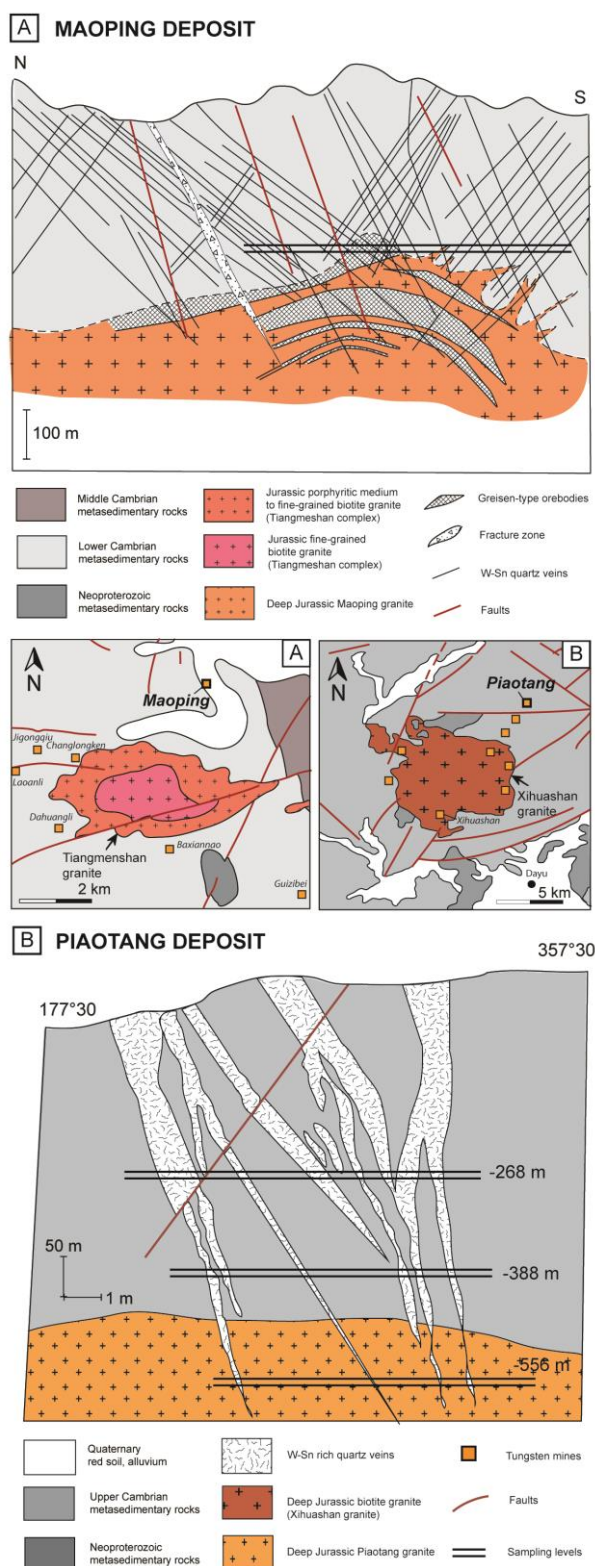


Figure 46. Location and schematic cross-sections and sampling levels of the studied deposits showing the relationships between the deep Jurassic granites, Cambrian host rocks, and W-Sn quartz veins. (A) Maoping deposit (Modified after Feng et al., 2011 and Legros et al., 2016), located near the Tiangmenshan granite. The Maoping granite is thought to be the North-plunging extension of this granite (Feng et al., 2011). (B) Piaotang deposit (Modified after Ni et al., 2015 and Legros et al., 2017), located near the Xihuashan deposit and granite. Similarly to the Maoping deposit, the Piaotang deposit exhibits a greisen cupola on the field that could not be defined properly enough in this work to modify the cross-section of Ni et al., (2015).

The greisen and the veins are respectively dated at 155.3 ± 2.8 and 150.2 ± 2.8 Ma (Re-Os on molybdenite; Feng et al., 2011). A drillcore-based cross-section of the Maoping deposit is presented in Figure 46A, illustrating greisen orebody at the top of the granitic intrusion and a network of radial, sub-vertical ore-bearing veins, rooted on top of the granite and mostly developed in the Cambrian host rocks (Feng et al., 2011). More than 400 W-Sn-bearing quartz-dominated veins from 5 cm to few meters thick and up to 400 m long have been identified (Feng et al., 2011). It is noteworthy that even though only one level in the mine galleries (-5 level, c.a. 200 m deep) could be observed in the present study, all veins appear as shallow-dipping in the mine gallery, contrary to the cross-section proposed by Feng et al. (2011).

A detailed petrographic study was carried out and a paragenetic succession was established by Legros et al. (2016) and summarized here. Four types of veins and seven successive paragenetic stages (I to VII) have been identified according to cross-cutting relationships in the mine galleries and petrographic observations (Figure 47). Hereafter, minerals refer to (i) their recommended IMA mineral abbreviations, (ii) their generation number (1 to 4, from the oldest to the youngest), and (iii) the paragenetic stage to which they belong (I to III, from the oldest to the youngest). For example, Wf_{2-III} stands for the second generation of wolframite in the whole paragenetic sequence, occurring during stage III.

The first stage (I) consists of centimetric microcline, quartz and Fe-Li-micas, forming the earliest veins. The size of quartz crystals did not allow FI analysis in this vein type. The second stage (II) consists of Fe-Li-micas-only veins. Stages III to VI correspond to the W-Sn and banded quartz veins and involve the successive crystallization of various minerals suitable for FI study as described below.

Stage III corresponds to the formation of the W-Sn ore-bearing veins (Figure 48A). The stage III minerals consist of quartz (Qtz_{2,3-III}), wolframite (Wf_{1-III}), cassiterite (Cst_{1-III}), topaz (Toz_{2-III}), Fe-Li-micas (Fe-Li-mca_{3-III}), molybdenite (Mlb_{1-III}), Fe-Li-muscovite (Li-Fe-ms_{1-III}) and Fe-Mn hydroxides. Evidence for plastic deformation at this stage allows distinguishing two successive mineral assemblages (IIIa and IIIb). During stage IIIa, quartz (Qtz_{2,3-III}), topaz (Toz₂₋

III), wolframite (Wf_{1-III}) and cassiterite (Cst_{1-III}) are successively deposited. These minerals were selected for the FI study. Quartz and topaz are abundant in the vein and form centimetric euhedral geodic crystals with no apparent zonation (Figure 48B). Then, wolframite and cassiterite crystallize as euhedral and well-zoned crystals (no chemical variation has been associated with that zonation) (Figure 48C and Figure 48D). The plastic deformation is particularly observed in quartz and topaz minerals where a rolling extinction and subgrains are observed. Stage IIIb displays undeformed Li-Fe-ms_{1-III} and Fe-Mn hydroxides.

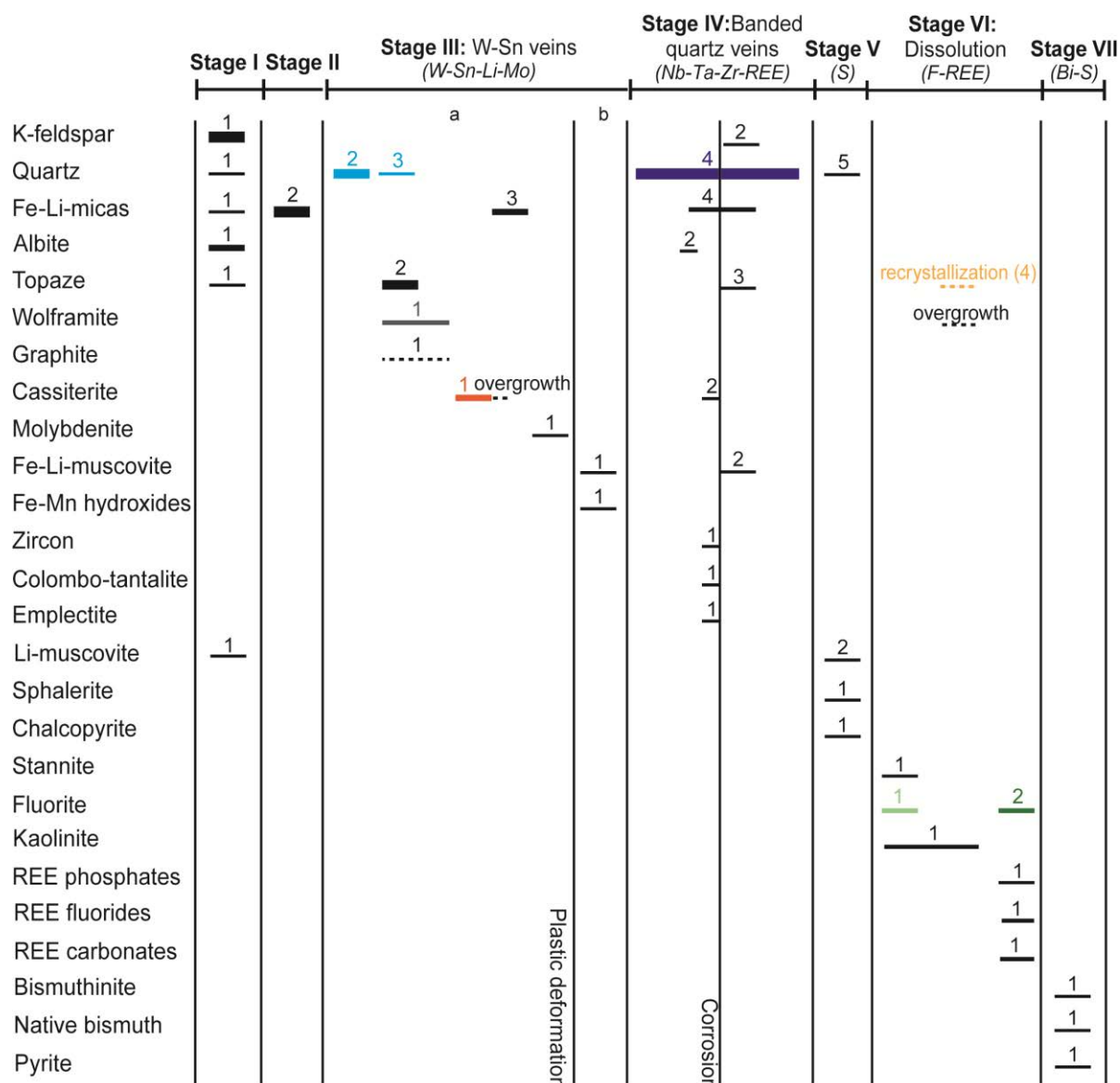


Figure 47. Paragenetic sequence of the Maoping W-Sn deposit based on samples from the mine gallery at level -5 (i.e. -200 m), only level drawn on the previous cross-section. Details about different stages and petrographic descriptions are provided in Legros et al. (2016). Minerals indicated in color (here, quartz, wolframite, cassiterite, fluorite and topaz) have been chosen for this fluid inclusion study. Colors are in accordance with Fig. 8 to 12.

Stage IV corresponds to the emplacement of the banded quartz veins (Figure 48E). Four different types of undeformed quartz are distinguished. The banding is formed by a repeated sequence consisting of (1) quartz corrosion; (2) accumulation of minerals (feldspars, micas and quartz) interpreted as relics of previous stages together with precipitation of disseminated Nb-Ta and REE minerals and Qtz_{4a-IV}; (3) deposition of Qtz_{4b-IV} and (4) deposition of laminated Qtz_{4c-IV}. A final generation of subhedral Qtz_{4d-IV} overprints the quartz banding (Figure 48F).

Stage V consists of very small quartz grains, Li-muscovite and sulfides and therefore could not provide any mineral of interest for this FI study.

Stage VI consists of minerals filling the cavities of the W-Sn and banded quartz veins by of a succession of fluorite (Fl_{1-VI}), kaolinite (Kln_{1-VI}) and late porous fluorite (Fl_{2-VI}) (Figure 48G). Dissolution cavities within Toz_{2-III} are marked by a rim of euhedral and zoned Toz_{4-VI} crystals (more than 100 µm in size) when filled by Kln_{1-VI} (Figure 48H). This generation of topaz was selected for the FI study. The first generation of Fl_{1-VI} is coeval with the kaolinite, euhedral and mineral inclusion-free while the second (Fl_{2-VI}), which postdates the kaolinite, is anhedral and zoned according to the yttrium content, kaolinite inclusions, REE mineral inclusions (phosphates, fluorides and carbonates) and porosity. Both types of fluorite were selected for the FI study.

Stages VII display sulfides (ie. bismuthinite and pyrite) that were not considered for the FI study.

2.3.2 Piaotang deposit

The polymetallic W-Sn-(Nb) Piaotang deposit is one of the biggest W-Sn deposits that belongs to the Nanling Range. The Piaotang mine produces 1,500 t WO₃ per year and ranks at the 8th position of China's reserves with 47,775 t WO₃ (MB Company database: www.metalbulletin.com).

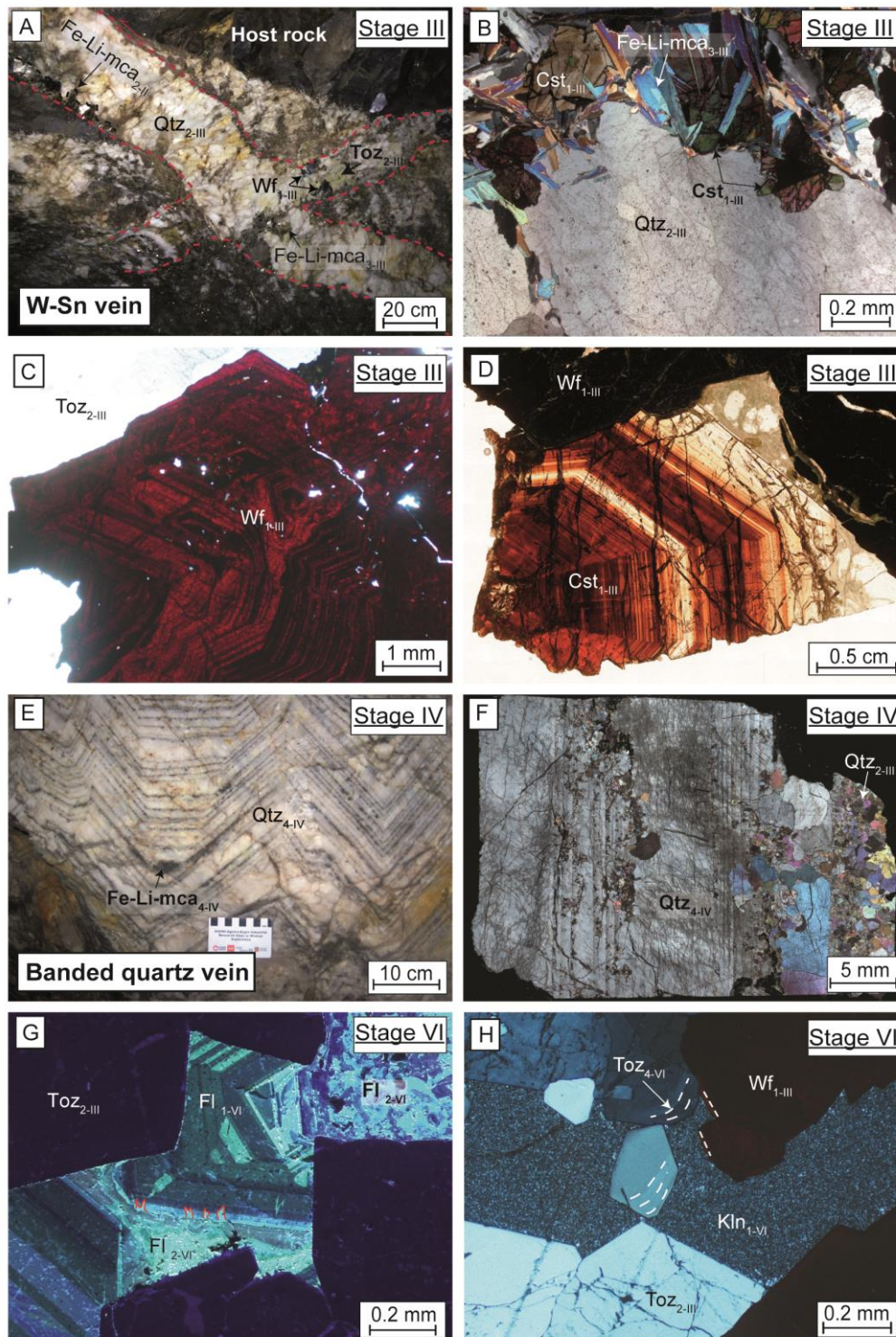


Figure 48. Textural characterization of the minerals selected for FI study according to their respective stages for the Maoping deposit as described by Legros et al. (2016). (A) W-Sn vein (Stage III). (B) Association of Qtz_{2-III} with Cst_{1-III} and $Fe-Li-mca_{3-III}$ (Stage III) (cross polars). (C) Complex growth-zones in the Mn-rich wolframite Wf_{1-III} (Stage III) (natural light). (D) Complex growth zones in the cassiterite Cst_{1-III} of stage III (natural light). (E) Banded quartz vein (Stage IV) showing the centimeter-scale banding of quartz and Fe-Li-micas. (F) Stage IV banded quartz (thick section observed under crossed polars). (G) Two generations of fluorite (FI_{1-VI} and FI_{2-VI}) from Stage VI (CL). (H) Overgrowth and growth-zone textures in Toz_{4-VI} and Wf_{1-III} in contact with kaolinite (Kln_{1-VI}) (crossed polars). Abbreviations according to the IMA: cst = cassiterite, fl = fluorite, kln = kaolinite, mca = mica, qtz = quartz, toz = topaz, wf = wolframite.

The Piaotang deposit is associated with a buried biotite granite (ca. 400 m deep) which contact with the surrounding metasedimentary rocks can only be observed on drill cores. The granite was emplaced during the Jurassic (159.8 ± 0.3 Ma, U-Pb dating on zircon; Zhang et al., 2017). Two types of mineralization are described: wolframite-cassiterite-quartz veins and disseminated wolframite in the greisen. The greisen has not been dated yet and the veins are dated at 159.5 ± 1.5 Ma (U-Pb dating on cassiterite; Zhang et al., 2017). A drillcore-based cross-section of the Piaotang deposit has been established showing steeply-dipping sub-parallel veins rooted in the buried granitic intrusion and hosted in the metasedimentary rocks (Ni et al., 2015). While the greisen cupola has not been drawn on the original cross-section of Ni et al. (2015), the extent of the greisen and its relationship with the granite could not be documented precisely in the present work to be implemented into Figure 46B.

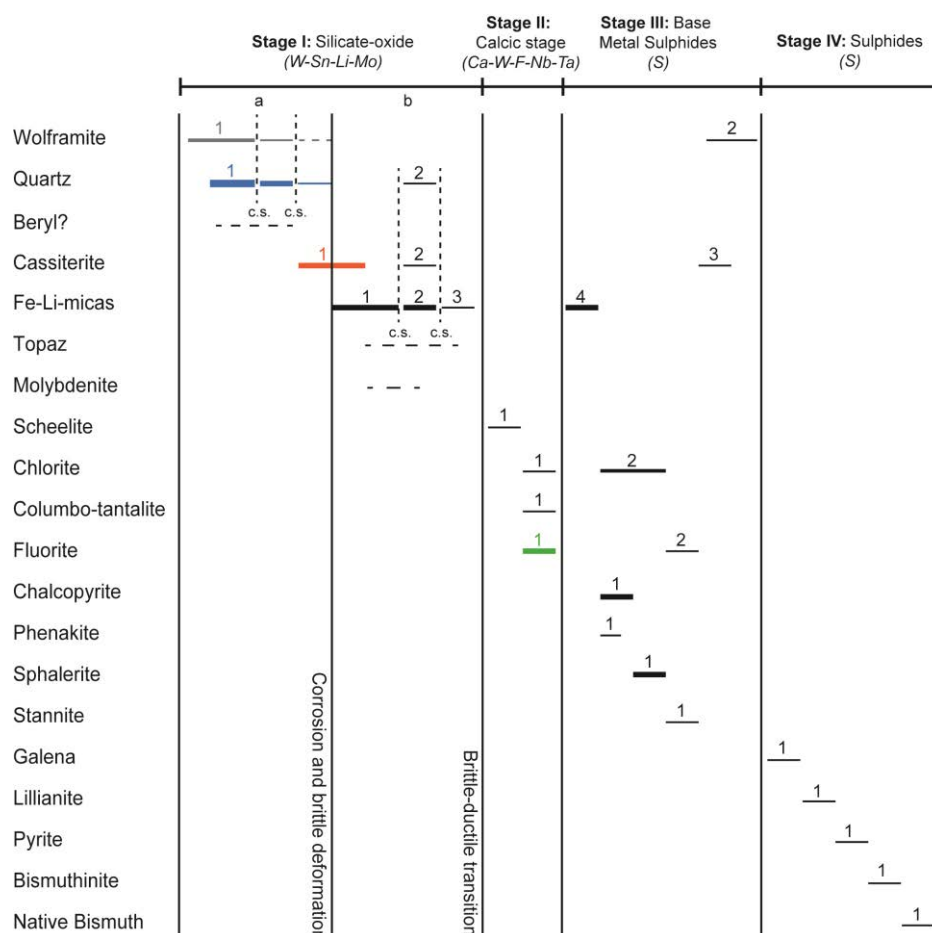


Figure 49. Paragenetic sequence of the Piaotang W-Sn deposit based on observations on samples from in mine galleries at three levels (-268, -388 and -556 m). Details about different stages and petrographic descriptions are provided in Legros et al. (2017). Minerals in color (wolframite, quartz, cassiterite and fluorite) have been chosen for this FI study. Colors are in accordance with Fig. 8 to 12.

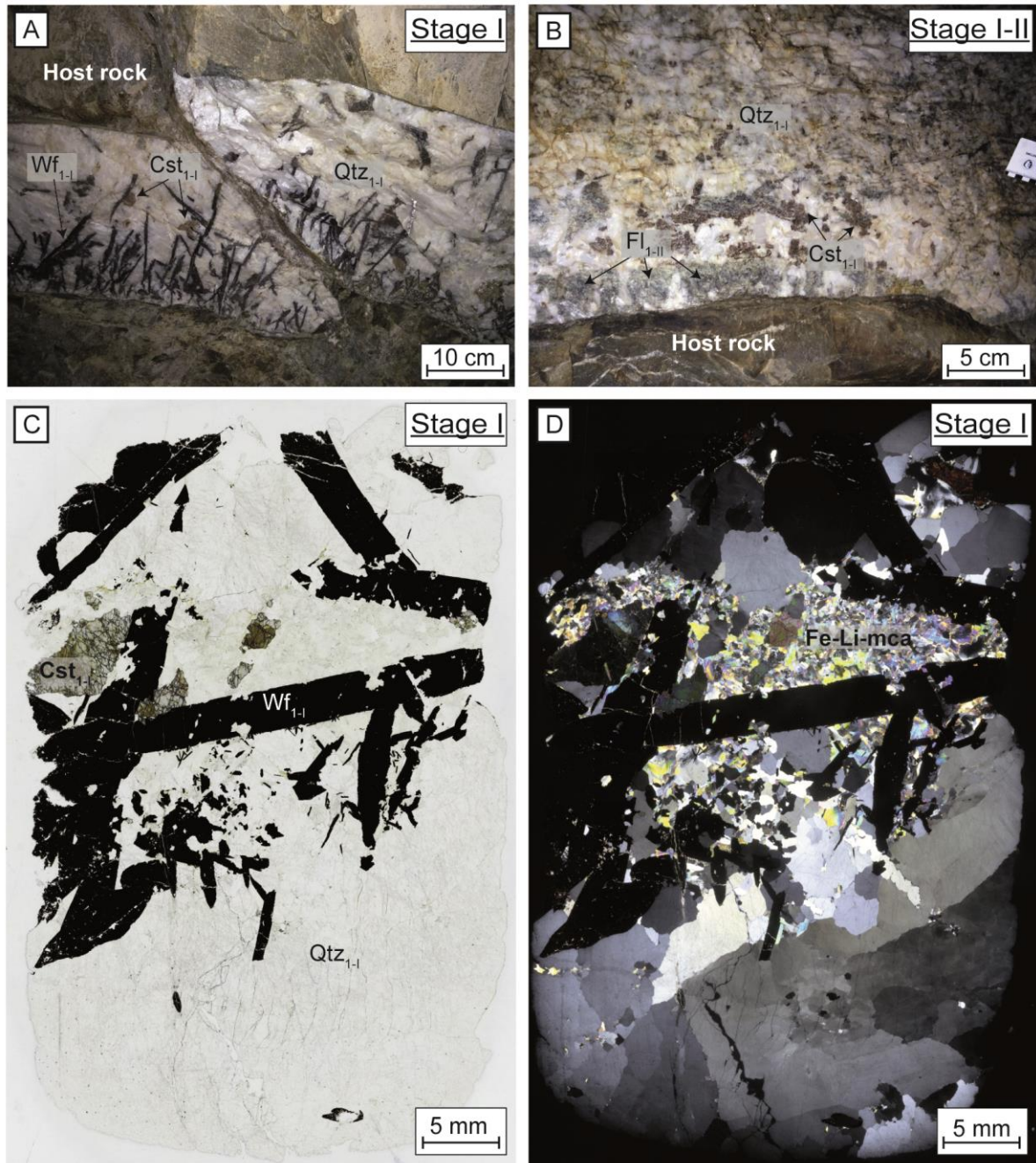


Figure 50. Textural characterization of the minerals selected for FI study according to their respective stages as described by Legros et al. (2017) for the Maoping deposit. (A) Wolframite-cassiterite-quartz vein from Stage I. (B) Fluorite-cassiterite-quartz vein, where fluorite is late and belongs to Stage II. (C) Whole thin-section observed in natural light. (D) Same whole thin-section as in (C) observed with crossed polars. The main minerals crystallizing on stage I (Cst_{1-I} , Wf_{1-I} , Qtz_{1-I} and Fe-Li-mca) show complex relationships. Abbreviations according to the IMA: cst = cassiterite, fl = fluorite, mca = mica, qtz = quartz, wf = wolframite.

One type of vein with heterogeneous infilling has been identified in the mine galleries of the Piaotang deposit by Legros et al. (2017). Their thickness increases from the top to the bottom of the vein system, from a few centimetres at the 556 level, to more than 1 meter at the

268 level. Based on petrographic observations, Legros et al. (in press) distinguished four paragenetic stages (I to IV) at the Piaotang deposit, as summarized below (Figure 49).

Stage I corresponds to the emplacement of the ore-minerals (Figure 50A and Figure 50B). Stage I consists of wolframite (Wf_{1-I}), quartz (Qtz_{1-I}), cassiterite (Cst_{1-I}), Fe-Li-micas (Fe-Li-mca_{1-I} to _{3-I}), topaz (Toz_{1-I}) and molybdenite (Mlb_{1-I}). Quartz is the most abundant mineral in the vein and displays centimetric euhedral crystals with no chemical zonation like those observed in cassiterite (Figure 50C and Figure 50D). All three minerals were selected for the FI study. Topaz is almost completely altered into clays and thus could not permit observing FIs.

Stage II corresponds to the replacement of Wf_{1-I} by scheelite (Sch_{1-II}) and the crystallization of chlorite (Chl_{1-II}), columbo-tantalite (Clb_{1-II}) and fluorite (Fl_{1-II}). Fluorite displays euhedral centimetric crystals with no zonation, mineral inclusions nor deformation and was selected for the FI study (Figure 50B).

Stages III and IV are characterized by important sulfide precipitation together with new generations of wolframite, cassiterite, Fe-Li-micas, chlorite and fluorite. All minerals of Stages III and IV were too small to permit a FI study.

3. Analytical methods

3.1 Fluid inclusion petrography and microthermometry

Doubly polished thick sections (110 to 200 μm -thick) from quartz, cassiterite, wolframite, fluorite and topaz samples were prepared at GeoRessources laboratory (Nancy, France). Petrographic observations were carried out on transparent minerals (quartz, fluorite and topaz) minerals using a Olympus BX-51 optical microscope at GeoRessources laboratory (Nancy, France) and in opaque and semi-opaque minerals (wolframite, cassiterite) using an Olympus BH-51 microscope, equipped with an Olympus XM-10 infrared camera at the University of Geneva (Switzerland).

FI microthermometry was performed on quartz-, fluorite- and topaz-hosted FIs using a Linkam THMSG600 heating-cooling stage mounted on a Olympus BX-51 microscope at GeoRessources laboratory (Nancy, France). The stage was calibrated with in-house and certified standards by measuring the temperature of the final melting of pure water (0.0 °C) in a silica glass capillary, the triple point of CO₂ ± Ar in a synthetic FI (−56.9 °C), and of the liquid + vapor → liquid homogenization of a natural FI at 165 °C. The temperatures of phase changes of aqueous FIs were measured for the following: final ice melting ($T_{m(ice)}$) and liquid + vapor → liquid total homogenization (T_h). Temperatures of final clathrate melting ($T_{m(cla)}$) and liquid + vapor → vapor homogenization of the CO₂-dominated carbonic phase ($T_{h(CO_2)}$) was also measured in the case of aquo-carbonic FIs. The temperature of phase changes is reported with an accuracy of about ±0.1 °C for $T_{m(ice)}$ and $T_{m(cla)}$ and ±1 °C for T_h and $T_{h(CO_2)}$.

FI microthermometry was carried out on wolframite at the University of Geneva (Switzerland) with a Linkam FTIR 600 stage mounted on an Olympus BH-51 microscope, equipped with an Olympus XM-10 infrared camera (Ortelli et al., submitted). An oriented section of a single prismatic crystal, cut perpendicular to the cleavage, was used. It has been demonstrated that the visible light absorbed by opaque minerals is partially converted into heat (the more opaque the zone is, the more the absorption is important), thus potentially precluding accurate microthermometric measurements (Moritz, 2006). In order to avoid this problem, a voltmeter has been connected directly to the lamp of the microscope to control the intensity of the incident light. It appears that sample heating is not sufficient to significantly affect microthermometric measurements in the conditions of a 90% closed diaphragm and condenser at 0.3, and a tension between 0 and 4 V according to the transparency of the different zones of the crystals (Casanova et al., submitted).

As cassiterite is a semi-opaque mineral, the same protocol was applied in transparent light at GeoRessources Laboratory (Nancy, France). Sample heating due to the light absorption was minor compared to wolframite. Analyses were performed with a fully opened

diaphragm and condenser and within a range of 0 to 6 V according to the transparency of the different zones in the crystals.

The reported salinity data of CO₂-rich FIs were calculated with the equation of state of Duan et al., (1992) using the Q2 programs from Bakker (1997, 2003). First ice melting temperature (i.e. eutectic temperature) could not be observed precisely, but the large range of likely transition (-25 to -15 °C) are close to the eutectic temperatures of the H₂O-NaCl and H₂O-KCl-NaCl systems (-21.2°C and -23.5°C respectively). The presence of potassium was quantified by LA-ICPMS in almost all FIs. However, no hydrohalite could be observed by microthermometry. Therefore, reported salinity of aqueous FIs was calculated in the H₂O-NaCl system with the Archer (1992) equation of state using Bulk program from Bakker (1997, 2003). As relative proportions of Na and K calculated by LA-ICPMS mainly vary from 90% Na - 10% K to 50% Na – 50% K and final ice melting temperatures vary from -5.5 °C to 0.0°C, the maximum error that could be induced by salinity calculations in the H₂O-NaCl system is estimated to 1 wt.% equiv. NaCl lower than reality, for the more elevated values. Isochores were calculated for aqueous and aquo-carbonic FIs with the ISOC program (Bakker, 1993) using the empirical equations of state of Bodnar and Vityk (1994) and, Anderko and Pitzer (1993a,b) and Duan et al., (1995) respectively.

3.2 Raman spectrometry

Raman spectrometry on aqueous (one-phase inclusions) and gas phases (two and three-phase inclusions) from FIs was performed at GeoRessources laboratory (Nancy, France) in order to detect possible traces of gases and their nature at room temperature. Raman spectra were recorded using a LabRAM spectrometer (Horiba Jobin Yvon) equipped with a 600 gr.mm⁻¹ grating and an edge filter. The confocal hole aperture is of 500 µm and the slit aperture is of 100 µm. The excitation beam is provided by a Stabilite 2017 Ar⁺ laser (Spectra Physics, Newport Corporation) at 514.53 nm and a power of 200 mW, focused on the sample using a ×100 objective (Olympus). Acquisition time and number of accumulation are chosen to optimize the signal-to-noise ratio (S/N), ideally lower than 1 %. Neither limits of detection

nor absolute concentrations of trace gases, can be determined. However the relative proportions of the different gas species identified with LabSpec software at room temperature can be estimated qualitatively using the peak area, acquisition time and the specific scattering cross-section of each peak (Schrotter and Klockner, 1979) as defined by Burke (2001).

3.3 LA-ICPMS analysis

Selected major, minor and trace elements were determined in FIs using a LA-ICPMS instrumental setup at GeoRessources laboratory (Nancy, France) which is similar to that described in Leisen et al. (2012) and Lach et al. (2013). It comprises a GeoLas excimer laser (ArF, 193 nm, Microlas, Göttingen, Germany) (Günther et al., 1997) and an Agilent 7500c quadrupole ICP-MS. The laser beam is focused onto the sample within a small-volume lozenge shaped ablation cell dedicated to FIs analysis with a Schwarzschild reflective objective (magnification $\times 25$; numerical aperture 0.4) mounted on an optical microscope (Olympus BX41) equipped with a X-Y motorized stage and a CCD camera. NIST610 glass (see composition in Jochum et al., 2011) was chosen as the external standard (analytical accuracy verified with NIST612 external standard) (Longerich et al., 1990). Used parameters are a fluence of 8 J/cm^2 and laser shot frequency of 5 Hz, $\text{He} = 0.5 \text{ L.min}^{-1}$ as a carrier gas mixed with $\text{Ar} = 0.7 \text{ L.min}^{-1}$ via a cyclone mixer prior to entering the ICP torch, spot sizes of $44 \mu\text{m}$ and ablation duration of 40 s. The recorded isotopes are: ^7Li , ^{23}Na , ^{24}Mg , ^{39}K , ^{43}Ca , ^{55}Mn , ^{85}Rb , ^{88}Sr , ^{89}Y , ^{93}Nb , ^{95}Mo , ^{118}Sn , ^{133}Cs , ^{138}Ba , ^{181}Ta and ^{182}W with an integration time of 0.01 s per mass channel. These elements have been chosen according to the paragenesis as they are suspected to be present in the fluids according to the compositions of the successive minerals in the paragenetic successions and/or because they are usually enriched in magmatic-hydrothermal fluids in this context (e.g. Na, K, Cs, Rb). Calibration and signal-integration were performed with the Matlab®-based SILLS program (Guillong et al., 2008). Absolute element concentrations were calculated from analysed ratios relative to Na. The Na concentration was obtained from the salinity estimated from microthermometry analysis (in wt.% NaCl equiv.) using the charge-balance technique (Allan et al., 2005). This method corrects the modelled

amount of Na (from the wt.% NaCl equiv.) for contributions from other chloride salts using the analysed elemental ratios to Na. A matrix correction was applied for cassiterite and fluorite-hosted inclusions using the SILLIS program and using Sn and Ca respectively as internal standards. Limits of detection (LODs) were calculated using the 3σ criterion (Longerich et al., 1996). The analytical precision for most elements is within 15% relative standard deviation (RSD). The analytical precision of the other elements is typically better than 30% RSD (Allan et al., 2005).

3.4 Isotopic measurements

3.4.1 Oxygen isotopes of minerals and fluids

Oxygen isotope analyses on quartz, Fe-Li-micas, quartz, topaz and wolframite were carried out at the Stable Isotope Laboratory of the University of Lausanne, Switzerland, using a CO₂-laser fluorination line coupled to a Finnigan MAT 253 mass spectrometer. Pure and clean mineral separates were picked under a binocular microscope and crushed in an agate mortar. For each acquisition, between 1.16 and 3.15 mg of sample was loaded with LS-1 reference material. The chamber was evacuated overnight under a vacuum better than 10^{-4} mbar before fluorination. Samples were heated with a CO₂-laser in presence of F₂ and the liberated oxygen was purified on an overheated KCl salt in the extraction line. Oxygen was absorbed on a molecular sieve (13x) held at liquid nitrogen temperature and then heated to liberate the O₂ into the mass spectrometer. For each acquisition, results are reported in per mil (‰) relative to VSMOW (Vienna Standard Mean Ocean Water) and are normalized to the quartz standard LS-1 (reference value: $\delta^{18}\text{O} = 18.1$ ‰ vs. VSMOW). The precision, based on replicate analyses of the samples (when possible) and the standard run together with the samples, was generally better than 0.2 ‰. Oxygen isotope compositions of fluids were calculated at selected temperatures using equilibrium equations of Zheng (1993a) for Fe-Li-micas, Zheng (1993b) for quartz, Zheng (1991) for wolframite and Zheng (1993a) for topaz. Cassiterite crystals did not permit oxygen isotope analysis due to complex zoning.

3.4.2 Hydrogen isotopes of fluid inclusion

Hydrogen isotope composition of FIs were measured at CRPG laboratory (Nancy, France). Between 2 and 4 g of clean and pure separates of quartz, wolframite and cassiterite were separated and introduced into steel tubes. In order to release H₂O molecules adsorbed at the mineral surface, the tubes were connected to a vacuum extraction line and heated overnight at 120 °C (Dublyansky and Spötl, 2009). Before any extraction, microthermometric tests have been conducted to make sure that FIs would neither decrepitate nor stretch below temperatures of 200 °C. The tubes were crushed under vacuum to release all fluids hosted in crystals. Incondensable gases have been directly removed from the extraction line so that H₂O (and possibly CO₂) only, were trapped under vacuum in a liquid nitrogen-cooled U-shaped tube. CO₂ was never found in sufficient amount to be analyzed, and was thus removed from the line. H₂O was reduced into H₂ by passing through a uranium reactor heated at 800 °C (Bigeleisen et al., 1952) and then trapped into glass tubes before being analyzed using a VG 602D mass spectrometer. To insure the accuracy of isotopic measurements, three in-house standards were also extracted and analyzed. Values of δD for H₂ and thus for FIs H₂O are reported in standard per mil (‰) notation relative to VSMOW.

The metal reduction method has been chosen for the preparation of gas sample for hydrogen isotope ratio analysis because of the small size of sample (< 1 μ l) needed (Wong et al., 1984). Because water adheres to the internal surfaces of the uranium reactor, D/H isotope analysis may show a memory effect. The magnitude of this memory effect is dependent on the difference between the isotopic compositions and amount of water reduced for two successive samples (Blaise et al., 2015). Therefore, δD values from the first extraction of a given sample were thus not considered, as potentially affected by this memory effect. δD values represent average of duplicates.

4. Results

4.1 Fluid inclusion petrography

For both Maoping and Piaotang deposits, fluid inclusions (FIs) have been observed in quartz (Figure 51A, B, C, F, J and K), wolframite (Figure 51E and L), cassiterite (Figure 51D and M) and fluorite (Figure 51G, I and N) with the exception of topaz which was only observed in Maoping (Figure 51H). FI petrographic observations are summarized in Table 6. Within the quartz samples, the petrography of FIs is hampered by their high abundance which confers a milky aspect to the crystals. Other minerals contain less abundant FIs and are therefore more translucent, favoring petrographic characterization of FIs. According to the criteria of Roedder (1984), the selected FIs are either pseudosecondary (i.e. distributed along intragranular FI planes (FIPs)) or presumably primary (isolated or randomly distributed or clustered or aligned and/or elongated along growth zones). In all selected samples, there is no optical or compositional difference between pseudosecondary and presumably primary FIs and the two types will not be further distinguished. Here, the application of the FI assemblage (FIA) concept (Goldstein and Reynolds, 1994) is limited either by the milky aspect of the quartz and/or the relatively small number of FIs above 5 μm in size (and therefore, not suitable for microthermometric study in a given FIA). The majority of analyzed FIs show two phases (liquid + vapor) at room temperature and typically range from 5 to 40 μm in size. FIs display rounded, elongated, irregular or negative crystal shapes regardless of their location. Optical estimation of the volumetric fraction of the vapor phase at room temperature (ϕ_{vap}), as deduced from area fractions (α), ranges from 10 to 50 %. α values are always randomly distributed in single crystals and possible FIAs. However, as ϕ_{vap} , as deduced from α values, are subjected to apparent variation due to two-dimensional projection of the FIs under the petrographic microscope, apparent variation of ϕ_{vap} should be considered with caution (Bakker and Diamond, 2006). Accordingly, there is no evidence for heterogeneous trapping of the fluid within a same type of inclusions (two-phase or three-phase).

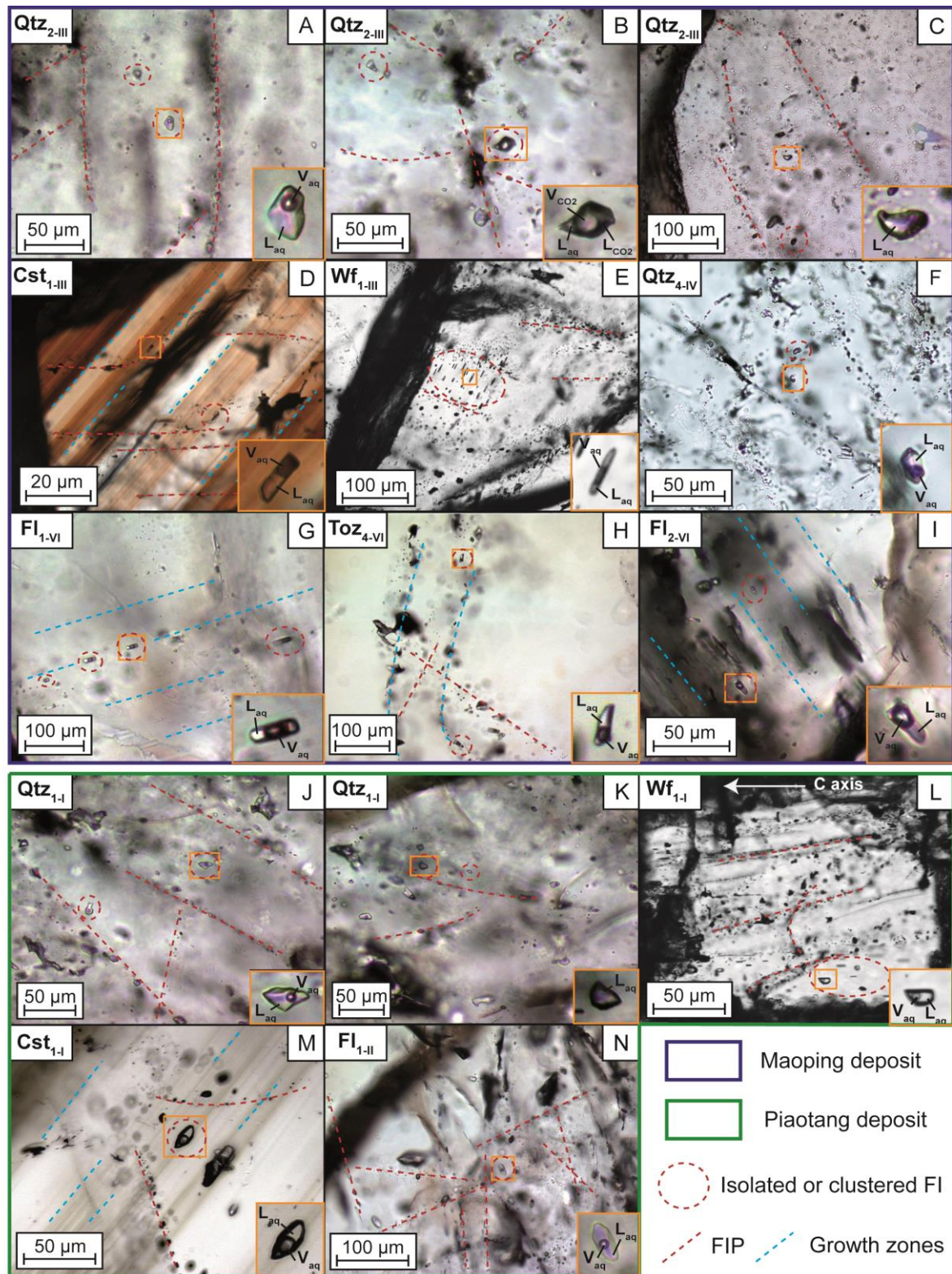


Figure 51. Petrography and phase filling of FIs studied at the Maoping and Piaotang deposits at room temperature. FIs from Maoping are shown in (A) to (I). FIs from Piaotang are shown in (J) to (N). (A) Two-phase liquid (L_{aq}) + vapor (V_{aq}) aqueous FI as isolated irregular inclusions and intragranular FIPs (dotted red lines) in Qtz_{2-III} . Detailed view on the isolated FI is shown in the inset. (B) Intragranular FIPs (dotted red lines) of two-phase liquid (L_{aq}) + vapor (V_{aq}) aqueous FI and isolated irregular three-phase liquid H_2O (L_{aq}) + liquid CO_2 (L_{CO_2}) + vapor CO_2 (V_{CO_2}) aquo-carbonic FI in Qtz_{2-III} . Detailed view on the isolated FI is shown in the inset. (C) Intragranular FIPs of two-phase liquid (L_{aq}) + vapor (V_{aq}) aqueous FI and isolated irregular one-phase liquid (L_{aq}) aqueous FI in Qtz_{2-III} . Detailed view on the isolated FI is

shown in the inset. (D) Two-phase liquid (L_{aq}) + vapor (V_{aq}) aqueous FIs as isolated regular inclusion parallel to growth zones and intragranular FIPs oblique to the growth zones in Cst_{1-III} . Detailed view on the isolated FI is shown in the inset. (E) Two-phase liquid (L_{aq}) + vapor (V_{aq}) aqueous FI as isolated irregular inclusions and intragranular FIP in Wf_{1-III} . Detailed view on an isolated FI is shown in the inset. (F) Two-phase liquid (L_{aq}) + vapor (V_{aq}) aqueous FI as isolated negative crystal-shape inclusions and intragranular FIP in Qtz_{4-IV} . Detailed view on the isolated FI is shown in the inset. (G) Two-phase liquid (L_{aq}) + vapor (V_{aq}) aqueous FI as isolated regular inclusions and intragranular FIP displayed parallel to the growth zone in Fl_{1-VI} . Detailed view on the FI is shown in the inset. (H) Two-phase liquid (L_{aq}) + vapor (V_{aq}) aqueous FI as isolated regular inclusions and intragranular FIP displayed along a Toz_{4-VI} growth zones. Detailed view on the FI is shown in the inset. (I) Two-phase liquid (L_{aq}) + vapor (V_{aq}) aqueous FI as isolated regular inclusions and intragranular FIP displayed along the Fl_{2-VI} growth zones. Detailed view on the FI is shown in the inset. (J) Two-phase liquid (L_{aq}) + vapor (V_{aq}) aqueous FI as isolated irregular inclusions and intragranular FIP in Qtz_{1-I} . Detailed view on the FI is shown in the inset. (K) One-phase liquid (L_{aq}) aqueous FI as isolated irregular inclusions and intragranular FIP in Qtz_{1-I} . (L) Two-phase liquid (L_{aq}) + vapor (V_{aq}) aqueous FI as isolated regular inclusions displayed along the Wf_{1-I} c-axis and in secondary FIP crossing the Wf_{1-I} c-axis. Detailed view on the FI is shown in the inset. (M) Two-phase liquid (L_{aq}) + vapor (V_{aq}) aqueous FI as isolated regular inclusions displayed along a Cst_{1-I} growth zones and secondary FIP crossing the Cst_{1-I} growth zones. Detailed view on the FI is shown in the inset. (N) Two-phase liquid (L_{aq}) + vapor (V_{aq}) aqueous FI as isolated regular inclusions and intragranular FIP in Fl_{1-II} . Detailed view on the FI is shown in the inset. Abbreviations according to the IMA: *cst* = cassiterite, *fl* = fluorite, *qtz* = quartz, *toz* = topaz, *wf* = wolframite.

Some rare (< 10%) one-phase (liquid) FIs have been randomly observed in Qtz_{2-III} and Qtz_{1-I} from both Maoping and Piaotang deposits. One-phase FIs are usually isolated or randomly displayed in FIA, associated with two-phase FIs. In the same way, very rare (< 5%) three-phase (liquid H_2O + liquid CO_2 + vapor CO_2) inclusions have been observed in the Qtz_{2-III} of the Maoping deposit, clustered together and isolated from other types of inclusions. Despite some ductile deformation and subgrains observed in Qtz_{2-III} and topaz of the Maoping deposit at the crystal scale, FIs do not show any sign of stretching or necking-down.

4.2 Microthermometry and Raman spectrometry data

Microthermometric and Raman spectrometry data of the Maoping and Piaotang FIs are summarized in Table 6 and Figure 52. Th and salinity data are plotted in a binary diagram for two-phase aqueous inclusions (Figure 52A and B). Traces of CO_2 , CH_4 and N_2 gases were detected by Raman spectrometry in the vapor phase of the two-phase aqueous FIs of Qtz_{2-III} , Cst_{1-III} , Qtz_{4-IV} , Toz_{4-VI} in Maoping and Cst_{1-I} in Piaotang but no corresponding phase transitions were observed during microthermometry analyses (Figure 52C). Therefore, salinity and isochore reconstructions were carried out considering volatile-free FIs. No dissolved gases were observed in the one-phase aqueous liquid FIs from the Maoping deposit. CO_2 only was detected in the rare three-phase inclusions observed in Qtz_{2-III} in Maoping.

Salinity values display a continuum in both deposits: 0.0 to 12.6 wt% equiv. NaCl for Maoping and 0.4 to 8.3 wt% equiv. NaCl for Piaotang. Salinity variation within single minerals are within 2 to 8 wt% equiv. NaCl. Two groups of T_h values can be distinguished in both deposits: a low- T_h group between 150 and 250 °C and higher T_h group between 300 and 350 °C (Figure 52A and B). In both deposits, similarities are observed: inclusions in the quartz of the main mineralizing events (Qtz_{2-III} at Maoping and Qtz_{1-I} at Piaotang) show distinctively lower salinity values than other studied minerals (0.0 to ca. 6 wt% equiv. NaCl) and share similarities with very late fluorite as described below. These quartz also show lower T_h values similarly to late fluorite. Wolframite and cassiterite display much higher salinity (twice the value of the quartz). At Maoping, both Wf_{1-III}- and Cst_{1-III}-hosted FIs display a narrow range of high T_h . At Piaotang, Cst_{1-I}-hosted FIs show the same range of high T_h as Maoping values while Wf_{1-I}-hosted FIs display much lower values (180 and 230 °C) close to Qtz_{1-I} values (150 to 180 °C). Cassiterite-hosted FIs trace gases are dominated by CO₂ in both deposits (Figure 52C). In late-stage minerals (Qtz_{4-IV}, Fl_{1-VI} and Toz_{4-VI} at Maoping and Fl_{1-II} at Piaotang), salinity is generally comparable to those of cassiterite- and wolframite-hosted FIs and even sometimes higher in Qtz_{4-IV} at Maoping. FIs observed in late-stage minerals at Maoping can be divided into two groups: Qtz_{4-IV}- and Fl_{2-VI}-hosted FIs which display a similar range as in Qtz_{2-III} and Fl_{1-VI}, and Toz_{4-VI}-hosted FIs that show a narrow range of T_h as in Wf_{1-III}- and Cst_{1-III}. The total salinity of rare three-phase aquo-carbonic FIs is lower than in the majority of two-phase aqueous FIs (around 0.5 wt% NaCl equiv.) and share a similar range as two-phase aqueous FIs in the same host mineral (Qtz_{2-III}). Moreover, the salinity of the aqueous phase of three-phase inclusions also shares a similar range of values (3.3 to 4.0 wt%. NaCl equiv.).

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Table 6. Petrography, Raman Spectrometry and microthermometry data on fluid inclusions from the Maoping and Piaotang deposits. Values in parentheses indicate modes.

Mineral	Phases at 25 °C (trace gases after Raman spectrometry)	Proportion within a crystal (visual estimation)	Petrography	Morphology	Size (µm)	α (%) (25°C)	N	Microthermometry				Salinity
								T _{m(ice)}	T _{m(cia)}	T _{h(CO2)} (to vapor)	T _h (to liquid)	(wt.% NaCl equiv.)
MAOPING												
Stage III: W-Sn veins												
Quartz _{2-III}	L _{aq} -V _{aq} + trace CO ₂ , CH ₄ and N	85%	intragranular FIP or isolated	irregular	5-30	20-50	96	-4.5 to -0.2 (-0.7)	-	-	157 to 287 (220)	0.4 to 9.0 (1.3)
	L _{aq} -L _{CO2} -V _{CO2}	5%	isolated or cluster	irregular	10-20	85-95	6	-1.7 to -0.9 (-1.2)	7.7 to 8.1 (8.0)	29.4 to 30.6 (30.3)	200 to 252 (230)	0.4 to 0.5 (0.4)
	L _{aq}	10%	intragranular FIP or isolated	irregular	5-30	20-50	c	-	-	-	-	-
Wolframite _{1-II}	L _{aq} -V _{aq} ^a	100%	FIP along growth zone or intragranular	regular	5-25	20-50	30	-5.2 to -3.7 (-4.4)	-	-	284 to 346 (340)	7.2 to 10.5 (8.7)
Cassiterite _{1-II}	L _{aq} -V _{aq} + trace CO ₂ , CH ₄ and N	100%	FIP along growth zone or isolated	irregular	10-20	30-60	25	-5.5 to -3.0 (-3.8)	-	-	299 to 338 (340)	5.8 to 11.2 (7.5)
Stage IV: Banded quartz veins												
Quartz _{4-IV}	L _{aq} -V _{aq} + trace CO ₂	100%	intragranular FIP	regular	5-15	20-30	41	-6.1 to -1.3 (-4.2)	-	-	148 to 291 (240)	7.7 to 12.6 (9.0)
Stage VI: Late phases												
Fluorite _{1-VI}	L _{aq} -V _{aq} ^b	100%	FIP along growth zone or intragranular	regular	5-15	30-60	20	-5.4 to -4.3 (-4.5)	-	-	195 to 325 (340)	8.1 to 11.1 (9.0)
Topaze _{4-VI}	L _{aq} -V _{aq} + trace CO ₂	100%	FIP along growth zone or clusters	irregular	5-30	10-50	46	-4.2 to -3.3 (-3.9)	-	-	307 to 346 (340)	5.6 to 7.0 (6.1)
Fluorite _{2-VI}	L _{aq} -V _{aq} ^b	100%	FIP along growth zone	regular	5-20	10-20	15	-0.9 to -0.1 (-0.1)	-	-	136 to 292 (230)	0 to 1.6 (0.1)
PIAOTANG												
Stage I: Silicate-oxide												
Quartz _{1-I}	L _{aq} -V _{aq}	100%	intragranular FIP or isolated	irregular	5-15	10-30	20	-2.2 to -0.2 (-2.2)	-	-	144 to 175 (160)	0.4 to 3.8 (2.2)
	L _{aq}	10%	intragranular FIP or isolated	irregular	5-15	10-30	c	-	-	-	-	-
Wolframite _{1-I}	L _{aq} -V _{aq} ^a	100%	FIP along growth zone	regular	5-15	10-40	18	-5.3 to -3.1 (-4.2)	-	-	173 to 222 (210)	5.6 to 8.3 (6.5)
Cassiterite _{1-I}	L _{aq} -V _{aq} + trace CO ₂ , CH ₄ and N	100%	FIP along growth zone or intragranular	regular	5-20	10-40	24	-3.7 to -1.4 (-3.5)	-	-	283 to 349 (340)	2.4 to 6.2 (5.8)
Stage II: Calcic stage												
Fluorite _{1-II}	L _{aq} -V _{aq} ^b	100%	intragranular FIP or isolated	regular	15-40	10-30	23	-5.0 to 0.0 (0)	-	-	142 to 185 (170)	0 to 7.7

^a Wolframite is an opaque mineral and therefore, could not be analyzed by Raman spectrometry

^b Due to fluorescence, gas peaks were impossible to observe by Raman spectror

^c Phase transitions could not be observed in one-phase liquid inclusions

Abbreviations: α = vapor phase, L = liquid, V = vapor, aq = aqueous, FIP = fluid inclusion plan, T_m = melting temperature, T_h = homogenization temperature, cia = clathrate

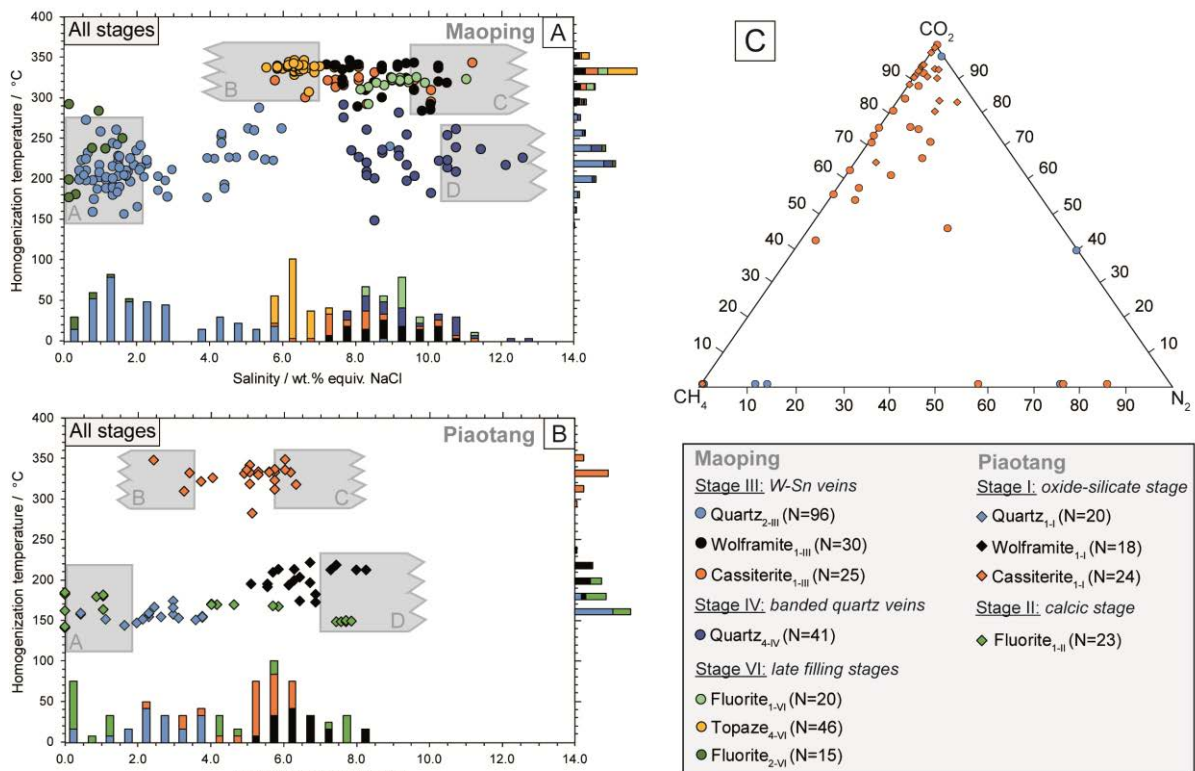


Figure 52. Microthermometric and Raman spectrometry data on two-phase aqueous FIs for the Maoping and Piaotang deposits. (A) Binary salinity vs. homogenization temperature diagram for Maoping FIs at all stages in quartz, cassiterite and wolframite from the W-Sn mineralized stage III, quartz crystallized in the banded quartz veins on stage IV and fluorite and topaz in late stage VI. Possible fluid end-members are indicated. Serrated sides of the boxes indicate the direction along which the exact position of the actual end-member is uncertain. (B) Binary salinity vs. homogenization temperature diagram for Piaotang FIs at all stages in quartz, cassiterite and wolframite from the silicate-oxide mineralizing stage I and late fluorite from the carbonate stage II. Possible fluid end-members are indicated. Serrated sides of the boxes indicate the direction along which the exact position of the actual end-member is uncertain. (C) Gas concentrations obtained by Raman spectrometry on in the vapor phase of FIs in quartz and cassiterite of stage III at Maoping and cassiterite from stage I at Piaotang (no gases detected in quartz) represented in ternary diagram showing relative molar proportions of CO₂, CH₄ and N₂.

Isochore estimates were calculated from microthermometry measurements in every mineral of interest of the Maoping and Piaotang deposit (Figure 53). In the Maoping deposit, Qtz_{2-III} is the only mineral where PT conditions could be constrained by both two-phase aqueous inclusions and three-phase aquo-carbonic inclusions. Estimates indicate hydrostatic conditions with a very high gradient (above 90°C/km) and temperature and pressure ranging from 150 °C to 300 °C and 5 to 40 MPa respectively. No petrographic observations could indicate a change from hydrostatic to lithostatic conditions in our systems. Therefore, all other minerals were considered under hydrostatic conditions. No PT constraints (besides microthermometry measurements) could be applied to these minerals and therefore a very large

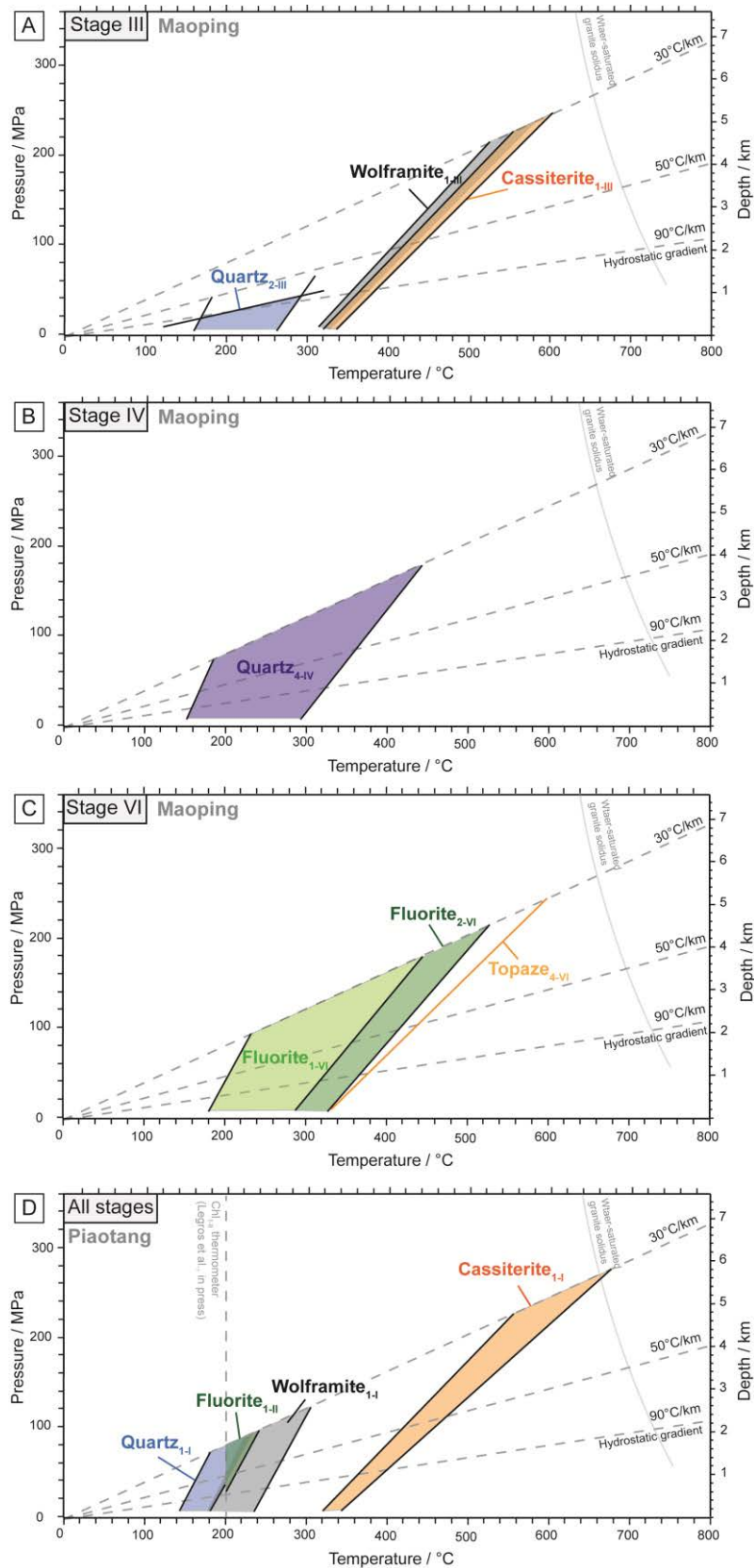


Figure 53. Calculated isochores for representative homogenization temperature ranges for FIs from the different minerals studied for the Maoping and Piaotang deposits, and their intersection with possible thermal gradients assuming hydrostatic conditions. (A) Qtz_{2-III}, Wf_{1-III} and Cst_{1-III} of stage III the Maoping deposit. (B) Qtz_{4-IV} of stage IV the Maoping deposit. (C) Fl_{1-VI}, Toz_{4-VI} and Fl_{2-VI} of the late stage VI of the Maoping deposit. (D) Qtz_{1-I}, Wf_{1-I}, Cst_{1-I} and Fl_{1-II} of stages I and II of the Piaotang deposit. Wet-saturated solidus of granitic magmas after Vigneresse et al. (1995).

range of possible hydrostatic gradients (from 30°C/km and above) were considered for our estimates. Overall, two groups can be distinguished in both deposits (reflecting previous T_h groups). First group is composed of Qtz_{2-III}, Qtz_{4-IV} and Fl_{1-VI} FIs in Maoping and Qtz_{1-I}, Wf_{1-I} and Fl_{1-II} FIs in Piaotang with pressures between 5 and 160 MPa and temperatures from 140 to 400 °C. The second group is composed of Wf_{1-III}, Cst_{1-III}, Fl_{1-VI} and Toz_{4-VI} FIs in Maoping and Cst_{1-I} in Piaotang with pressures from 5 to 250 MPa and associated temperatures between 300 °C and 700 °C.

4.3 LA-ICPMS data

Two-phase aqueous FIs from the Maoping and Piaotang deposits were selected for LA-ICPMS analyses according to (i) previous characterization by optical microscopy and microthermometry; (ii) relatively large-size (10-30 µm) and (iii) appropriate distance to the polished thick section surface (< 15 µm). From Maoping, a total of thirty-two FIs were selected from quartz (N = 11), cassiterite (N = 7), fluorite (N = 4) and topaz (N = 10). From Piaotang, a total of twenty-nine FIs were selected from quartz (N = 8), cassiterite (N = 11) and fluorite (N = 10). LA-ICPMS data are reported in Table 7, Figure 54 and Figure 55. As the results are similar for FIs from equivalent host minerals at Maoping and Piaotang, LA-ICPMS data will be further described without distinction between the deposits.

Due to the relatively small size of FIs combined with their relatively low-salinity, many data lie below LODs. Generally speaking, LODs vary from one analysis to another as a function of the amount of the given element in the matrix, the magnitude of the signal for the inclusions and the background intensity. Here, the proportion of analyses quantified above the LOD values is of: Na (100%), Cs (92%), Li (90%), K (87%), Rb (85%), Mn (64%), Sn (54%), Mg (48%), W (48%), Sr (25%), Ba (25%), Mo (20%), Ta (20%), Nb (18%), Y (16%) and Ca (13%). For FIs with concentration <LOD, LODs show similar range to measured values on other inclusions from the same mineral.

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Table 7. LA-ICPMS measurements performed on fluid inclusions from the Maoping and Piaotang deposits. Values preceded by a < symbol stand for element concentrations below the limit of detection.

MAOPING	Salinity (wt.% equiv. NaCl)	Li (ppm)	Na (ppm)	Mg (ppm)	K (ppm)	Ca (ppm)	Mn (ppm)	Rb (ppm)	Sr (ppm)	Y (ppm)	Nb (ppm)	Mo (ppm)	Sn (ppm)	Cs (ppm)	Ba (ppm)	Ta (ppm)	W (ppm)	Na/Li (molar ratio)	K/Na (molar ratio)	K/Rb (mass ratio)
Quartz _{2-III}	1.3	37	1000	43	490	<5200	160	2	<1.5	<1.6	<1.4	<8.6	42	<1	<1.4	1.1	24	8.4	0.3	196
	1.5	84	1600	28	530	<4000	810	7	<1.5	<1.1	<1.4	58	18	1.8	3.0	<1	41	5.8	0.2	75
	0.5	41	1200	55	110	<410	74	<1	<1	<1	<1	11	4.7	2.8	8.5	<0.06	7.8	9.1	0.1	-
	6	360	13000	220	9000	<24000	180	22	11	<7.6	<8.5	<37	49	18	<9.2	<3.9	<21	11.3	0.4	414
	1.5	61	2300	32	500	2500	120	<1	1.0	<1	<1	4.4	37	6.0	1.3	<1	10	11.7	0.1	-
Cassiterite _{1-III}	4.1	250	6300	180	1200	<21300	220	<11	<8.3	<7.9	<6.6	<39	73	26	<7.9	<3.7	<18	7.7	0.1	-
	8.3	2600	17000	<60	9500	<25000	1280	660	<8	<11	<65	<55	<8060	960	<8	<61	<40	2.0	0.3	14
	10	3200	26000	<50	4300	<21000	<90	650	<10	<5	<40	<35	<7850	500	<7	<31	<34	2.4	0.1	7
	8.5	2700	21000	<90	3600	<37000	170	380	<12	<12	310	<77	<11100	500	<10	650	<45	2.4	0.1	10
	8.5	2300	19000	<250	8500	<96000	<420	540	<38	<20	230	<170	<34600	700	<21	<170	150	2.6	0.3	16
	8.5	1800	20000	<430	10000	<138000	1000	560	<47	<44	<200	<250	<48200	640	<55	<190	<170	3.3	0.3	18
	8.5	3200	20000	<54	2400	<25000	<110	280	<7	<9	33	<45	<5200	410	<8	<22	<28	1.9	0.1	9
	8.5	1600	21000	<250	10400	<91000	970	390	<31	<34	<460	<110	<26000	460	<27	<840	<80	4.0	0.3	27
	9	2200	18500	<150	8700	<43000	4200	320	<16	<12	<14	110	450	60	16	<13	270	2.5	0.3	27
	9	<90	6000	<51	9800	20000	450	250	<6	<5	<5	57	<67	9	<4	5	160	-	1.0	39
Quartz _{4-IV}	9	90	1400	8300	1600	<17000	500	<9	9	<5	<6	<48	140	<4	<5	<3	220	4.9	0.7	-
	9	260	7000	9900	2200	<12000	1400	27	10	<4	<3	83	120	40	<4	<2	300	8.1	0.2	82
	9	400	7300	5300	4800	<19000	760	19	7	<5	<6	87	220	23	6	<4	200	5.5	0.4	250
	8	<2200	27700	<970	<15600	<432000	<1800	<240	1100	<2400	<130	<780	<1800	<120	<140	<75	<340	-	-	-
Fluorite _{1-VI}	9	<2800	34100	<1200	<19200	<608000	<2500	<290	370	<4800	<200	<870	2800	<140	<200	<79	<430	-	-	-
	9.6	<890	37700	<450	<6250	<196000	<830	<120	<82	<1700	<59	<220	220	<73	<21	<150	<150	-	-	-
	1	<55	3900	<26	<400	<116000	<49	<5	<4	<85	<4	<20	<45	<3	<4	<2	<9	-	-	-
Topaz _{4-VI}	6.4	230	20000	<13	<1700	<7600	79	330	<3	<2	<2	<27	200	310	<2	1	190	27.5	-	-
	6.4	1600	19000	<38	<18000	<23000	360	560	12	<6	<6	<100	100	660	<5	<4	<56	3.8	-	-
	6.4	280	3200	<1	36000	<1100	120	170	<1	1	<1	<4	7	150	<1	<1	10	3.5	6.6	1200
	6.4	280	6400	<7	27000	<4700	99	730	2	2	<2	<12	54	340	<1	<1	190	6.9	2.5	200
	6.4	1500	9400	5	12000	<3500	65	630	2	<1	<1	<8	18	420	<1	1	160	2.0	0.8	37
	6.4	710	10000	120	13000	<6400	380	730	4	430	5	<13	140	930	<2	5	180	4.4	0.7	19
	6.4	1400	6500	33	8200	<11000	580	910	<8	<28	7	<36	<66	850	<15	7	420	4.8	1.3	9
	6.4	1000	10000	<44	18000	<13000	440	1400	<9	<11	<10	<48	150	1000	<25	<14	240	9.3	1.8	13
	6.4	1200	14000	<33	10000	<8600	300	1000	62	<3	<6	<23	110	1500	<24	<6	190	11.4	0.7	10
PIAOTANG	6.4	980	10000	<33	13000	<12000	660	1600	<9	34	<5	<25	<71	2300	<16	<4	160	10.2	1.3	8
	4.9	200	2300	670	<350	13000	<38	<3	<3	<3	<2	<20	100	4	30	<2	<7	10.7	-	-
	4.7	98	5400	120	670	10000	<18	7	19	<1	<2	10	200	18	6	<1	<4	54.6	0.1	99
	4.5	<31	4100	35	1500	10000	<29	8	<3	<2	<2	<10	<22	4	19	<1	5	-	0.4	190
	4.7	400	9100	<37	2200	<14000	1000	49	<6	<4	<4	<20	<41	5	<5	<3	<10	22.8	0.2	45
	3.1	120	3400	100	160	6400	30	<3	<2	<1	<1	12	130	4	43	9	850	28.5	0.0	-
	3.7	80	1530	560	1200	9200	71	9	3	<2	<2	17	330	11	<5	<1	350	19.0	0.8	130
	5.2	180	16338	15	1200	<2800	330	150	2	<2	<1	<8	62	400	<5	<1	<5	89.2	0.1	8
	0.9	10	960	430	450	1200	9	2	3	<1	<1	<1	51	1	1	<1	<1	98.4	0.5	200
	7.9	2400	17000	<59	7400	<21000	<94	350	<8	<6	10	<40	<7100	600	<6	<3	130	2.1	0.3	21
	8.3	2400	18000	<52	5700	<14000	<63	200	<5	<5	<5	<29	<5900	430	<6	<2	<18	2.3	0.2	27
	9.6	2800	23000	<21	7400	<8500	<37	350	<3	<3	25	<14	<2400	750	<2	<2	<9	2.5	0.2	21
	9.6	2300	23000	<80	11400	<27000	<123	420	<9	<9	<23	<49	<8000	560	<7	<5	<37	3.1	0.3	27
	7.6	2400	17000	<44	8200	<16000	<72	350	<5	<6	15	<36	<5100	800	<6	7	<22	2.2	0.3	23
	8	2900	18000	<50	4400	<17000	<80	220	<7	<7	71	<36	<4400	610	<6	14	<23	1.8	0.1	20
	10.3	3700	23000	<12	8700	<4300	<20	240	<1	<1	<2	<7	<2100	780	<1	1	9	1.9	0.2	36
	6.2	1800	13000	<41	4600	<20000	110	160	<8	<6	41	<23	<8700	400	<8	<3	130	2.2	0.2	28
	9.6	3300	22000	<56	7500	<31000	<140	220	<11	<12	<14	<51	<10300	490	<11	<6	220	2.1	0.2	34
	6.2	1800	15000	<90	4500	<32000	<150	200	<11	<10	<12	<63	<9700	460	<12	<5	<40	2.6	0.2	23
	7.4	2200	16000	<29	7500	<12000	280	250	<5	<4	15	<24	<4300	600	<4	<2	140	2.2	0.3	30
	9.6	290	18000	300	1700	<19000	120	39	3200	27	<1	<6	340	61	15	2	16	18.5	0.1	44
	9.6	510	32000	690	3700	<29000	280	93	<220	<5	<3	32	360	150	14	3	<7	19.0	0.1	40
	4.7	310	11000	40	1100	<4400	<8	31	1100	20	<1	<4	12	32	2	1	<1	10.5	0.1	36
	4.7	21	1100	40	<78	<4300	38	3	3300	10	<1	9	74	7	<1	1	10	15.7	-	-
	9.6	650	29000	390	2400	<42000	250	100	770	<15	<15	<88	390	120	<47	<8	<42	44.2	0.1	30
	4.7	260	12000	1200	5200	<75000	<320	<69	<540	<48	<35	<200	<360	70	<95	<22	<110	45.9	0.4	25
	9.6	300	13000	1500	1500	<11000	110	110	3700	71	<4	<22	59	41	<15	<3	<14	44.8	0.1	-
	9.6	110	26000	800	1300	<11000	63	50	1700	100	<3	<16	60	33	23	<1	<9	226.0	-	14
	9.6	530	24000	470	5600	<32000	<100	130	1400	28	<10	<58	530	160	<32	<6	<33	45.7	0.2	26
	10	670	31000	200	2900	<2900	62	96	720	<1	<1	<4	<8	140	2	1	<1	14.1	0.1	43

Altogether, the two main cations are Na and K, ranging from 960 to 37,700 ppm and from 110 to 36,000 respectively. Ca also shows high values from 1200 to 20,000 ppm but could only be quantified in few quartz-hosted FIs from both deposits (N=8). Quantified minor and trace element concentrations are as follows: Mg (5 to 9,900 ppm), Mn (38 to 4,200 ppm), Li (21 to 3,700 ppm), Sr (0 to 3,700 pm), Sn (5 to 2,800 ppm), Cs (2 to 960 ppm), Ba (2 to 960 ppm), Rb (1 to 730 ppm), Ta (1 to 650 ppm), Y (1 to 430 ppm), Nb (5 to 310 ppm), W (5 to 300 ppm) and Mo (4 to 110 ppm). The following systematics are observed. In early Qtz_{2-III}- and Qtz_{1-I}-hosted, FIs are the most depleted in Rb, Cs and Li compared to all other minerals. Cst_{1-III}- and Cst_{1-I}-hosted FIs from both deposits show the highest concentrations in Li, Nb, Sn (Sn < LOD in Maoping cassiterite) and high amount of Cs and W (if detected). Fl_{1-VI}- and Fl_{1-II}-hosted FIs show the highest contents in Sr. At Maoping, Qtz_{4-IV}-hosted FIs show high contents in Li, Sn, W and the highest contents in Mg and Mn. Toz_{4-VI}-hosted FIs show the highest K concentration, the lowest detected Mg content. In a more general matter, in both deposits, low-Rb concentrations are broadly correlated with low-W-Sn-Nb-Ta concentrations and high-Li and/or Cs concentrations are broadly correlated with high-W and/or Sn concentrations (Figure 54).

In a Na-Li-K ternary diagram (Figure 55A), quartz- and fluorite-hosted FIs plot in comparable fields (relatively Na-rich) while cassiterite- and topaz-hosted FIs plot in two distinctive fields (relatively Li-rich and K-rich respectively). In Na vs. Li and Na vs. K diagrams, the data are scattered between a Na-K-Li-rich (high salinity) end-member and a Na-K-Li-poor (low-salinity) end-member (Figure 55B and C).

Quartz from both deposits shows high K/Rb and low Cs concentrations, while fluorite has low K/Rb and intermediate Cs content and cassiterite and topaz have low K/Rb and high Cs content (Figure 55D).

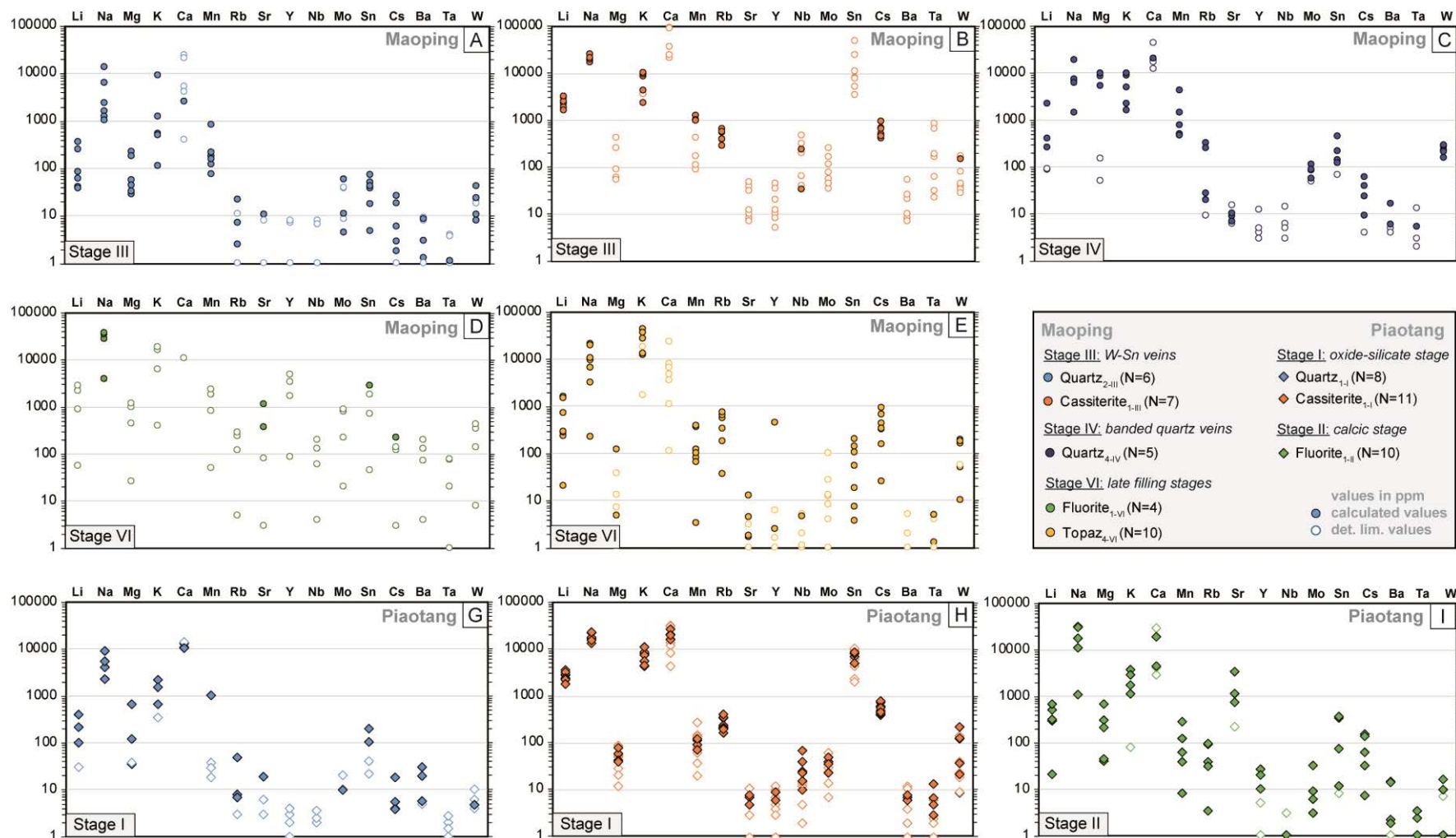


Figure 54. Major and trace element concentrations in FIs from the Maoping and Piaotang deposits as determined by LA-ICPMS analysis. All concentrations are expressed in ppm. (A) Qtz_{2-III}, (B) Cst_{1-III}, (C) Qtz_{4-IV}, (D) Fl_{1-VI}, (E) Toz_{4-VI} and (F) surroundings from the Maoping deposit. (G) Qtz_{1-I}, (H) Cst_{1-I} and (I) Fl_{1-II} from the Piaotang deposit. Each symbol represents a single ablated fluid inclusion. Color symbols are calculated values above the LODs. Empty symbols represent the LODs when the element was considered undetected.

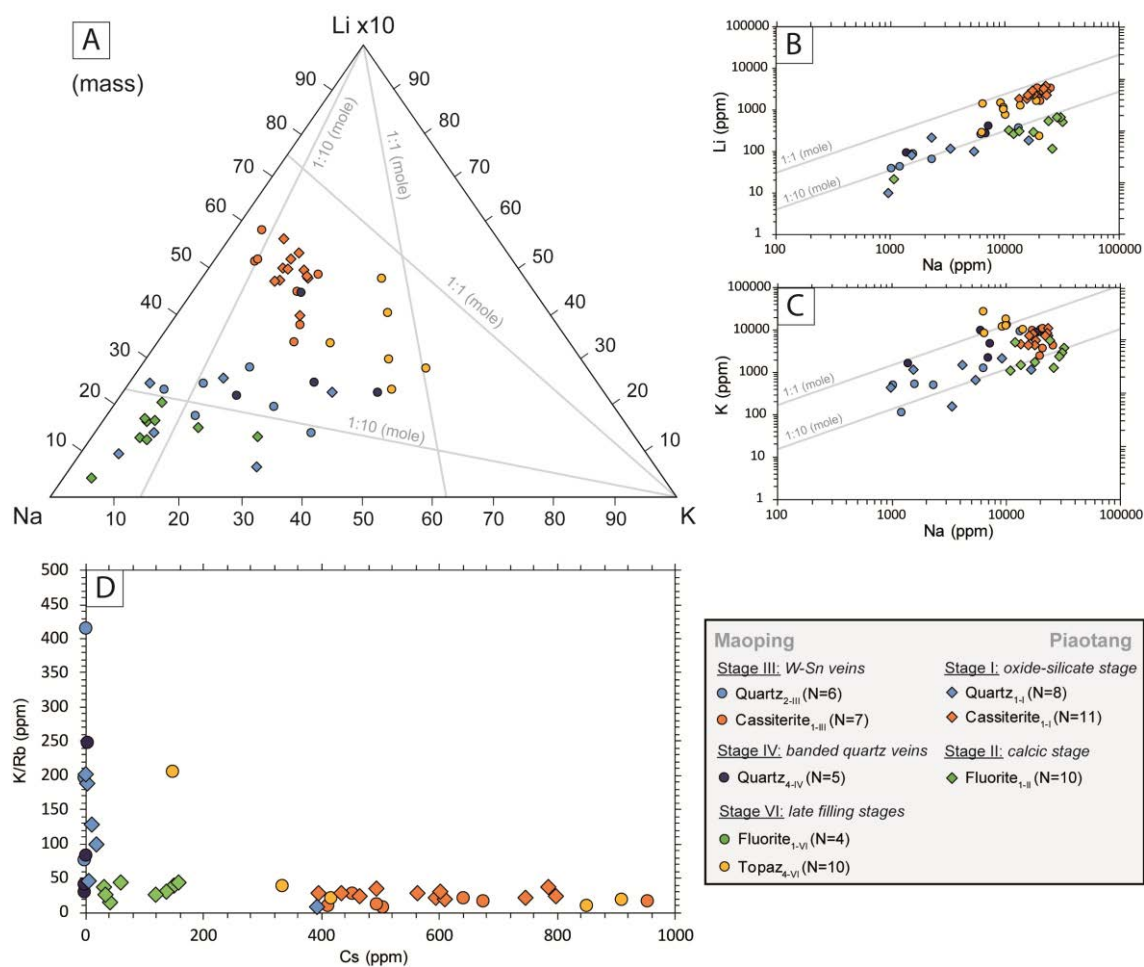
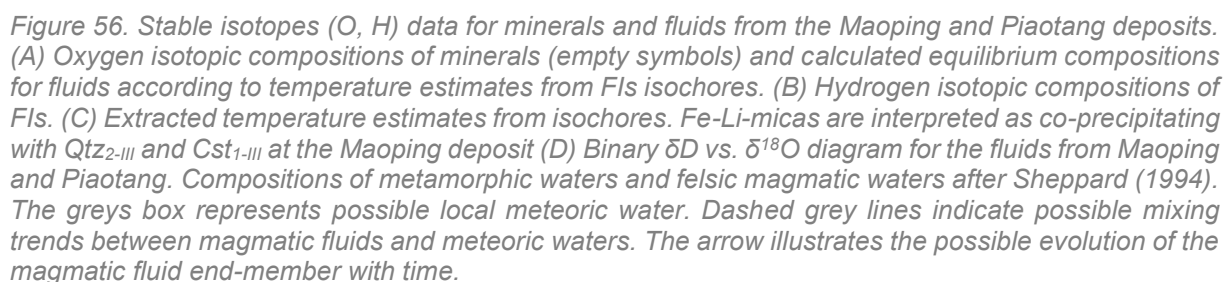


Figure 55. Selected element concentrations relationships from LA-ICPMS analyses of fluid inclusions from the Maoping and Piaotang deposits. Each symbol represents a single ablated fluid inclusion. (A) Na-K-Li ternary diagram. The compositions are calculated as a function of their respective concentrations expressed in ppm. Lines indicating equal values for K:Na and Li:Na molar ratios (1:1 and 1:10) are plotted in grey. (B) and (C) Na vs. Li and Na vs. K concentration diagrams. Lines indicating equal values for K:Na and Li:Na molar ratios (1:1 and 1:10) are plotted in grey. (D) K/Rb vs. Cs concentrations diagram where K/Rb is expressed as a function of the respective concentrations as expressed in ppm.

4.4 Oxygen and hydrogen stable isotopes data

Oxygen and hydrogen isotopic compositions of minerals and fluids from the Maoping and Piaotang deposits are presented in Table 8 and Figure 56.

At the Maoping deposit, oxygen stable isotopes analyses were performed on crystals of Fe-Li-micas_{2-II} from stage II, Qtz_{2-III}, Wf_{1-III}, Cst_{1-III} and Fe-Li-micas_{3-III} from stage III, Qtz_{4-IV} from stage IV and Toz_{4-VI} from stage VI. At the Piaotang deposit, Qtz_{1-I}, Wf_{1-I} and Cst_{1-I} were analyzed. Calculated values of the fluids are highly variable (from -0.7 to +9.1 ‰) in both deposits (Figure 56A) due to the large range of temperatures interpreted from isochores



At the Maoping deposit, hydrogen isotopes analyses were performed on FIs from Qtz_{2-III}, Wf_{1-III} and Cst_{1-III} from stage III and Qtz_{4-IV} from stage IV. At the Piaotang deposit, hydrogen isotopes analyses were performed on FIs from Qtz_{1-I}, Wf_{1-I} and Cst_{1-I} (Figure 56B). δD values of FIs are within -77.6 to -50.5 ‰ for each analyzed minerals. At the deposit scale, δD values are from -77.6 to -55.2 ‰ at Maoping and from -69.6 to -50.5 ‰ at Piaotang. For both deposits, the following systematics can be observed for δD values: early Qtz_{2-III} and Qtz_{1-I} range from -59.2 to -50.5 ‰ (highest measured values), Wf_{1-III} and Wf_{1-I} range from -77.6 to -75.8 ‰ (lowest measured values) and Cst_{1-III} and Cst_{1-I} display intermediate values from -71.5 to -57.9 ‰.

Table 8. Stable isotopes (O, H) results on minerals and fluids from the Maoping and Piaotang deposits.

Mineral	$\delta^{18}O$ (‰VSMOW) _{mineral} measured	δD (‰VSMOW) _{mineral} measured	T range (°C) estimated from isochores	$\delta^{18}O$ (‰VSMOW) _{fluid} calculated	δD (‰VSMOW) _{fluid} measured
MAOPING					
<i>Stage II: Fe-Li-micas veins</i>					
Fe-Li-micas _{2-II}	8.3	-	200-350	3.8 to 7.7	-
<i>Stage III: W-Sn veins</i>					
Quartz _{2-III}	10.9	-	200-350	-0.7 to 5.3	-59.2 to -55.2
Wolframite _{1-III}	0.5	-	380-420	3.1 to 3.3	-77.6 to -76.8
Cassiterite _{1-III}	-	-	380-450	-	-71.5
Fe-Li-micas _{3-III}	8.7	-	380-450	8.5 to 9.1	-
<i>Stage IV: Banded quartz veins</i>					
Quartz _{4-IV}	10.8	-	200-350	-0.8 to 5.2	-65.9 to -61.6
<i>Stage VI: Late phases</i>					
Topaze _{4-VI}	8.4	-	420-450	7.9 to 8.2	-
PIAOTANG					
<i>Stage I: Silicate-oxide</i>					
Quartz _{1-I}	11.6	-	160-200	-3.0 to 0.0	-76.7 to -75.8
Wolframite _{1-I}	-3.0	-	200-270	-3.6 to -1.8	-53.6 to -50.5
Cassiterite _{1-I}	-	-	380-450	-	-69.6 to -57.9

5. Discussion

5.1 Fluid identification and characterization

In both deposits, microthermometric characteristics as well as elemental and isotopic compositions of FIs show compositional variations indicating several end-members and mixing trends associated with successive episodes of mineral crystallization. Four end-members representing successive fluids are observed in Maoping and Piaotang FIs. The identification of these end-members is primarily made from the interpretation of microthermometric data.

The scattering of T_h and salinity values (Figure 52A and B) suggest the involvement of four possible fluid end-member in each deposits. The data plot between a low-salinity low-temperature end-member (**A**), an intermediate-salinity high-temperature end-member (**B**), a high-salinity high-temperature end-member (**C**) and a high-salinity low-temperature end-member (**D**). The continuum of data between the possible end-member suggest mixing trends between (**A**) and (**B**), (**B**) and (**C**), and possibly (**B**)-(**C**) and (**D**) at Maoping and between (**A**) and (**D**), and (**C**) and (**D**) at Piaotang.

The first end-member (**A**) is identified from quartz- and fluorite-hosted FIs. Qtz_{2-III} in contrast to other minerals of this FI study, display three-phase aquo-carbonic inclusions in addition to the mainly observed two-phase aqueous FIs (Figure 51). This end-member is characterized by low salinity (between 0 and 6 wt% NaCl equiv.) and homogenization temperatures between 100 and 250 °C (Figure 52). Reconstructed PT conditions for trapping using isochores and a very large range of hydrostatic gradients suggest temperatures of emplacement of Qtz_{2-III} and Qtz_{1-I} between 150 and 300°C and associated pressure below 100 MPa for the (**A**) end-member (Figure 53). According to LA-ICPMS results, this end-member is associated with lower Li/Na and K/Na ratios, low salinity, and low Li and K contents (below 100 and 1,000 ppm respectively). In the binary diagrams (Figure 55B and C), the extrapolation of the (**A**) end-member to the most depleted Li and K contents do not reach the origin of the diagram, implying a remaining Na content. The (**A**) end-member is also depleted in most incompatible elements such as rare metals, Cs and Rb (Figure 54 and Figure 55) in comparison to other mineral FIs.

Two other end-members called (**B**) and (**C**) can also be identified and are mostly associated to ore minerals. They are defined as the two extremities of the trend formed by microthermometric data of wolframite, cassiterite, topaz and fluorite at Maoping and cassiterite at Piaotang. These trends are characterized by a nearly constant range of homogenization temperature (270-350 °C), which is higher than what observed for the previous (**A**) end-member (Figure 52). Values display a wide range of salinity values (from 5.0 to 12.0 wt.% equiv. NaCl at Maoping and 2.0 to 7.0 wt.% equiv. NaCl at Piaotang). These two end-members

are characterized by minerals where aqueous two-phase FIs were observed only. Reconstructed PT conditions for trapping using isochores indicate temperatures of trapping between 350 and 600°C and pressures of trapping between 20 and 300 MPa (Figure 53). The **(B)** and **(C)** end-members are discriminated by their salinity: the **(B)** end-member represents the low-salinity high-temperature end-member while **(C)** represents the high-salinity high-temperature end-member (Figure 52). These end-members are both characterized by higher Li/Na and K/Na contents (above 7,000, 100 and 1,000 ppm for Na, Li and K respectively). Moreover, they show very low K/Rb ratios and an enrichment in incompatible elements such as rare metals and Cs compared to the **(A)** end-member.

Therefore, it appears that FIs in the main gangue mineral (quartz) do not reflect either wolframite- or cassiterite-forming fluids. Moreover, in the case of the Piaotang deposit, wolframite-forming fluids seems to be distinct from cassiterite-forming fluids.

The last end-member **(D)** is identified through two-phase aqueous quartz-hosted FIs at Maoping (Qtz_{4-IV}) and wolframite- and fluorite-hosted FIs at Piaotang and is characterized by homogenization temperatures equivalent to the **(A)** end-member and salinity similar to the **(C)** end-member (Figure 52). Reconstructed PT conditions for trapping conditions using isochores indicate temperatures between 150 and 400 °C and pressures below 150 MPa (Figure 53). The **(D)** end-member is defined by intermediate Li/Na and K/Na ratios and incompatible elements content compared to the **(A)** and **(B)-(C)** end-members (Figure 55).

It is noteworthy that, although Maoping and Piaotang do not share exactly the same timing of end-members involvement as described by the paragenesis, the same four fluid types seem to have been involved.

Moreover, it is remarkable that in every FIs analyzed from each deposits, tungsten as well as other rare metals (Sn Nb, Ta) have been quantified. This observation demonstrates that tungsten, in particular, is still present in the fluids at all stages without precipitating systematically. Therefore, P-T-x conditions must have been varying through stages. From pre- to post-mineralizing stages, the following parameters are varying: salinity, temperature, cation ratios (Na, K) and gaseous content of FIs. As tungsten remains in the fluid during these

contrasting conditions and do not precipitate at all stages, these parameters must not play a major role in ore mineral crystallization. An alternative hypothesis would be that tungsten saturation in the fluid was only reached during wolframite crystallization while tungsten was preferentially incorporated into other mineral structure during other stages (e.g. cassiterite, micas...).

5.2 Nature and origin of fluids

Major ore minerals in the Maoping and Piaotang deposits are characterized by wolframite and cassiterite that mainly crystallize in association with quartz during one early mineralization stage that follows the magmatic-hydrothermal transition (Legros et al., 2016 and in press). Stable isotopes analyses (oxygen and hydrogen) allow us to distinguish three end-members during the mineralizing stage among quartz and wolframite FIs data from both deposits. Both wolframite and quartz display a mixing trend toward a meteoric end-member (Figure 56D). This latter would likely represent the **(A)** end-member defined previously with low salinity and low temperature. On the other hand, the high-temperature fluids involved in the quartz and wolframite crystallization are characterized by two distinct δD values. The high δD end-member, associated with quartz crystallization, is likely to represent the **(B)** end-member while the low δD end-member, associated with wolframite crystallization, is likely to be the **(C)** or **(D)** end-member (at Maoping and Piaotang respectively). Given this observation, it is clear that quartz and wolframite did not coprecipitate in either Maoping or Piaotang deposits.

Stable isotope compositions (oxygen and hydrogen) of both deposits, displayed in a binary diagram (Figure 56D), tend to suggest a possible magmatic or metamorphic origin of the **(B)** end-member while the **(C)** and **(D)** end-member (associated to wolframite crystallization) are likely to represent a magmatic fluid.

On one hand, as quartz-wolframite-cassiterite succession seems to display a consistent evolution in both deposits, the hypothesis of a high-temperature rare-metal common end-member evolving in composition from quartz crystallization through wolframite and finally

cassiterite precipitation, can be suggested. Moreover, the hypothesis of a magmatic fluid is consistent with the high salinity, high temperature and rare metal content observed for these end-members even if the Maoping and Piaotang deposits do not display such high temperatures ($>600\text{ }^{\circ}\text{C}$) and salinity as often observed in the case of magmatic fluids (Audétat et al., 2000; Webster et al., 2004; Rickers et al., 2006). Indeed, the FIs observed in our study reflect an intermediate composition which has already been diluted by the meteoric fluid ((**A**) end-member).

On the other hand, few three-phase aquo-carbonic inclusions and traces of CO_2 gases were observed in quartz and cassiterite. The observation of such inclusions is usually associated with a metamorphic origin as interpreted in multiple deposits from the Variscan belt (Weisbrod, 1988; Norohna et al., 1992 and 1999; Vindel et al., 1995). This criteria was established according to Papale et al. (2006) who demonstrated that low-pressure water-rich granitic melts have very low- CO_2 content by definition and thus, could not provide for these gas-rich fluids. Yet, magmatic fluids can also host CO_2 gases. Solubility of CO_2 in silicate melts is usually one magnitude order lower than H_2O . Thus, CO_2 will preferentially partition into the fluid phase, decreasing the $\text{CO}_2/\text{H}_2\text{O}$ ratio of the remaining melt (Yoshimura, 2015). In that case, the rare abundance of CO_2 -rich fluids during quartz crystallization could still be of magmatic origin as noticed by Vigneresse (2006). Moreover, no relation with the host rock was observed in the mine. Therefore, both (**B**) and (**C**)-(D) end-members crystallizing quartz and ore minerals respectively would be of magmatic origin. In that case, some parameters of the evolving magmatic fluid must be changing between the quartz and ore mineral crystallization. Wolframite at the Maoping and Piaotang deposit crystallizes at different ranges of temperatures and do not show any gas content in FIs. Therefore, temperature as well as the presence of gas does not play an important role for the metal transporting capacities of the fluid. Moreover, comparing the Maoping and Piaotang (**B**) and (**C**)-(D) end-members, the salinity is also fluctuating.

In our case, the difference between the quartz and ore mineral crystallizing fluids could be explained by the differentiation of the granite. Indeed, the decrease of the K/Rb ratio is a

good indicator of differentiation (Shaw, 1968; Klemm et al., 2008; Luders et al., 2009). In our case, quartz K/Rb ratios are significantly higher than ratios observed for ore mineral FIs (Figure 55D). Moreover, FIs in ore minerals show higher content in incompatible elements as Cs and rare metals (Figure 54 and Figure 55) that could have been enriched in the residual melt during differentiation. The decreasing δD range between quartz and ore mineral FIs could be explained by a degassing of water from the magma (Taylor et al., 1983; France-Lanord et al., 1988). Moreover, FIs in quartz display two different contents (aqueous and aquo-carbonic) with two different vapor fractions (from 10 to 95 vol %) but similar salinity and temperature. Unmixing could also explain the vapor separation process, and thus give an alternative explanation to this low δD remaining ore-crystallizing fluid (Koděra et al., 2005; Yoo et al., 2010; Wei et al., 2012).

In both deposits, late stages are associated with fluorine and more or less metals depending on the source. Those stages are associated with a new heat input between 200 °C and more than 400 °C ((**D**) end-member). This input could be due to the supposed hidden second intrusion proposed by Legros et al. (2016) at Maoping. K/Rb vs Cs binary diagram of LA-ICPMS FI data of Qtz_{4-IV} at Maoping and fluorite at Piaotang show that this fluid would be a new magmatic fluid from a low differentiated intrusion. However, this intrusion was described as a peralkaline source providing for the Nb-Ta-Zr-REE mineralization and no FI data this far can confirm this hypothesis besides an abnormal amount of calcium in FIs.

5.3 Processes involved in W-Sn deposits

5.3.1 Fluid evolution in the Maoping and Piaotang deposits

This study is focused on the early crystallization of hydrothermal veins after the magmatic-hydrothermal transition.

The emplacement starts under hydrostatic conditions, ca. 150 and 250 °C and pressure below 100 MPa. The granitic melt is providing fluids with low metal content (because of early differentiation) that form the veins ((**B**) end-member). Temperature and pressure are enforced by the granite emplacement as well as chloride complexes in fluids. The emplacement of the

granite in the crust, mobilizes external fluids (meteoric **(A)** fluid equilibrated with the surroundings) which start mixing with early magmatic fluids (**(B)** end-member). Evidences of unmixing of the early magmatic fluid observed in quartz FIs as well as degassing explains the low δD in the residual fluid observed in ore minerals (**(C)** end-member in Maoping and **(D)** end-member in Piaotang). Through the crystallization and differentiation of the melt, magmatic fluids are becoming predominant, involving higher temperature, higher salinity and higher metal content associated with the crystallization of ore minerals.

At the Maoping deposit, another type of vein (banded quartz veins, stage IV) crystallizes during cooling under the same conditions. FIs in Qtz_{4-IV} do not provide any suspicion of any more changes in the system at that time. Ore components are still present in the fluid (**(D)** end-member) but do not reach precipitation conditions. As wolframite do not crystallize in the following stage but cassiterite does, the limiting factor could be the available amount of Fe and Mn leached from the host rocks.

5.3.2 Comparison to other deposits from the Nanling Range and worldwide

FI studies have been performed on eleven deposits from the Jiangxi and Hunan provinces from the Nanling Range (as described previously) and many deposits worldwide (e.g. Norohna et al., 1992; Luders et al., 2009; Ni et al., 2015). Aqueous inclusions are predominantly observed but early quartz FIs often show varying amounts of gases (mostly CO₂) as observed in our case (e.i. Norohna et al., 1992; Wei et al., 2012; Ni et al., 2015; Chicharro et al., 2016). In the literature, those FIs often show variable proportions of the vapor fraction that could indicate boiling (not observed in our case). This processes was however described as not decisive for W-Sn mineralization by demonstrating the effects of boiling on $\delta^{18}O$ isotopic composition at different temperatures (Wei et al., 2012, Wei et al., 2017) and was in fact, never observed in wolframite nor cassiterite.

Microthermometric data from the literature, as well as our study, define different conditions of trapping of gangue and W-Sn minerals (Wei et al., 2012; Ni et al., 2015) with higher trapping temperature and salinity associated with ore fluids. Here, we demonstrate for

the first time that these conditions and mixing process are maintained during post-mineralizing stages while tungsten is still present in the fluids. Our stable isotope study (oxygen and hydrogen) agrees with proposed origins of fluids: magmatic and meteoric (Beuchat et al., 2004; Carruzzo et al., 2004; Wei et al., 2012; Liu et al., 2002; Wang et al., 2012; Gong et al., 2015; Feng et al., 2012). The trace element content of FIs permitted us to give some complementary information on the meteoric end-member by showing an equilibration of the fluid with the surroundings prior to mixing and permitted to propose magma differentiation as a prerequisite process involved in the formation of W-Sn in this region. Such processes were already proposed in some few deposits (e.g. Audétat et al., 2000; Luders et al., 2009). Finally, all analytical methods raise the evidence of similarities between the Maoping and Piaotang deposits. Such similarities as well as similar timing of mineral phases, must involve regional fluid processes associated with this giant granitic province emplaced between 160 and 150 Ma.

6. Conclusion

Microthermometry, Raman spectrometry, LA-ICPMS and stable isotope (O and H) analysis of fluid inclusions from the Maoping and Piaotang deposits from the Nanling Range allow us to complement our knowledge on processes involved in W-Sn deposit of the richest region of the world:

- Quartz and ore-bearing minerals of early mineralizing stages indicate mixing processes between a magmatic fluid exsolution from the associated granite and a meteoric fluid that already interacted with the surrounding rocks. Post-mineralizing stages share these conditions without crystallizing any ore-bearing minerals.

- The timing of crystallization of ore-bearing minerals (wolframite and cassiterite) in early mineralization stages depends on the differentiation of the granite. Early fluids crystallizing quartz after the magmatic-hydrothermal transition will be more depleted in metals

compared to later fluids, exsolved from a melt enriched in incompatible elements as metals. Therefore, quartz crystallizing fluids do not reflect ore-bearing fluids.

- Both deposits show a second heat input postponing the mineralizing stage where other type of mineralizations occur, implying a second unknown event at the regional scale which was never described before. Tungsten is still present in the fluid at these stages but do not precipitate wolframite, implying other parameters than mixing (as usually proposed) involved in ore-bearing minerals crystallization.

- Such similarities between all mineralogical and fluid inclusion studies of W-Sn deposits from the same Nanling Range and timing, point to regional fluid processes involved during the emplacement of the giant associated granitic province between 160 and 150 Ma.

Finally, this study shows that the world-class deposits from the Nanling Range are complex polyphased deposits where differentiation of the magma source of ore minerals is the only decisive parameter of wolframite and cassiterite crystallization.

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Part 3 – Geochronology

Preamble

In the Nanling metallogenic province, most W-Sn deposits are associated to Jurassic-Cretaceous granitoids that are believed to be the source of the metals. The 160-150 Ma period appears to be the peak of this magmatic-related “W-Sn event”. A lot of dating approaches have been performed on these deposits, on the granites but also on greisen and minerals of the veins. However, taking the case of few deposits, the relation and timing between the granite and veins crystallization stays ambiguous. A recent model was recently published by Wang et al. (2016), indicating that these veins belong to a much later undefined event at ca. 130 Ma coevally to the crystallization of muscovite granites. Yet, very few direct dating have been performed on minerals from the veins. The major problem lies in the lack of adequate standards to perform *in situ* dating analysis of minerals such as wolframite (W), cassiterite (Sn) and columbo-tantalite (Nb-Ta). Fortunately, wolframite and columbo-tantalite are good candidates for ID-TIMS dating but cassiterite remains difficult to dissolve. However, some few articles are recently developing methods of cassiterite dating by LA-ICPMS (i.e. Che et al., 2015). Chapter 5 is dedicated to the application of multiple dating approaches (Ar-Ar on micas, U-Pb on wolframite and U-Pb on xenotimes). The aim of this chapter is to provide the first age on wolframite for the richest W-Sn province in the world and discuss the age of this “W-Sn” event. Chapter 6 is focused on analytical developments through a collaborative project dedicated to define new standards for *in situ* direct dating. This chapter is dedicated to columbo-tantalite, using ID-TIMS and SIMS techniques.

Chapter 5 - Multiple hydrothermal pulses in the Nanling metallogenic belt: Constraints from the W-Sn Maoping deposit (Jiangxi, China)

Article in preparation for Ore Geology Reviews

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Abstract

The Nanling metallogenic belt is the richest province worldwide in term of W-Sn resources. The mineralized veins as well as their associated granitic intrusions have been widely dated and give a large spectrum of ages within the Jurassic and Cretaceous periods. At this day, dating analysis performed on minerals from the veins are mainly focused on molybdenite, micas, hydrothermal zircons and quartz fluid inclusions. Few dating results were recently published on cassiterite crystals but no direct dating of wolframite remains available although recent fluid inclusion studies suggest different fluid circulations and processes responsible for the crystallization of gangue and ore minerals.

In this study, multiple dating methods including ^{40}Ar - ^{39}Ar on micas, U-Pb direct dating of wolframite and U-Pb-Th on xenotime, were performed on multiple stages of crystallization of the veins, the granite and the greisen. Results yield a continuum of ages reflecting perturbations of the isotopic systems by the circulations of multiple fluids. Four fluid circulations could be identified: (i) W-Sn mineralizing fluids at ca. 160 Ma, (ii) fluids associated to the greisenization at ca. 156 Ma, (iii) REE mineralizing fluids associated with late sulfides at ca. 152 Ma and, and (iv) REE- and Zr-rich fluids circulating at ca. 130 Ma.

This study highlights an early crystallization of ore tungsten minerals by the first direct measurements performed on wolframite in the Nanling Range and the necessity to complement studies on other deposits from this region where gangue minerals were not necessarily formed with the same processes nor at the same timing as ore minerals.

Keywords

Maoping - U-Pb dating - Ar-Ar dating – wolframite – xenotime - tungsten deposit - Nanling

1. Introduction

The Nanling Range is the world's richest metallogenic belt in term of tungsten resources (Wang et al., 2016). Yet, no direct dating of wolframite minerals exists at this day in this region.

In the Nanling range, fractional crystallization of granitic magmas is thought to have promoted ore-forming processes leading to ore minerals crystallization along fractures during and/or after the solidification of the granites (Mao et al., 2007; Xi et al., 2007; Pirajno, 2009; Zhou et al., 2010). Ore-forming processes include either two fluids (magmatic dominant and meteoric minor) mixing or a single fluid (magmatic) cooling, leading to the precipitation of ore minerals (Wei et al., 2012). Tungsten is believed to be transported by fluids exsolved from the granitic intrusion and gangue mineral dating analyses usually match the granite age, these minerals have been used as a proxy to determine the age of W-Sn mineralizations.

The widely accepted range of the main W-Sn mineralization in the Nanling Range is defined between 150 and 160 Ma. However, recent fluid inclusion studies performed on W-Sn deposits from the Nanling Range highlight different hydrothermal processes involved in gangue and ore minerals, raising the evidence of non-coeval crystallization and polyphase fluid circulations (Legros, submitted; Wei et al., 2012; Ni et al., 2015). These studies raise several questions: (i) can gangue mineral dating studies be used as a proxy to determine the age of ore minerals? and (ii) do such fluid circulations may induce perturbations in the isotopic systems of the studied minerals? Recent studies started to address this problematic in the case of the Piaotang and Xihuashan deposits and performed U-Pb measurements on cassiterite crystals from the veins (Bai et al., 2011; Zhang et al., 2017). These studies yield ages at ca. 160 Ma, interpreting an earlier stage of emplacement of the ore minerals.

Previous studies including detailed petrography, mineralogical studies of Fe-Li-micas and fluid inclusions in the Maoping deposit suggest that at least four types of fluid showing variable rates of mixing are involved (Legros et al., 2016; Legros et al., submitted). This study uses the Maoping deposit as a proxy to unravel the timing of multiple fluid circulations that could have also circulated in the Nanling Range. To address this matter, multiple dating methods were performed: U-Pb direct dating of wolframite to unravel the timing of the mineralization, ^{40}Ar - ^{39}Ar dating on several generations of Fe-Li-micas from the mineralizing and post-ore stages, granite and greisen, and U-Th-Pb dating of xenotime associated with rare-earth minerals and fluorite in late filling stages.

2. Geological setting and sampling

2.1 The Jiangxi metallogenic province

The southern Jiangxi W-Sn province is part of the Nanling metallogenic belt which hosts the main W reserves in China (90%, ca. a million tons: Wang et al., 2016). This belt is located in the Cathaysia Block, which is part of the South China Craton (SCC).

The SCC was assembled during late Jiangnan orogeny (1000-950 Ma) by the suturing between two terranes: the Yangtze Block to the north and the Cathaysia Block to the south (Charvet 2013 and references therein), separated by the major Jiangshan-Shaoxi fault zone (Figure 57). The Jiangnan orogeny is separate in two major phases: the accretionary Sibao orogeny and the Jinning event that inverted the great faults to create the Nanhua rift. The final age of the collision is still debated, comprised between 900 Ma and 800 Ma (e.g., Li et al. 2009, Hu and Zhou 2012, Yao et al. 2013). This period was decisive in the history of the SCC and emplaced most lineaments that reworked at later periods.

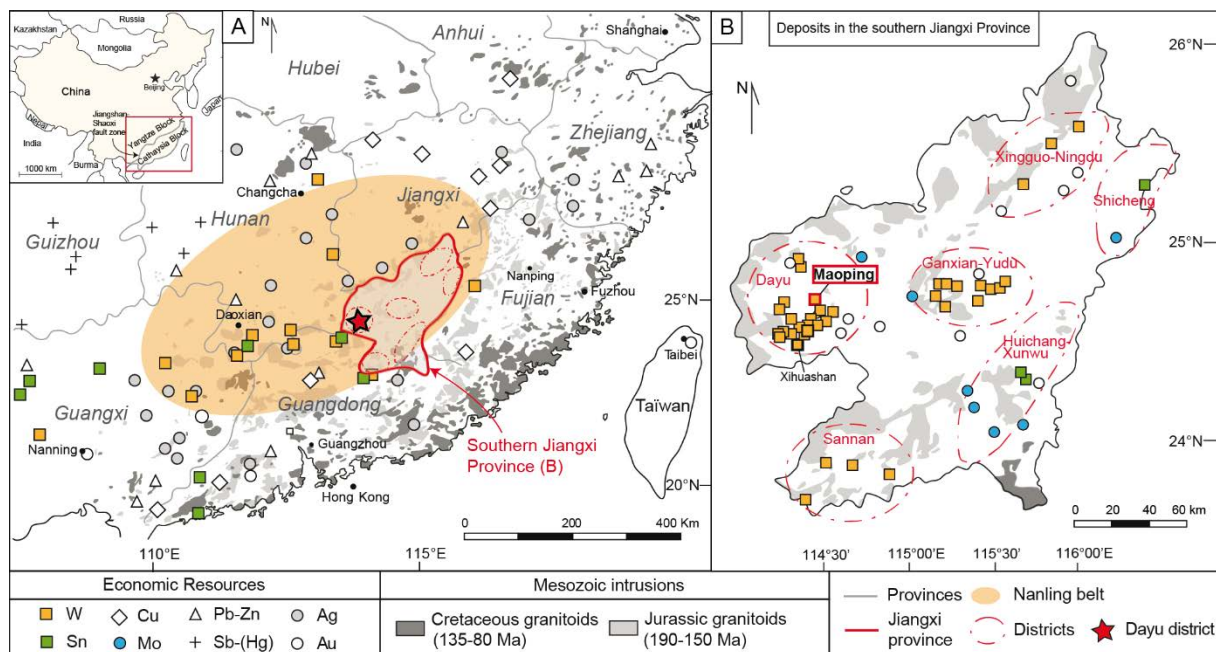


Figure 57. Ore mining districts and metal deposits of the Cathaysia Block (A) with zoom on the southern part of the Jiangxi Province (B), South-East China. The distribution of Jurassic (peraluminous dominant) and Cretaceous (peralkaline dominant) granitoids is shown in different grey tones. The Maoping deposit is located in the Dayu district in the south-west of the Jiangxi province. (A- Modified and compiled from Sun et al. (2012), Jiang et al. (2006), Mao et al. (2006), Wu et al. (1993) and Wang and Fan (1987). B- Modified after Huang et al., 2014 and Lecumberri-Sanchez et al., 2014).

Indeed, the SCC was progressively cratonized through a sequence of intra-continental events from the late Neoproterozoic (Nanhua Rift: Wang et al. 2006), to the Middle Paleozoic Kwangsi orogenesis (ca. 470 Ma to 400 Ma), the Early Mesozoic Indosinian events (ca. 270 to 200 Ma) and the Jurassic-Cretaceous Yanshanian events (ca. 200 to 65 Ma). In particular,

the Cathaysia Block was by far the most affected by the metacratonization and registered the most pervasive magmatic activity in the Yanshanian.

The Middle Palaeozoic Kwangsi orogeny (ca. 470 Ma to 400 Ma) is currently interpreted as resulting from the inversion of the NE-trending Nanhua Rift (e.g., Charvet et al., 2010, Charvet, 2013). The first intra-cratonic event yielded a high-grade belt of migmatites and granulites due to crustal thickening and HT metamorphism at ca. 450 Ma (e.g., Liu et al. 2010a). This episode was followed by basaltic underplating, dehydration melting in the lower crust, and the emplacement of large syn-kinematic S-type granitic bodies (more than 20,000 km²: Li et al. 2011a) at ca. 430-420 Ma (e.g., Wang et al. 2011, Xia et al. 2014).

At the end of the Trias (ca. 245 to 210 Ma; Indosinian), the suturing of the SCC with the North China Craton marks a second major intracratonic event, reworking the Kwangsi structures (e.g., Wang et al. 2001). This Indosinian event, is associated with HT metamorphism (metamorphic core complexes: Faure et al. 1996), and produced large volumes of granitic bodies (presently covering 14,300 km²). These bodies were emplaced in two stages: I-types (245-230 Ma), and S-types (220-210 Ma) (Shu et al. 2008 and references therein) associated with Nb-Ta granites in the Nanling Range (e.g. Dahutang deposit, Mao et al. 2013).

The Yanshanian event (ca. 200 to 65 Ma), is known to be at the origin of a giant granitic (and rhyolitic) province (Li et al., 2014). This period is associated with successive transpression and transtension settings along the same NE-SW lineaments that controlled the Kwangsi and Indosinian tectonics (Li et al., 2014, Liu et al., 2012, Shu et al., 2009). Four periods are commonly interpreted: the J₁ period from 200-165 Ma, the J₂₋₃ period between 165-150 Ma, the K₁ period from 150-110 Ma and the K₂ period between 110-65 Ma.

The J₁ period (200-165 Ma) starts with the progressive replacement of the tethysian tectonic settings by the Paleo-Pacific regime and the initiation of Basin and Range tectonics associated with the flat subduction of the Paleo-pacific plate under the SCC (Wang and Mo, 1995; Wang and Zhou, 2002; Li and Li, 2007). In the Nanling belt, E-W trending zones of S-

type granitoids are emplaced and believed to be issued from the Tethysian settings or the post-collisional Tethysian belt (Zhou et al., 2006; Shu et al., 2009). The subduction settings have led to the remobilization of intracontinental faults and magmatism. At ca. 190 Ma, the production of A-type granites was interpreted as originated from the slab break-off and evolved into anorogenic magmatism (Li and Li, 2007). Following the delamination of the slab, the settings changed from Tethysian compressional regime to Paleo-Pacific extension (Li and Li, 2007). During the J₁ period, a stage of large-scale magmatism was emplaced from 180 to 160 Ma, caused by the underplating of basaltic magmas.

During the J₂ and J₃ periods (165-150 Ma), a second stage of NE-trending magmatism is expressed and constrained by the Paleo-Pacific plate subduction settings (Wang and Shu, 2012). The magmatism is characterized by I-type granites associated with A-type granites and rift-type syenite, issued from the slab rollback (Li and Li, 2007). W-Sn deposits in the Cathaysia are believed to be associated with this 160-150 Ma event, although a recent study challenged this hypothesis (Wang et al., 2016). Three parallel granitic belts associated with W-Sn deposits are formed in the E-W trending Nanling range during this period (Zhao et al. 2017).

During the K₁ period (150-110 Ma), intracontinental deformation and metamorphism associated with the subduction introduced a wide fold-and-thrust belt (Lin et al., 2008; Zhang et al., 2008; Xu et al., 2009). From 140 Ma, back-arc extensional settings are emplaced in the SCC and induced partial melting of the upper slab, crustal thinning and basalt underplating in association with mixed crust-mantle magmas (Wang and Zhou, 2002; Shu and Zhou, 2002; Zhou and Li, 2000; Zhou et al., 2006a). From 136 to 118 Ma, the SCC is under extensional settings, followed by tectonic inversion from 117 to 108 Ma and alternating again at ca. 107 Ma (Li et al., 2014).

The K₂ period (110-65 Ma) starts with an uplift of the coastal areas due to the collision of the SCC with the Philippine plate (Charvet et al., 1985, 1999; Maruyama et al., 1997; Shu et al., 2004, 2009). The Pacific Plate then started to rotate eastward and generated extensional settings, introducing NE-NNE basins associated to A-type granites and bimodal volcanism

(Zhou and Li, 2000; Shu et al., 2009). The slab started to migrate from the SCC and granitic magmatism ceased at ca. 90 Ma (Wang and Zhou, 2002).

2.2 Sampling and description of the Maoping deposit

The Maoping deposit is located in the Dayu district of the Southern Jiangxi Province, where numerous major tungsten deposits are exploited, e.g., Xihuashan (Giuliani 1985, Guo et al., 2012). The Maoping deposit is estimated to 63 kt WO_3 and 15 kt Sn with average grades of 0.93% and 0.3% respectively (Feng et al., 2011a).

The Maoping deposit consists of a buried peraluminous granite and a supposed hidden peralkaline granite (Legros et al., 2016) with W-Sn-bearing quartz veins hosted in metamorphosed Cambrian sandstones and schists. The granite hosts in its apical parts greisen-type disseminate W-Sn ore bodies (Figure 58). The veins are 5 cm to several meters thick and up to 400m long and are shallow dipping (Legros et al., 2016).

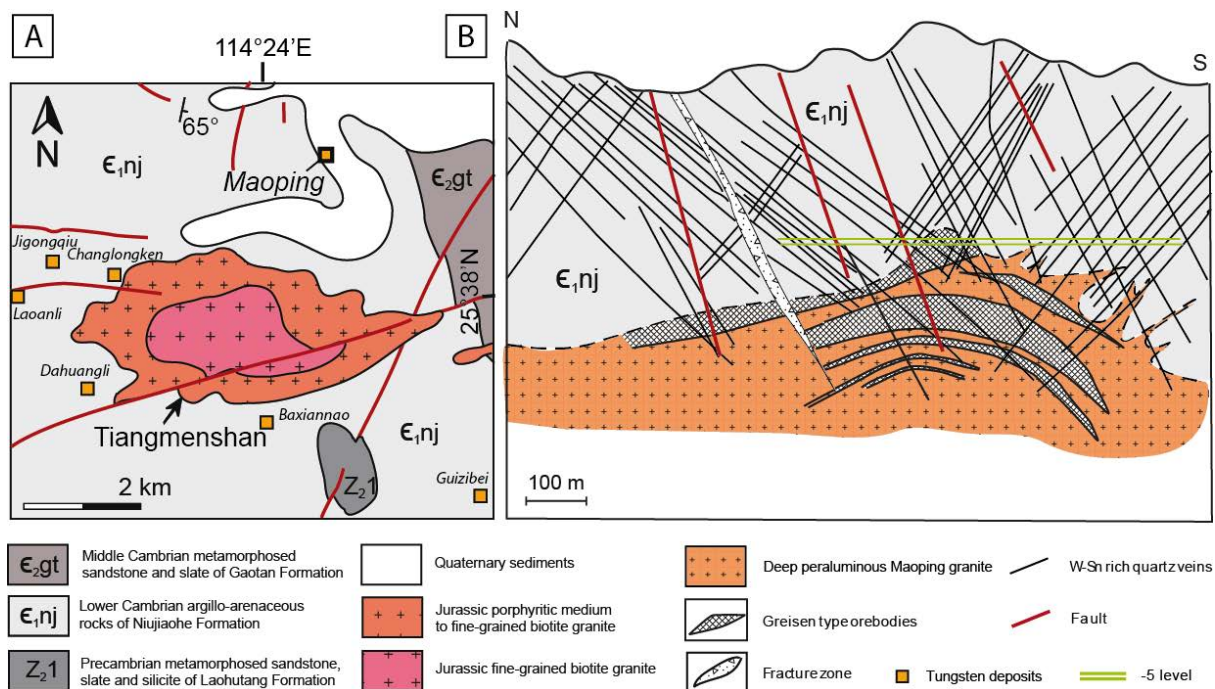


Figure 58. Location (A) and cross-section (B) of the Maoping W-Sn deposit, showing in particular, the relationships between the deep granite, Lower Cambrian host rocks, greisenized granite and W-Sn rich quartz veins, as well as the sampling zone (-5 level, in green) (modified after Feng et al., 2011b).

According to Legros et al. (2016), four types of veins (I to IV) and seven paragenetic stages (I to VII) are identified in the Maoping deposit based on cross-cutting relationships

observed in the field and petrographic observations (Figure 59 and Figure 60). In the following descriptions, minerals refer to (i) their IMA abbreviation, (ii) their generation number according to the paragenesis (1 to 5) and (iii) their paragenetic stages (I to VII).

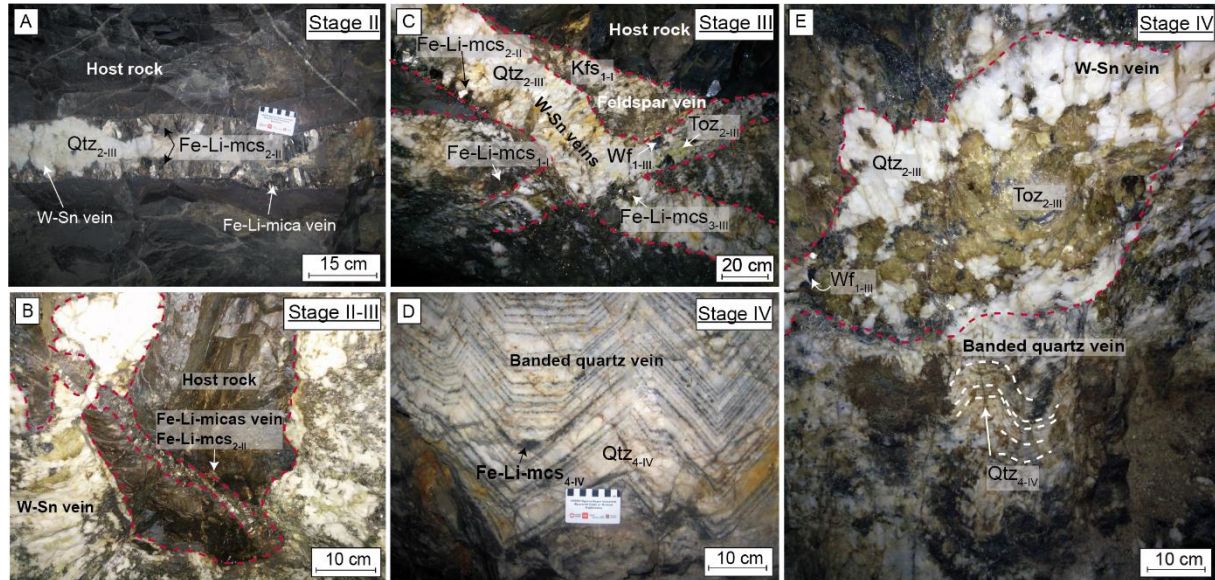


Figure 59. Relationship between vein types and paragenetic stages at the Maoping deposit. (A) Fe-Li-mica veins reopened and filled with Stage III quartz. (B) W-Sn vein at Stage III, cross-cutting Fe-Li-micas vein. (C) W-Sn vein cross-cutting the Feldspar vein. (D) Banded quartz vein showing the centimeter-scale banding of quartz and Fe-Li-micas at Stage IV. (E) Crystallization of the Banded quartz vein at Stage IV, invading a previous W-Sn vein. Abbreviations accorded to IMA, Qtz: Quartz, Fe-Li-mcs: Fe-Li-micas, Toz: Topaz, Wf: Wolframite.

The first stage (*Stage I*) consists of the early formed feldspar veins that mainly contain centimetric-size microcline, quartz and Fe-Li-micas.

Stage II corresponds to the emplacement of the Fe-Li-mica veins (Figure 59A). These veins crystallize Fe-Li-micas only, with centimetric euhedral crystals organized perpendicularly to the vein edges (Fe-Li-mcs_{2-II}).

Stage III is associated with the emplacement of the ore-bearing W-Sn veins (Figure 59C). The Stage III minerals are quartz (Qtz_{2,3-III}), wolframite (Wf_{1-III}), cassiterite (Cst_{1-III}), topaz (Toz_{2-III}), Fe-Li-micas (Fe-Li-mcs_{3-III}), molybdenite (Mlb_{1-III}), Fe-Li-muscovite (Fe-Li-ms_{1-III}) and, Fe-Mn hydroxides, crystallizing in that order. Quartz and topaz crystallize as geodic crystals. They usually represent the majority of the vein filling. In the cavities, wolframite and cassiterite crystallize as euhedral and clearly zoned crystals (no chemical variation has been associated

with that zonation). Wolframite and cassiterite are usually associated with posterior centimetric Fe-Li-mcs_{3-III} at the center of the veins.

	Stage I: Feldspar veins	Stage II: Fe-Li mica veins	Stage III: W-Sn veins (W-Sn-Li-Mo)		Stage IV: Banded quartz veins (Nb-Ta-Zr-REE)	Stage V	Stage VI: Dissolution (F-REE)	Stage VII (Bi)
K-feldspar	1							
Quartz	1		2 3		4 2	5		
Fe-Li-micas	1	2		3	4			
Albite	1				2			
Topaze	1		2		3		recrystallization (4)	
Wolframite			1		Corrosion		overgrowth	
Graphite			1					
Cassiterite			1 overgrowth		2			
Molybdenite				1				
Fe-Li-muscovite				1	2			
Fe-Mn hydroxides				1				
Zircon					1			
Colombo-tantalite					1			
Emplectite					1			
Li-muscovite	1					2		
Sphalerite						1		
Chalcopyrite						1		
Stannite							1	
Fluorite							1	2
Kaolinite							1	
REE phosphates								1
REE fluorides								1
REE carbonates								1
Bismuthinite								1
Native bismuth								1
Pyrite								1

Figure 60. Paragenetic sequence of the Maoping W-Sn deposit based on observations and sampling done in mine gallery at level -5 (i.e. -200 m). Numbers from 1 to 5 represent the generation of the mineral in the overall paragenetic sequence. Stage III has been separated by a plastic deformation event into IIIa and IIIb. See text and previous figures for details about different stages, mineral abbreviations and notations.

Stage IV corresponds to the emplacement of the Banded quartz veins (Figure 59D). Four generations of undeformed quartz are distinguished among this vein as a sequential crystallization. The banding is underlined by zoned Fe-Li-mcs_{4-IV}, among other minerals.

Stages V to VII are late veins with mostly fluorite, REE minerals (e.g. xenotime and monazite) and sulfides (e.g. sphalerite, chalcopyrite, bismuthinite). Stage VI is a late stage

composed of cavities-filling minerals within the W-Sn and Banded quartz veins. Stage VI minerals consist of a succession of fluorite (Fl_{1-VI}), kaolinite (Kln_{1-VI}) and a late porous fluorite (Fl_{2-VI}). The first generation of Fl_{1-VI} is euhedral and inclusion-free while the second (Fl_{2-VI}), is anhedral and zoned. This zonation displays many REE minerals inclusions as phosphates (i.e. xenotime Xnt_{1-VI} and monazite Mnz_{1-VI}), fluorides and carbonates.

The granite emplacement was dated at 151.8 ± 2.9 Ma by SHRIMP U-Pb on zircon (Feng et al., 2011b), whereas the greisen and the tungsten-bearing veins yielded Re-Os molybdenite ages at 155.3 ± 2.8 Ma and 150.2 ± 2.8 Ma, respectively, the latter have been interpreted as the emplacement age of the W mineralization (Feng et al., 2011a).

3. Analytical methods

Petrographic data were obtained from observations of polished thin-sections using conventional transmitted and reflected light microscopy and a HITACHI FEG S4800 scanning electron microscope (SEM) equipped with an energy dispersive spectrometer (EDS), using a Si(Li) semi-conductor detector, at the GeoRessources laboratory (Nancy, France). Cathodoluminescence (CL) was also performed on thin-sections, using a CITL cold cathode instrument CL8200 Mk4 (15 kV and 400 mA).

3.1 EMPA analysis

Electron microprobe analyses (EMPA) of Nb, W, Mn, Fe and Ta were performed on wolframite samples and P, Ca, Y, La, Ce, Nd, Sm, Gd, Pr, Dy, Er, Yb on xenotime samples using a CAMECA SX100 instrument equipped with a wavelength dispersive spectrometer (WDS) at the GeoRessources laboratory (Nancy, France). Calibration was performed using natural and synthetic oxides and silicates (olivine, $MnTiO_3$, scheelite, Fe_2O_3 , $LiTaO_3$ and $LiNbO_3$, monazite). A current of 20 nA and an accelerating voltage of 15 kV was used with a counting time of 10 s per element. Total Fe is presented as FeO.

3.2 LA-ICPMS analysis

Minor and trace elements (^7Li , ^{24}Mg , ^{45}Sc , ^{47}Ti , ^{51}V , ^{55}Mn , ^{57}Fe , ^{59}Co , ^{60}Ni , ^{63}Cu , ^{66}Zn , ^{69}Ga , ^{72}Ge , ^{85}Rb , ^{88}Sr , ^{89}Y , ^{90}Zr , ^{93}Nb , ^{95}Mo , ^{111}Cd , ^{115}In , ^{118}Sn , ^{139}La , ^{140}Ce , ^{141}Pr , ^{146}Nd , ^{147}Sm , ^{153}Eu , ^{157}Gd , ^{159}Tb , ^{163}Dy , ^{165}Ho , ^{166}Er , ^{169}Tm , ^{172}Yb , ^{175}Lu , ^{178}Hf , ^{181}Ta , ^{208}Pb , ^{209}Bi , ^{232}Th , ^{238}U) were measured in wolframites using a LA-ICPMS instrumental setup similar to that described in Leisen et al. (2012) and Lach et al. (2013), with a GeoLas excimer laser (ArF, 193 nm, Microlas, Göttingen, Germany) (Günther et al., 1997) and an Agilent 7500c quadrupole ICP-MS at the GeoRessources laboratory (Nancy, France). The laser beam was focused onto the sample within a 24.5 cm³ cylindrical ablation cell using a Schwarzschild reflective objective (magnification $\times 25$; numerical aperture 0.4) mounted on an optical microscope (Olympus BX41) equipped with a X–Y motorized stage and a CCD camera. Tungsten (determined by EPMA) was chosen as an internal standard as it is abundant in both the samples and the external standards and can be accurately quantified by EMPA. NIST610 (values from Jochum et al., 2011) was chosen as the external standard (accuracy verified with NIST612) (Longerich et al., 1990). The following parameters were used for the LA-ICPMS analyses: a fluence of 6J/cm² and a laser shot frequency of 5 Hz; He = 0.5 L.min⁻¹ as a carrier gas, mixed with Ar = 1.0 L.min⁻¹ via a cyclone mixer prior to entering the ICP torch. Ablation lines of 60 μm diameter and 200 μm length were analysed at a 2 $\mu\text{m/s}$ speed. Each isotope was acquired during a dwell time of 10 ms. Data was processed using the Iolite software (Paton et al., 2011).

3.3 $^{40}\text{Ar}/^{39}\text{Ar}$ Fe-Li-micas dating

$^{40}\text{Ar}/^{39}\text{Ar}$ analytical work was performed at the University of Manitoba (Canada) using a multi-collector Thermo Fisher Scientific ARGUS VI mass spectrometer, linked to a stainless steel Thermo Fisher Scientific extraction/purification line, Photon Machines (55 W) Fusions 10.6 CO₂ laser, and Photon Machines (Analyte Excite) 193 nm laser. Argon isotopes (from mass 40 to 37) were measured using Faraday detectors with low noise $1 \times 10^{12} \Omega$ resistors and mass 36 was measured using a compact discrete dynode (CDD) detector. All specimens (samples and standard) were irradiated in the Cadmium-lined, in-core CLICIT facility of the TRIGA reactor at the Oregon State University (USA). Standard used was the GA1550 biotite (Spell

and McDougall, 2003). The samples selected for the $^{40}\text{Ar}/^{39}\text{Ar}$ dating were previously investigated by optical microscopy and SEM, in order to spot homogeneous areas within the analyzed minerals (mica or adularia) devoid of alteration, micro-inclusions or internal zoning. Ablation pits were excavated to an estimated depth of 50 μm . Five argon isotopes were measured simultaneously over a period of 6 minutes. Measured isotope abundances were corrected for extraction-line blanks, which were determined before every sample analysis. Line blanks in both the Excimer and CO_2 system averaged ~ 3 fA for mass 40 and ~ 0.013 fA for mass 36. A value of 295.5 was used for the atmospheric $^{40}\text{Ar}/^{36}\text{Ar}$ ratio (Steiger and Jäger, 1977) for the purposes of routine measurement of mass spectrometer discrimination using air aliquots, and correction for atmospheric argon in the $^{40}\text{Ar}/^{39}\text{Ar}$ age calculation. Corrections are made for neutron-induced ^{40}Ar from potassium, ^{39}Ar and ^{36}Ar from calcium, and ^{36}Ar from chlorine (Roddick, 1983; Renne et al., 1998; Renne and Norman, 2001). Detailed in Appendix A.

3.4 U-Pb wolframite ID-TIMS dating

Wolframite samples U-Pb ratios were analyzed at the GFZ German Research Centre for Geosciences (Potsdam), following the analytical procedure described in detail in Romer and Lüders (2006). Samples of a concentrate and centimetric crystals from the veins were selected for analyses. Each sample was part of a distinct batch of analyses. Samples were crushed and grains selected under binocular microscope showed no alteration, fractures or overgrowths. Any surface contamination was removed by washing the samples in warm H_2O and acetone before reselecting the cleaner grains. A ^{205}Pb - ^{235}U tracer was added to the sample and an excess of 40% concentrated HF was used to dissolve all the samples on a heated plate overnight. Pb and U were separated and purified using ion exchange chemistry (Romer et al., 2005) and loaded on Re single filaments with H_3PO_4 and silica gel. Isotopic ratios of Pb and U were measured separately on a Triton thermal ionization mass spectrometer (TIMS) using Faraday collectors or an ion counter. Data reduction procedures are described in Schmid et

al. (2003). The concordia plot and age calculations were carried out using the Isoplot/Ex add-in for Excel (Ludwig 2008).

3.5 U-Th-Pb xenotime LA-ICPMS dating

Ablation of xenotime was performed at the Université de Rennes (Géosciences Rennes, France) using a ESI NWR193UC Excimer laser system coupled to an Agilent 7700x quadrupole ICPMS. A spot diameter of 25µm was chosen with a repetition rates of 3Hz. The ablated material was transported to the mass spectrometer in He, before mixing with N₂ (Paquette et al., 2014) and Ar and introduced in the ICP source of an Agilent 7700x quadrupole ICP-MS equipped with a dual pumping system to enhance sensitivity. NIST SRM 612 reference glass was used for mass calibration at the beginning and end of each batch. ²⁰⁴Pb, ²⁰⁶Pb, ²⁰⁷Pb, ²⁰⁸Pb, ²³²Th and ²³⁸U signals were analyzed. For each analyses, 20s of background was integrated, followed by 60s of laser-firing acquisition. Each analysis was separated by a 10s delay to wash out. The Weinsberg xenotime (Klötzli et al. 2007; 317.3±2.0 Ma) standard was used to correct for Pb/U and Pb/Th laser-induced elemental fractionation and instrumental mass discrimination. More information on the dating protocol can be found in Boutin et al. (2016). Data reduction was carried out with the GLITTER software package (Van Achterbergh et al. 2001; Jackson et al. 2004). The Wetherill concordia plot and age calculations were carried out using the Isoplot/Ex add-in for Excel (Ludwig 2008). Uncertainties are reported at 1σ.

4. Sample descriptions

Thirty-five samples were collected at the -5 underground level of the Maoping mine (i.e. approximately 200 m below current surface). This sampling yielded to a representative set of samples from the mineralized, greisen and granite.

Among the seven paragenetic stages identified at the Maoping deposit (Legros et al., 2016; Figure 60), four of them display a mineralogical diversity permitting multiple dating: the Stage II Fe-Li-micas veins, the Stage III W-Sn veins, the Stage IV banded quartz veins and

the Stage VI late filling stage (Figure 60). Three types of minerals have been selected for multiple dating: Fe-Li-micas in order to compare ages of the granite, greisen and three distinct hydrothermal stages, wolframite to date the tungsten mineralization and xenotime to evaluate the age of crystallization of the late filling stages.

4.1 Fe-Li micas grains

Five samples containing Fe-Li-micas were selected from the greisen, granite and three paragenetic stages (II, III and IV; Figure 61) for ^{40}Ar - ^{39}Ar dating. For detailed major and trace element content, refer to Legros et al., (2016).

The Fe-Li-micas_{2-II} and the Fe-Li-micas_{3-III} are centimetric palisadic crystals from the W-Sn veins crystallizing respectively on stage II and III. The Fe-Li-micas_{2-II} were sampled at the edge of the vein while the Fe-Li-micas_{3-III} are observed at the center of the vein. Paragenetically speaking, the Fe-Li-micas_{3-III} only are associated with the ore-bearing minerals (wolframite and cassiterite). Both types of crystals show some alteration into Fe-Li-ms within the cleavages, sometimes associated with Fe-Mn hydroxides (Figure 61).

The Fe-Li-micas_{4-IV} correspond to millimetric crystals underlining the centimetric quartz zonation of the banded quartz veins of Stage IV. This vein does not present particular enrichment in ore-bearing minerals. These crystals are oscillatory zoned and contain rare inclusions of zircon and columbo-tantalite (Figure 61). The fine oscillatory zoning alternates between trioctahedral and dioctahedral compositions while the larger rim is of homogeneous zinnwalditic composition.

The greisen Fe-Li-micas are millimetric crystals that are the result of the alteration of the granite. Most of the selected grains display alteration on the rim and in fractures (transformation from trioctahedral to dioctahedral structure). Micas show symplectic texture with quartz grains and present some rare inclusions of late fluorite-monazite and uranothorite-monazite assemblages (Figure 61).

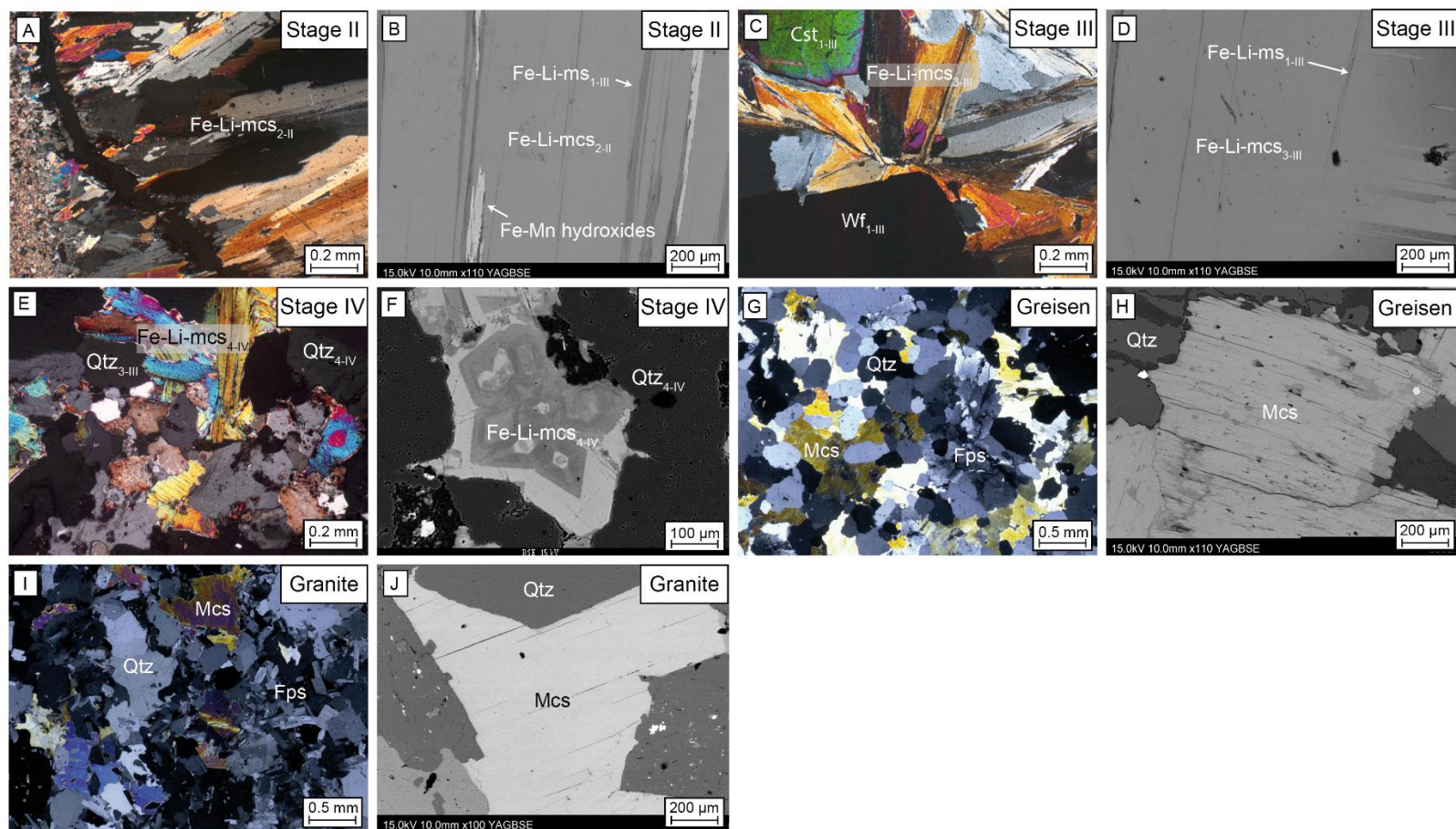


Figure 61. Micas textures and associations from the granite, greisen and Stages II to IV. (A) Stage II feather-like centimetric Fe-Li-mcs_{2-II} at contact to host rock (cross polars). (B) Back-scattered electron (BSE) image of homogeneous Stage II mica with Fe-Li-ms_{1-III} and Fe-Mn hydroxides alteration along the cleavage planes. (C) Stage III Fe-Li-mcs_{3-III} associated with Wf_{1-III} and Cst_{1-III} (cross polars). (D) BSE image of Stage III homogeneous micas with few Fe-Li-ms_{1-III} alteration along the cleavage planes. (E) Banded accumulation zone in Stage IV veins with zoned Fe-Li-micas_{4-IV} and Qtz_{4-IV} (cross-polars). (F) Zoning and rhythmic banding of Stage IV the Fe-Li-mcs_{4-IV}. (G) Deformed quartz, corroded mica and euhedral feldspar from the granite (cross polars). (H) BSE image of homogeneous mica from the granite. (I) Quartz sub-grains, corroded mica and altered feldspar from the greisen (cross polars). (J) BSE image of homogeneous granite micas with rare alteration along edges and fractures. Abbreviations from IMA, Cst: Cassiterite, Mcs: micas, Qtz: quartz, Ms: muscovite, Toz: topaz, Wf: Wolframite.

Finally, the Fe-Li-micas from the granite are millimetric homogeneous crystals. These crystals are devoid of alteration (Figure 61), usually form symplectic associations with quartz and are developed at the expense of feldspars and sometimes topaz. Some rare inclusions of fluorite rimmed by yttrium-carbonate aggregates are observable.

4.2 Wolframite grains

The wolframite are blood red centimetric samples (under transmitted light microscopy), porous, fractured and plastically deformed. Most crystals are characterized by highly developed porosity that could correspond to either fluid inclusions or micro-cracks.

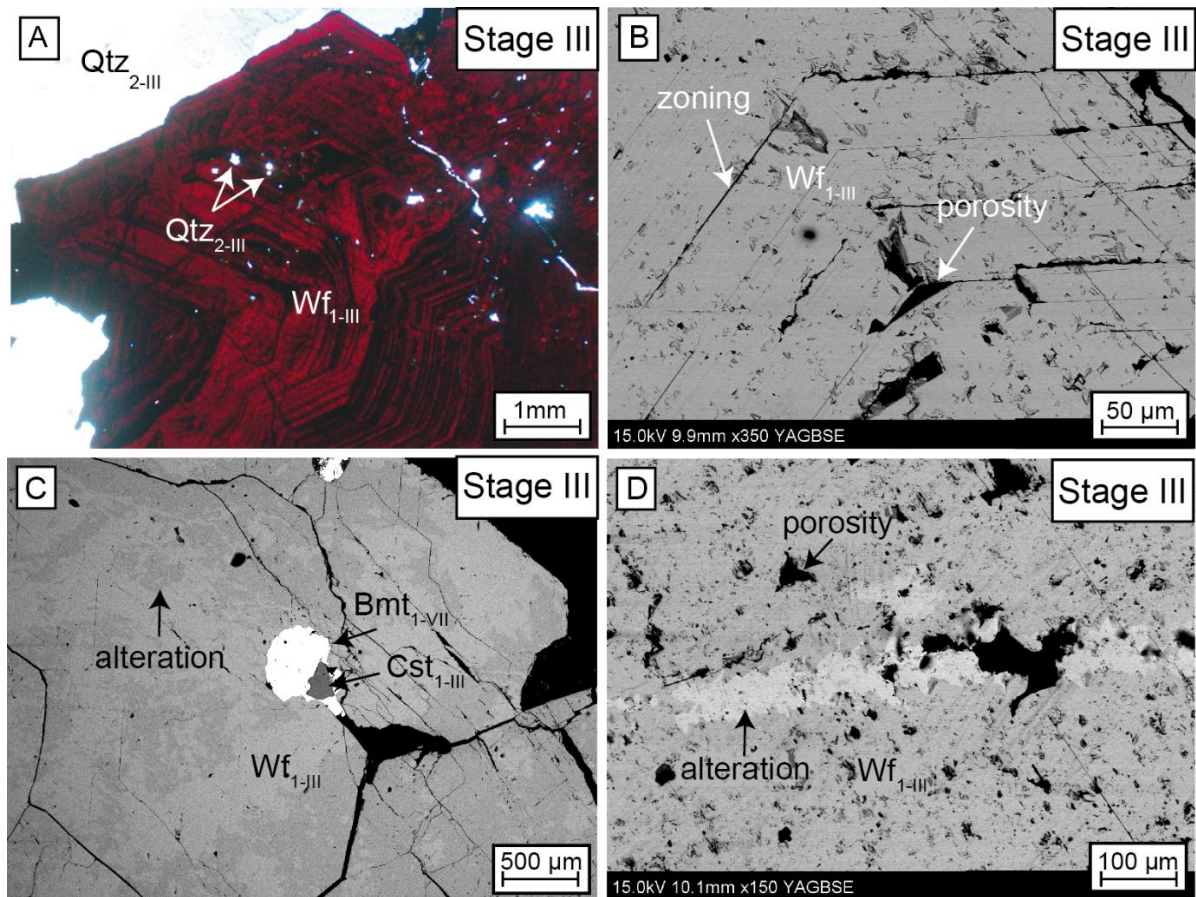


Figure 62. Stage III wolframite crystals from the Maoping deposit. (A) Optical image showing complex crystallographic zoning of wolframite associated with main Qtz_{2-III}. There are rare quartz-filled cavities (arrows) in wolframite. (B) BSE image highlighting the crystallographic zoning and absence of alteration. (C) Patchy alteration increasing the Mn content and inclusions of bismuthinite and cassiterite within a wolframite crystal (BSE image). (D) High porosity and line of alteration within a wolframite crystal (BSE image). Abbreviations from IMA, Bmt: bismuthinite, Cst: cassiterite, Qtz: quartz, Wf: wolframite.

Crystals show fine oscillatory zoning under the microscope with multiple overgrowths that could not be chemical distinguished (Figure 62). Within wolframite crystals, some inclusions of quartz, cassiterite and sulfides can be observed (e.g. bismuthinite). Some rare patchy zoning is observed and associated with the alteration of the grains. This alteration increases the Mn/Fe ratio of the crystals (2 wt. % more Mn; Table 9).

Table 9. EMPA analyses of wolframite samples. Values preceded by the "<" symbol are below the detection limit.

Wt. %	WO ₃	MnO	FeO	Nb ₂ O ₅	Ta ₂ O ₅	Total
MP-1	76.0	17.6	7.4	0.6	<0.5	101.5
MP-2	76.8	17.1	7.1	0.5	<0.5	101.5
MP-3	75.2	17.6	7.1	0.7	1.1	101.7
MP-4	76.1	17.8	7.2	0.5	0.6	102.1
MP-5	75.4	18.0	7.1	0.8	<0.5	101.3
MP-6	76.1	18.1	6.6	<0.5	<0.5	100.8
MP-7	73.7	17.6	6.9	1.0	0.5	99.6
MP-8	76.7	17.9	7.0	0.5	<0.5	102.1
MP-9	75.6	17.8	7.0	<0.5	<0.5	100.4
MP-10	75.5	17.4	6.8	0.5	<0.5	100.3
MP-11	75.4	17.9	6.8	0.6	<0.5	100.6
MP-12	75.9	17.7	6.9	0.5	<0.5	100.9
MP-13	76.7	16.3	7.5	0.9	<0.5	101.4
MP-14	75.7	17.5	6.8	0.6	0.8	101.4
MP-15	74.7	17.0	7.2	1.1	<0.5	99.9
MP-16	77.0	17.5	7.3	<0.5	<0.5	101.8
MP-17	77.9	17.4	7.2	<0.5	<0.5	102.4
MP-18	77.6	16.8	7.2	<0.5	<0.5	101.5
MP-19	74.0	17.2	7.4	1.1	<0.5	99.7
MP-20	75.7	17.5	7.0	0.7	<0.5	100.9

Wolframite crystals are globally homogeneous in major elements with Mn/(Mn+Fe) varying from 68% to 73% depending on the rate of alteration (Table 9). Crystals are enriched in Sc (135 to 367 ppm), Zn (122 to 178 ppm), Y (59 to 192 ppm), Zr (27 to 132 ppm), Nb (1900 to 4100 ppm), Mo (46 to 157 ppm), Sn (46 to 75 ppm), Ta (637 to 1100 ppm) and HREE (Σ from 240 to 730 ppm) (Appendix B). These variations in trace elements could not be correlated to the altered zones of the crystals.

4.3 Xenotime grains

Two generations of fluorite are observed during stage VI. Both generations show similar chemical compositions and zoning. The main difference resides in the occurrence of inclusions within the banding zones. Anhedral to subhedral grains from 50 to 400 μm large, hydrothermal xenotime crystals occur on yttrium-rich growth surfaces of $\text{Fl}_{2\text{-VI}}$ in association with porosity, kaolinite and other REE minerals (Legros et al., 2016). No corrosion or dissolution features have been observed (Figure 63). Some grains show geometrical zoning, but no chemical variations or alterations (Table 10; Appendix C).

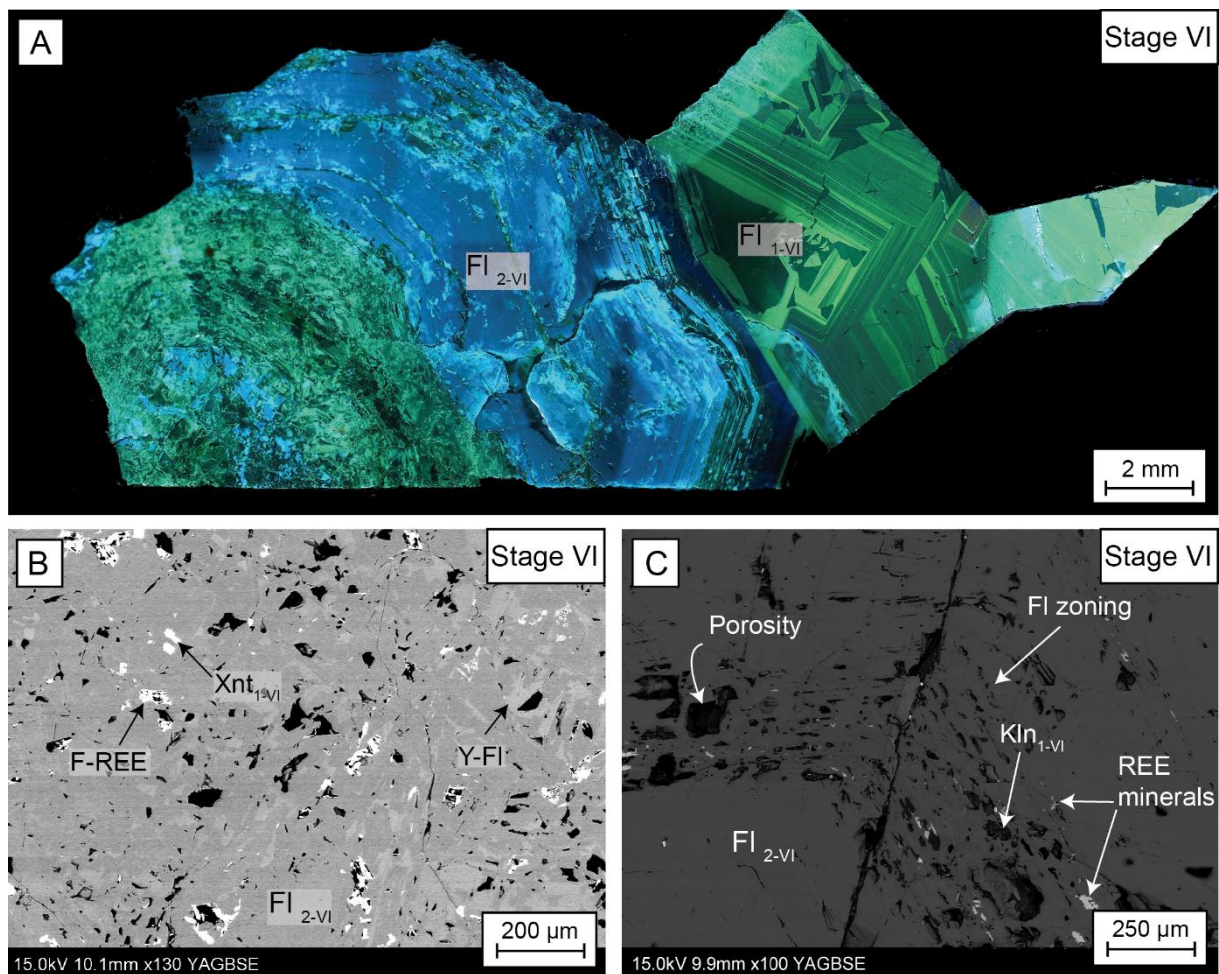


Figure 63. Organisation and mineral assemblages associated to xenotimes in fluorite crystals. (A) Association of the two generations of fluorite (CL). (B) SEM image of the core of $\text{Fl}_{2\text{-VI}}$ with heterogeneous enrichment in yttrium and filling of REE-fluorides and xenotimes. (C) SEM image of the banding zones observed in $\text{Fl}_{2\text{-VI}}$ where associations of high porosity, kaolinite and REE minerals are observed. Abbreviations from IMA, Fl: fluorite, F-REE: REE-fluoride, Kln: kaolinite, Xnt: xenotime, Y-Fl: yttrium-fluorite.

Xenotime grains have variable amounts of CaO (0.1 to 13.5 wt.%), Y₂O₃ (30.0 to 44 wt.%), Gd₂O₃ (2.5 to 6.3 wt.%), Dy₂O₃ (4.5 to 9.7 wt.%), Er₂O₃ (3.5 to 5.2 wt.%) and Yb₂O₃ (4.0 to 11.4 wt.%). These chemical variations are not correlated to the shape of the xenotime grains, their organization within the fluorite zoning nor the zoning of the grains. All EMPA analyses were performed on grains that were used for age calculation and when possible, in the core and rim of the grains but no chemical distinction is observed.

5. Results

5.1 ⁴⁰Ar-³⁹Ar Fe-Li-micas dating

Five samples containing Fe-Li-micas were selected from the greisen, granite and three paragenetic stages (II, III and IV; Figure 61) for ⁴⁰Ar-³⁹Ar dating, as described previously. ⁴⁰Ar-³⁹Ar dating results are shown on Figure 64. Associated errors are quoted at the 1σ level (Table 11).

The Fe-Li-micas_{2-II} and the Fe-Li-micas_{3-III} are centimetric crystals sampled from the W-Sn veins. The Fe-Li-micas_{2-II} were sampled at the edge of the vein while the Fe-Li-micas_{3-III} were sampled at the center of the vein.

The Fe-Li-micas_{2-II} calculated apparent ages range from 150.4 to 160.5 Ma. Among six measurements, two groups of ages are observable on Figure 64 (including errors): (i) from 146 to 157 Ma and (ii) from 155 to 163 Ma. Integration of all data (n=6) yields an age of 157±2 Ma (1σ, MSWD=1.33; Figure 64). No petrological evidences seem to be related to the large range of ages from 146 to 163 Ma. Laser spots digging and reaching alteration zones could be an explanation.

The Fe-Li-micas_{3-III} calculated apparent ages range from 151.9 to 166.5 Ma. All calculated ages overlap within errors in a range from 145 to 163 Ma similarly to Fe-Li-micas_{2-II} calculated ages at the exception of one measurement which yields an older age. The latter could reflect the occurrence of a mineral inclusions that could not be defined optically. Integration of the data (n=5) yields an age of 155±2 Ma (1σ, MSWD=0.74; Figure 64).

Table 10. EMPA analyses of xenotime samples. Values preceded by the "<" symbol are below the detection limit.

	34-1	34-a	36-a	36-b	24-a	24-c	24-e	16-b-c	16-b-b	18-a	7-a	2-b	8-c	8-b	13-a	3-b	5	18-c	18-b	23	28-b
P₂O₅	36.07	36.84	35.84	37.36	36.82	36.29	34.71	37.09	32.27	32.92	30.55	31.27	35.66	28.07	35.46	35.29	35.42	33.94	27.09	31.97	30.06
CaO	0.35	0.10	0.36	<0.1	0.69	0.66	0.19	4.46	0.60	0.36	0.26	0.35	0.31	10.14	0.46	0.96	0.25	0.59	13.54	10.90	1.80
Y₂O₃	42.06	43.60	41.24	42.98	37.32	39.88	40.21	35.45	41.34	39.48	43.96	42.85	39.67	34.90	34.78	37.41	39.04	38.25	32.57	30.01	37.17
La₂O₃	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Ce₂O₃	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
Nd₂O₃	0.68	0.59	0.66	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	<0.4	0.45	<0.4	<0.4	<0.4	0.72	0.50	<0.4
Sm₂O₃	1.64	0.76	1.45	0.84	0.81	1.54	0.55	1.99	0.56	0.80	<0.5	<0.5	0.94	0.54	1.68	0.92	0.89	<0.5	<0.5	<0.5	<0.5
Gd₂O₃	5.85	4.62	5.16	4.32	5.25	5.09	4.07	5.87	4.12	6.13	3.90	3.55	4.75	4.29	5.63	5.65	5.58	6.36	4.33	4.74	2.59
Pr₂O₃	0.32	<0.2	<0.2	0.23	0.68	0.27	<0.2	0.63	0.35	0.55	0.33	<0.2	0.33	<0.2	0.38	0.45	0.20	0.26	0.38	0.36	0.52
Dy₂O₃	7.08	5.81	7.01	6.26	9.34	7.98	7.78	8.22	7.82	8.49	6.73	6.63	8.20	6.68	8.68	9.25	9.58	9.68	7.48	7.93	4.50
Er₂O₃	3.46	3.72	3.97	4.02	4.58	3.99	4.48	3.89	5.11	4.02	4.61	3.89	3.76	4.36	3.79	4.56	4.09	4.02	3.68	3.51	5.18
Yb₂O₃	4.03	4.90	4.03	4.18	5.04	4.87	5.28	4.01	5.11	4.08	5.27	5.77	4.45	5.46	4.63	5.42	4.91	4.73	4.51	4.10	11.44
Total	101.5	100.9	99.7	100.2	100.5	100.6	97.3	101.6	97.3	96.8	95.6	94.3	98.1	94.4	95.9	99.9	100.0	97.8	94.3	94.0	93.3

The Fe-Li-micas_{4-IV} correspond to millimetric crystals underlining the centimetric quartz zonation of the banded quartz veins of Stage IV. The Fe-Li-micas_{4-IV} zoning is too thin to place spots along the growth zonation (Figure 61F). Analyzed data reflect analysis performed in the large and homogeneous rims of the grains. The calculated apparent ages range from 144.4 to 161.5 Ma. One group of calculated ages can be defined with a range between 153 and 163 Ma. Two other measurements show younger ages at ca. 143 and 152 Ma that could reflect alteration. No particular petrological evidence could be associated with these two distinct measurements. Integration of the data (n=5) yields an age of 158 ± 2 Ma (1σ , MSWD=1.14; Figure 64).

The greisen Fe-Li-micas from sample 14-MP-20 are millimetric crystals that are the result of the alteration of the granite. Most of the selected grains display alteration on the rim and in fractures. These zones have been avoided during analyses. The calculated apparent ages range from 149.8 to 160.9 Ma. Three measurements are grouped within a range between 153 and 159 Ma. Three other measurements yield an older age between 157 and 163 Ma and two younger ages between 147 and 155 Ma. These measurements are likely to reflect alteration or the occurrence of inclusions. Integration of the data (n=5) yields an age of 155 ± 1 Ma (1σ , MSWD=1.29; Figure 64).

Finally, the Fe-Li-micas from granite sample MP-5-21 are millimetric homogeneous crystals. These selected crystals are devoid of alteration or inclusions. The calculated apparent ages of these Fe-Li-micas range from 145.5 to 160.4 Ma. Seven among nine measurements display a continuum of ages between 151 and 163 Ma. Two other measurements display younger ages between 143 and 149 Ma that could indicate alteration. Integration of the data (n=7) yields an age of 156 ± 1 Ma (1σ , MSWD=0.35; Figure 64).

From overall measurements among samples, a global continuum of calculated ages is observed from 163 to 142 Ma and could reflect common processes among samples.

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Table 11. $^{40}\text{Ar}/^{39}\text{Ar}$ analytical data for Fe-Li-micas from Stages II, III and IV, greisen and granite from the Maoping deposit.

Samples	Relative isotopic abundances (fAmps)*															Apparent age			MSWD	
	⁴⁰ Ar	±	³⁹ Ar	±	³⁸ Ar	±	³⁷ Ar	±	³⁶ Ar	±	Ca/K	±	Cl/K	±	⁴⁰ Ar*/ ³⁹ Ar _(K)	±	⁴⁰ Ar*	Age		±
	(1s)		(1s)		(1s)		(1s)		(1s)		(1s)		(1s)		(1s)		(%)	(Ma)	(1s)	
Stage II Fe-Li-micas																				
Spot 1	47.877	0.069	2.397	0.063	0.056	0.03	0.036	0.028	0.003	0.001	0.509	0.397	0.032	0.037	19.536	0.541	97.917	150.3	4	
Spot 2	52.734	0.060	2.477	0.062	-0.024	0.028	-0.03	0.027	0.01	0.001	-0.41	0.38	-0.066	0.034	20.009	0.536	94.134	153.8	3	
Spot 3	53.542	0.066	2.392	0.063	0.059	0.028	-0.008	0.026	0.013	0.001	-0.121	0.378	0.033	0.034	20.69	0.573	92.571	158.9	4	
Spot 4	65.411	0.071	3.056	0.061	0.041	0.03	0.068	0.027	0.005	0.001	0.749	0.308	0.002	0.029	20.907	0.443	97.761	160.4	3	
Spot 5	63.353	0.068	3.055	0.068	0.047	0.027	0.001	0.026	-0.001	0.001	0.013	0.299	0.01	0.026	20.82	0.487	100.523	159.8	3	
Spot 6	62.223	0.071	3.104	0.063	0.117	0.029	0.015	0.027	0.001	0.001	0.171	0.299	0.073	0.028	19.976	0.429	99.772	153.6	3	
Fluence J: 0.0044501 ± 3.38E-6															Integrated age (all spots):			157	2.0	1.33
Stage III Fe-Li-micas																				
Spot 1	27.783	0.056	1.298	0.065	0.003	0.021	-0.04	0.023	-0.002	0.001	-0.317	0.187	-0.026	0.048	21.83	1.14	102.098	166.5	8	
Spot 2	22.979	0.066	1.153	0.067	0.029	0.029	0.013	0.026	-0.001	0.001	0.114	0.234	0.039	0.074	20.066	1.217	100.746	153.6	8	
Spot 3	39.574	0.067	2.01	0.065	0.056	0.028	0.033	0.025	-0.001	0.001	0.165	0.127	0.046	0.041	19.841	0.669	100.838	151.9	4	
Spot 4	42.619	0.072	1.876	0.061	0.029	0.028	0.006	0.027	0.017	0.001	0.036	0.151	0.004	0.045	19.902	0.691	87.689	152.3	5	
Spot 5	45.513	0.067	2.195	0.064	0.033	0.03	0.033	0.028	0.002	0.001	0.153	0.131	0.008	0.04	20.403	0.624	98.493	156.0	4	
Spot 6	45.251	0.063	2.221	0.065	0.059	0.031	-0.01	0.028	-0.001	0.001	-0.049	0.129	0.041	0.042	20.453	0.62	100.49	156.4	4	
Fluence J: 0.0044289 ± 5.09E-6															Integrated age (spots 2-6):			155	2.0	0.74
Stage IV Fe-Li-micas																				
Spot 1	61.006	0.065	2.861	0.06	0.035	0.03	-0.004	0.027	0.002	0.001	-0.063	0.369	0.001	0.031	21.044	0.46	98.818	161.4	3	
Spot 2	64.251	0.062	3.402	0.061	0.062	0.029	-0.035	0.027	0.001	0.001	-0.390	0.305	0.017	0.025	18.721	0.354	99.254	144.3	2	
Spot 3	63.802	0.074	3.117	0.061	0.042	0.030	-0.001	0.026	-0.001	0.001	-0.021	0.327	0.004	0.028	20.492	0.423	100.223	157.4	3	
Spot 4	62.474	0.073	3.008	0.066	-0.002	0.030	-0.054	0.027	-0.001	0.001	-0.685	0.344	-0.037	0.029	20.834	0.479	100.450	159.9	3	
Spot 5	64.886	0.066	3.170	0.062	-0.018	0.029	0.005	0.025	0.006	0.001	0.060	0.307	-0.053	0.027	19.872	0.408	97.189	152.8	3	
Spot 6	67.337	0.069	3.067	0.060	0.074	0.030	0.070	0.024	0.012	0.001	0.876	0.310	0.033	0.029	20.796	0.430	94.805	159.6	3	
Fluence J: 0.0044501 ± 3.38E-6															Integrated age (all spots -2):			158	2.0	1.14

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Fe-Li-micas from the greisen																				
Spot 1	67.122	0.069	3.290	0.062	0.061	0.028	0.005	0.027	0.005	0.001	0.057	0.290	0.017	0.025	19.917	0.398	97.748	152.8	3	
Spot 2	65.679	0.065	3.176	0.062	0.069	0.028	-0.012	0.025	0.003	0.001	-0.136	0.281	0.027	0.026	20.357	0.417	98.554	156.1	3	
Spot 3	66.738	0.072	3.168	0.068	-0.007	0.030	-0.050	0.025	0.005	0.001	-0.553	0.285	-0.043	0.028	20.458	0.463	97.252	156.8	3	
Spot 4	68.029	0.070	3.291	0.065	0.047	0.030	-0.018	0.025	0.012	0.001	-0.196	0.272	0.004	0.027	19.509	0.407	94.489	149.8	3	
Spot 5	63.828	0.068	2.946	0.066	0.017	0.029	-0.030	0.029	0.006	0.001	-0.360	0.357	-0.0195	0.029	21.009	0.492	97.104	160.9	3	
Spot 6	68.017	0.072	3.298	0.059	0.064	0.032	0.017	0.027	0.001	0.001	0.189	0.294	0.021	0.028	20.458	0.386	99.318	156.8	2	
Fluence J: 0.0044404 ± 3.38E-6															Integrated age (spots 2-6):			155	1.0	1.29
Fe-Li-micas from the granite																				
Spot 1	68.008	0.072	2.952	0.065	0.009	0.029	0.013	0.026	0.039	0.001	0.487	0.95	-0.033	0.029	19.040	0.463	82.775	145.9	3	
Spot 2	98.543	0.071	3.733	0.061	0.099	0.029	0.017	0.026	0.093	0.001	0.476	0.757	0.028	0.023	18.990	0.361	72.044	145.5	2	
Spot 3	67.648	0.067	3.201	0.053	-0.037	0.031	-0.017	0.025	0.007	0.001	-0.575	0.825	-0.071	0.029	20.369	0.372	96.542	155.6	2	
Spot 4	73.026	0.067	3.084	0.062	0.044	0.029	0.044	0.029	0.035	0.001	1.492	1.014	-0.000	0.028	20.295	0.454	85.806	155.1	3	
Spot 5	78.688	0.069	2.993	0.066	0.009	0.030	0.008	0.027	0.053	0.001	0.281	0.958	-0.036	0.030	21.023	0.515	80.084	160.4	3	
Spot 6	91.833	0.065	3.012	0.062	0.075	0.029	0.033	0.026	0.104	0.001	1.139	0.912	0.018	0.029	20.255	0.483	66.506	154.8	3	
Spot 7	64.393	0.066	2.968	0.062	0.037	0.029	0.057	0.025	0.011	0.001	2.034	0.933	-0.001	0.029	20.652	0.469	95.263	157.7	3	
Spot 8	77.888	0.073	3.081	0.061	0.010	0.026	-0.035	0.026	0.048	0.001	-1.228	0.928	-0.034	0.026	20.576	0.454	81.547	157.1	3	
Spot 9	72.840	0.072	3.049	0.061	0.009	0.028	0.013	0.026	0.038	0.001	0.458	0.939	-0.033	0.027	20.103	0.442	84.263	153.7	3	
Fluence J: 0.0044243 ± 4.55E-6															Integrated age (spots 3-9):			156	1.0	0.35

* Corrected for blank. mass discrimination. and radioactive decay

Sensitivity: 6.312E-17 ± 1.047E-18 (mol/fAmp)

Values in italic were not used for age calculation.

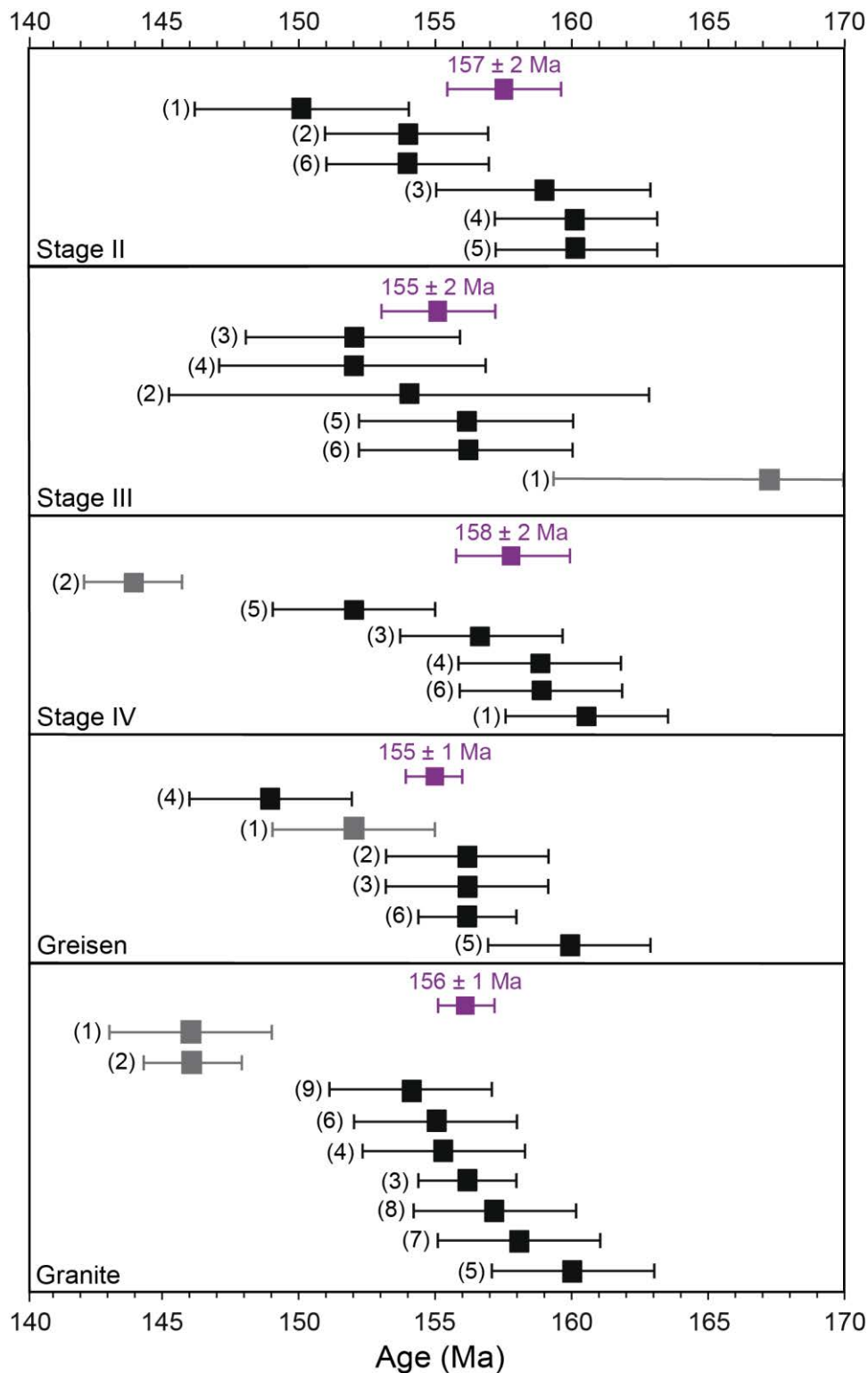


Figure 64. $^{40}\text{Ar}/^{39}\text{Ar}$ ages for Fe-Li-micas from stages II, III and IV from veins, greisen and granite of the Maoping deposit. (A) Six analyses from Stage II Fe-Li-micas displayed at the edge of the mineralized vein yield an age of 157 ± 2 Ma (1σ). (B) Five samples from Stage III Fe-Li-micas associated with ore-bearing minerals (wolframite and cassiterite) overlap within error and define an age of 155 ± 2 Ma (1σ). (C) Five analyses from Stage IV Fe-Li-micas from the Banded quartz veins yield an age of 158 ± 2 Ma (1σ). (D) Five samples from the greisen Fe-Li-micas (cupola at the top of the granite) define an age of 155 ± 1 Ma (1σ). (E) Seven analyses from the unaltered peraluminous Maoping granite Fe-Li-micas overlap within error and yield an age of 156 ± 1 Ma (1σ). $^{40}\text{Ar}/^{39}\text{Ar}$ isotopic data are given in Table 11. Grey dots represent analyses that were not used for age calculation.

5.2 U-Pb wolframite dating

The wolframite samples of the two series have U contents from 14 to 22 ppm (concentrate) and from 8 to 12 ppm (vein sample), whereas Pb contents are below 1.5 ppm with variable contributions of common lead (Table 12). The variable contributions of common lead account for the large range of measured $^{206}\text{Pb}/^{204}\text{Pb}$ ratios from 55 to 420. The wolframite samples show essentially no variation in $^{208}\text{Pb}/^{204}\text{Pb}$ (37.90 – 39.96) with $^{206}\text{Pb}/^{204}\text{Pb}$, which indicates the Th contents are low in comparison to the amounts of common Pb present (0.05 to 0.72 ppm). Only sample 9 (Table 12) has a high $^{208}\text{Pb}/^{204}\text{Pb}$ at a high $^{206}\text{Pb}/^{204}\text{Pb}$. Using $^{208}\text{Pb}_{\text{rad}}/^{206}\text{Pb}_{\text{rad}}$, the measured U contents, and an age of 160 Ma indicates that this sample had a Th content ca. 120 ppm. As Th content in wolframite typically are low, this high Th content indicates that this sample hosted micro-inclusions of a mineral with high Th contents and slightly enhanced common Pb contents. Monazite might be such an inclusion. Sample 13 (Table 12) has a high Pb (29.4 ppm) content, which may be due to small sulfide inclusions, and in situ growth of radiogenic Pb contributed only insignificantly to the Pb budget of this sample. Therefore, we used the U and Pb concentration and the measured Pb isotopic composition of this sample to estimate the initial Pb isotopic composition for an age of 160 Ma and used this Pb isotopic composition to calculate the $^{206}\text{Pb}/^{238}\text{U}$ and $^{207}\text{Pb}/^{235}\text{U}$ ratios of the other samples.

The $^{206}\text{Pb}/^{238}\text{U}$ vs $^{207}\text{Pb}/^{235}\text{U}$ diagram with all data from each sample both show two clusters of ages defined around 160 and 155 Ma (Figure 65A and B). Two samples are displayed over these clusters with higher Pb content in the concentrate sample (4 and 5) and could be due to micro-inclusions (Figure 65A). Five samples of the concentrate wolframite define a concordia age of 160.37 ± 0.36 Ma (1σ , MSWD = 0.29; Figure 65C) and three samples of the vein wolframite define a concordia age of 156.28 ± 0.46 Ma (1σ , MSWD = 0.27; Figure 8D).

Table 12. U-Pb analytical results (ID-TIMS) for wolframite from the main W-Sn vein from the Maoping deposit, Jiangxi (China)

Sample ^a	Weight (mg)	Concentrations (ppm)		²⁰⁶ Pb	Common lead (pg)	Radiogenic Pb (at%) ^c			Th/U ^d	Atomic ratios ^c			Apparent ages (Ma) ^e		
		U	Pb	²⁰⁴ Pb		²⁰⁶ Pb	²⁰⁷ Pb	²⁰⁸ Pb		²⁰⁶ Pb	²⁰⁷ Pb	²⁰⁷ Pb	²⁰⁶ Pb	²⁰⁷ Pb	²⁰⁷ Pb
				Measured ratios ^b						²³⁸ U	²³⁵ U	²⁰⁶ Pb	²³⁸ U	²³⁵ U	²⁰⁶ Pb
Concentrate															
1	0.128	17.6	0.57	190.8	27	94.71	4.60	0.69	0.0227	.02520	.16888	.04861	160	158	129
2	0.144	19.2	0.62	190.4	32	94.59	4.60	0.81	0.0267	.02517	.16881	.04863	160	158	130
3	0.119	21.9	0.78	143.7	79	94.91	4.66	0.42	0.0140	.02522	.17071	.04909	160	160	152
4	0.152	13.9	0.62	117.5	67	94.63	4.71	0.66	0.0218	.02904	.19926	.04977	184	184	184
5	0.141	20.6	0.73	168.2	54	94.61	4.63	0.76	0.0252	.02665	.17973	.04891	169	168	144
6	0.119	16.5	1.09	55.17	519	95.85	4.67	0.51	0.0170	.02506	.16829	.04871	159	158	134
7	0.093	0.21	0.74	128.1	82	94.35	4.72	0.92	0.0306	.02461	.16982	.05004	157	159	197
8	0.399	20.1	0.54	420.3	6	94.26	4.71	1.02	0.0340	.02525	.17415	.05001	161	163	196
9	0.331	19.1	1.37	266.6	15	32.49	1.62	65.89	6.3578	.02460	.16942	.04994	157	159	192
Vein sample															
10	1.114	10.2	0.49	83.38	64	94.04	4.80	1.15	0.0384	.02515	.17722	.05110	160	166	245
11	1.029	12.2	0.32	385.6	3	94.60	4.67	0.73	0.0243	.02442	.16629	.04939	155	156	166
12	1.000	8.16	29.4	19.22	835000	<i>f</i>	<i>f</i>	<i>f</i>							
13	1.254	8.16	0.31	125.5	15	94.22	4.68	1.10	0.0365	.02539	.17374	.04963	162	163	178
14	1.139	8.11	0.27	156.4	8	94.86	4.58	0.56	0.0185	.02456	.16349	.04828	156	154	113
15	1.175	11.0	0.46	99.20	46	94.33	4.61	1.06	0.0352	.02468	.16643	.04890	157	156	143

a Small fragments from single columbite grains. Fragments were selected to show only fresh fracture surfaces. Samples were washed in H₂O and acetone.

b Lead isotope ratios corrected for fractionation, blank and isotopic tracer. Samples were analyzed at GFZ German Research Centre for Geosciences, Potsdam, Germany, using a ²⁰⁵Pb-²³⁵U mixed isotopic tracer. Analytical details are given in Romer and Luders (2006). During the measurement period total blanks were less than 15 pg for lead and less than 1 pg for uranium.

c Lead corrected for fractionation, blank, isotopic tracer, and initial lead. The initial lead isotopic composition was estimated using the intercept in the ²⁰⁶Pb/²⁰⁴Pb vs. ²³⁸U/²⁰⁴Pb diagram and correlation in the Pb isotopic composition of the wolframite sample.

d ²³²Th/²³⁸U calculated from radiogenic ²⁰⁸Pb/²⁰⁶Pb and the age of the sample.

e Apparent ages were calculated using the constants recommended by IUGS. $\lambda_{238} = 1.55125 \text{ E-10 y}^{-1}$, $\lambda_{235} = 9.848 \text{ E-10 y}^{-1}$.

f Sample dominated by common lead; measured ²⁰⁶Pb/²⁰⁴Pb = 19.22, ²⁰⁷Pb/²⁰⁴Pb = 15.67, ²⁰⁸Pb/²⁰⁴Pb = 38.79.
Data in italic were not used for age calculation.

The 160 Ma age is interpreted as dating the crystallization of the main wolframite of the Maoping deposit whereas the younger 156 Ma age seems to reflect a later remobilization.

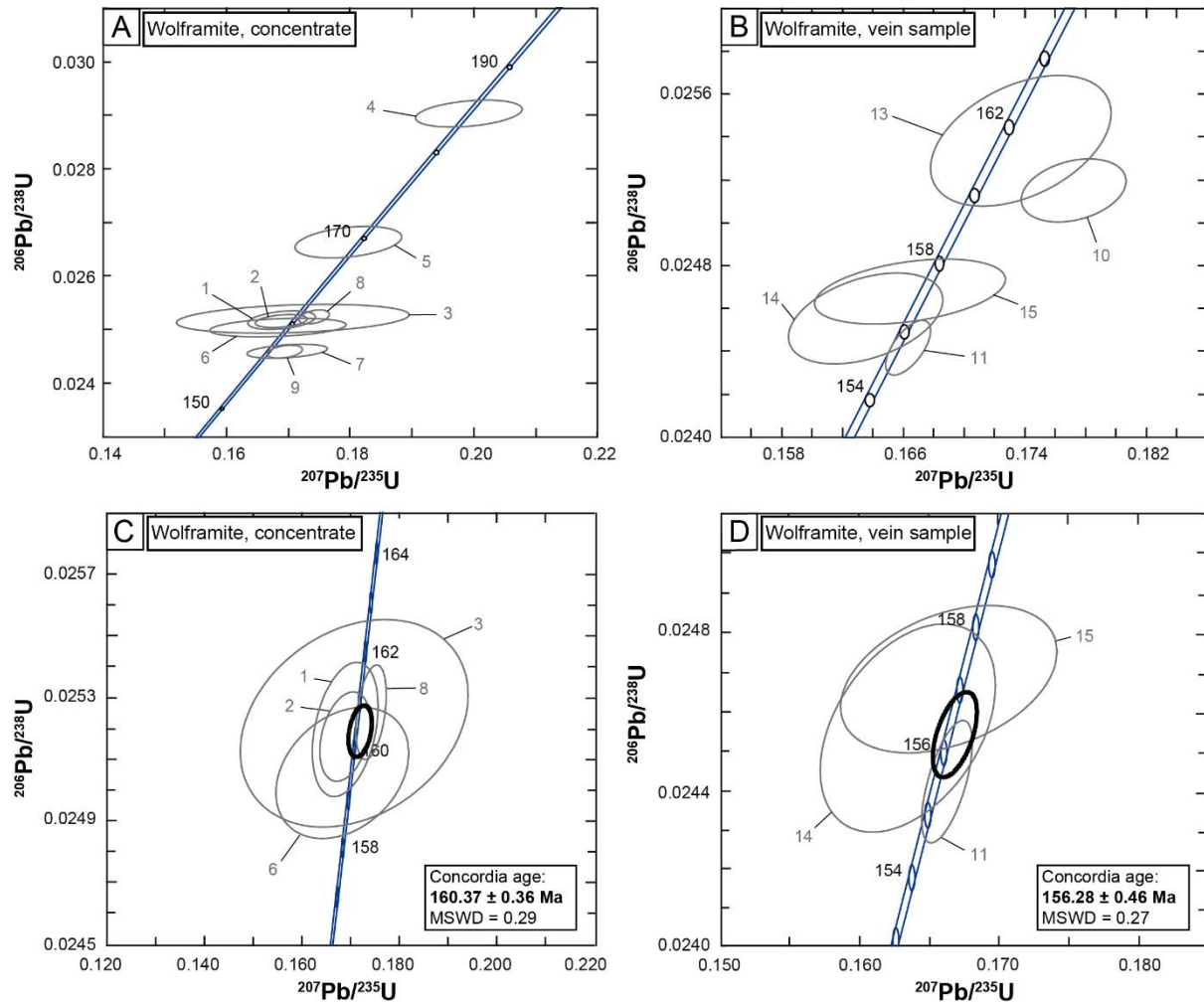


Figure 65. Concordia diagrams for two samples of Stage III wolframite from the Maoping deposit. (A) and (B) full data set for both samples: (1) wolframite concentrate and (2) fragments of single crystal from a vein sample. Note, whether samples were selected from the concentrate or the vein, most samples plot in two clusters on the concordia (C) Five samples of the concentrate yield an age of 160.37 ± 0.36 Ma (1σ). (D) Three samples from the vein crystal define an age of 158.26 ± 0.46 Ma (1σ). Error ellipses are shown at 1σ level. Data from Table 12.

5.3 U-Pb dating of Stage VI xenotime

A total of 34 analyses on 27 chemically homogeneous xenotime grains were performed (Figure 66; Table 13; Appendix C). The combined measured U-Pb-Th data are reported in a $^{206}\text{Pb}/^{238}\text{U}$ vs. $^{208}\text{Pb}/^{232}\text{Th}$ concordia diagram (Figure 66A) rather than in the conventional $^{206}\text{Pb}/^{238}\text{U}$ vs. $^{207}\text{Pb}/^{235}\text{U}$ diagram due to a very low ^{207}Pb signal (average of 650 cps). Eight-

teen concordant analyses cluster in two main groups. Nine concordant analyses overlap within error and yield an age of 152.5 ± 1.3 Ma (1σ , MSWD = 1.9), whereas another nine samples yield an age of 130.1 ± 1.2 Ma (1σ , MSWD = 2.1; Figure 66C and D). The two generations could not be distinguished by shapes, position in the fluorite zoning nor chemical composition. Moreover, ages do not represent the core and rim timing.

Between the two clusters, ellipses spread a continuum of measurements broadly aligned on the concordia line. These measurements seem to represent a mixing between two distinct end-members. Two ellipses are also located on the concordia line but yield younger ages compared to the two clusters (10 and 4 measurements). These ellipses could reflect alteration of the grains although no alteration textures were observed by SEM. The same thing applies to the two ellipses that are isolated, far from the concordia line with high $^{208}\text{Pb}/^{232}\text{Th}$ ratios (15 and 28). These measurements could reflect the occurrence of inclusions in xenotime crystals (i.e. sulphides) that were not previously observed by SEM but could have been revealed by laser ablation.

Table 13. U-Pb-Th analytical data (LA-ICPMS) for xenotime from Stage VI of the Maoping deposit.

Spot#	Isotopic ratios				Measured ages			
	$^{208}\text{Pb}/^{232}\text{Th}$	1 σ	$^{206}\text{Pb}/^{238}\text{U}$	1 σ	$^{208}\text{Pb}/^{232}\text{Th}$	1 σ	$^{206}\text{Pb}/^{238}\text{U}$	1 σ
1	0.00613	0.00007	0.02012	0.00026	123.6	1.36	128.4	1.67
2	0.00752	0.00008	0.02397	0.00031	151.4	1.64	152.7	1.93
3	0.00683	0.00008	0.02264	0.00031	137.5	1.57	144.3	1.98
4	0.00567	0.00007	0.01874	0.00030	114.3	1.31	119.7	1.89
5	0.00668	0.00008	0.02223	0.00031	134.6	1.54	141.8	1.96
6	0.00665	0.00008	0.02078	0.00029	134.0	1.60	132.6	1.82
7	0.00716	0.00009	0.02161	0.00031	144.3	1.77	137.8	1.99
8	0.00736	0.00009	0.02297	0.00035	148.3	1.88	146.4	2.22
9	0.00662	0.00008	0.02050	0.00030	133.3	1.51	130.8	1.88
10	0.00526	0.00006	0.0178	0.00024	106.0	1.14	113.8	1.51
11	0.00683	0.00008	0.01997	0.00027	137.7	1.65	127.5	1.69
12	0.00650	0.00008	0.02111	0.00029	130.9	1.57	134.7	1.83
13	0.00743	0.00009	0.02317	0.00031	149.7	1.74	147.7	1.97
14	0.00726	0.00008	0.02423	0.00033	146.3	1.68	154.3	2.08
15	0.00815	0.00009	0.02329	0.00031	164.0	1.90	148.4	1.96
16	0.00628	0.00007	0.02042	0.00026	126.6	1.36	130.3	1.66
17	0.00638	0.00007	0.02066	0.00027	128.5	1.39	131.8	1.68
18	0.00698	0.00008	0.02345	0.00031	140.6	1.52	149.4	1.93
19	0.00732	0.00009	0.02377	0.00035	147.5	1.73	151.4	2.22
20	0.00635	0.00007	0.02012	0.00026	127.9	1.40	128.4	1.67
21	0.00641	0.00008	0.0207	0.00029	129.1	1.54	132.1	1.81
22	0.0064	0.00007	0.02055	0.00027	128.9	1.44	131.2	1.71
23	0.00763	0.00008	0.02434	0.00032	153.6	1.70	155.0	2.03
24	0.00631	0.00007	0.02035	0.00027	127.1	1.42	129.9	1.72
25	0.00651	0.00007	0.02084	0.00028	131.2	1.50	133.0	1.76
26	0.00724	0.00008	0.02271	0.00031	145.9	1.69	144.8	1.96
27	0.00676	0.00008	0.02199	0.00031	136.1	1.54	140.2	1.93
28	0.00797	0.00009	0.02038	0.00029	160.5	1.86	130.0	1.84
29	0.00764	0.00009	0.02435	0.00033	153.8	1.75	155.1	2.08
30	0.00773	0.00009	0.02456	0.00036	155.6	1.88	156.4	2.26
31	0.00768	0.00009	0.02428	0.00035	154.6	1.82	154.7	2.20
32	0.00777	0.00009	0.02376	0.00032	156.4	1.83	151.4	2.04
33	0.00752	0.00009	0.02415	0.00033	151.4	1.75	153.8	2.05
34	0.00750	0.00009	0.02446	0.00033	151.1	1.75	155.8	2.08

Data in italic were not used for age calculation.

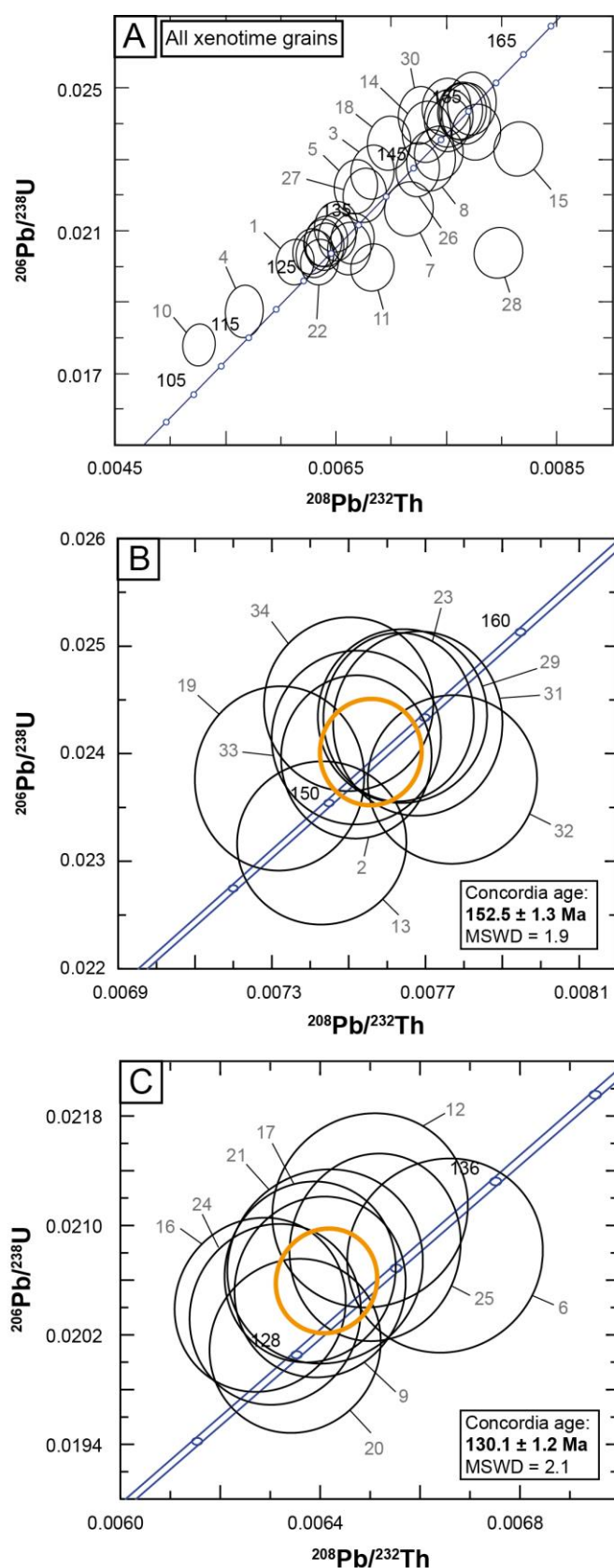


Figure 66. Concordia diagram for Stage VI xenotime from the Maoping deposit. (A) Full data set. The data straddle along the Concordia with two data clusters. (B) Nine concordant analyses overlap within error and yield an age of $152.5 \pm 1.3 \text{ Ma}$ (1σ). (C) Nine concordant analyses overlap within error and yield an age of $130.1 \pm 1.2 \text{ Ma}$ (1σ). Error ellipses show at 1σ level. Data from Table 13.

6. Discussion

6.1 Definition of multiple fluid circulations in the Maoping deposit and the Dayu district

Three types of dating methods including ^{40}Ar - ^{39}Ar on micas, U-Pb on wolframite and U-Pb-Th on xenotime were performed on multiple generations of minerals from the granite, greisen and veins from the Maoping deposit.

^{40}Ar - ^{39}Ar measurements on micas from the granite, greisen and the veins yield a continuum of ages from 163 and 142 Ma (considering errors). This range is observed on every sample and indicates perturbations of the ^{40}Ar - ^{39}Ar isotopic system of the grains. These perturbations are likely to represent the circulation of hydrothermal fluids in our system as observed by Legros et al. (2016). Two hypothesis can be drawn: (i) the isotopic system is completely reset and the calculated age indicates the age of the fluid circulation, or (ii) the calculated ages reflect a mixed age between the initial stage of crystallization and the circulation of the fluid. Considering the continuum of ages observed, the second hypothesis appears to be more likely. In that case, the minimum age of crystallization of micas would be at ca. 160 Ma while the maximum age of circulation of the remobilizing fluid would be at ca. 145 Ma. Therefore, the crystallization of the granitic intrusion cannot be constrained by this study.

Concerning, the U-Pb measurements on wolframite and xenotime, perturbations are also observed. Both wolframite and xenotime samples yield two concordant distinct ages that could reflect both crystallization and fluid circulation ages that also affected the micas. Knowing that wolframite, micas and xenotime from the veins all crystallize from hydrothermal fluids circulating after the magmatic-hydrothermal transition, this hypothesis allows to propose four distinct stages of fluid circulation.

The first stage is constrained by the age of crystallization of the wolframite at ca. 160 Ma. This stage is also constrained by U-Pb measurements on cassiterite samples from the Xihuashan and Piaotang deposits, located in the same district (Bai et al., 2011; Zhang et al.,

2017). Therefore, this stage defined in the Maoping deposit could reflect the circulation of W-Sn-rich fluids in association with the district scale magmatism.

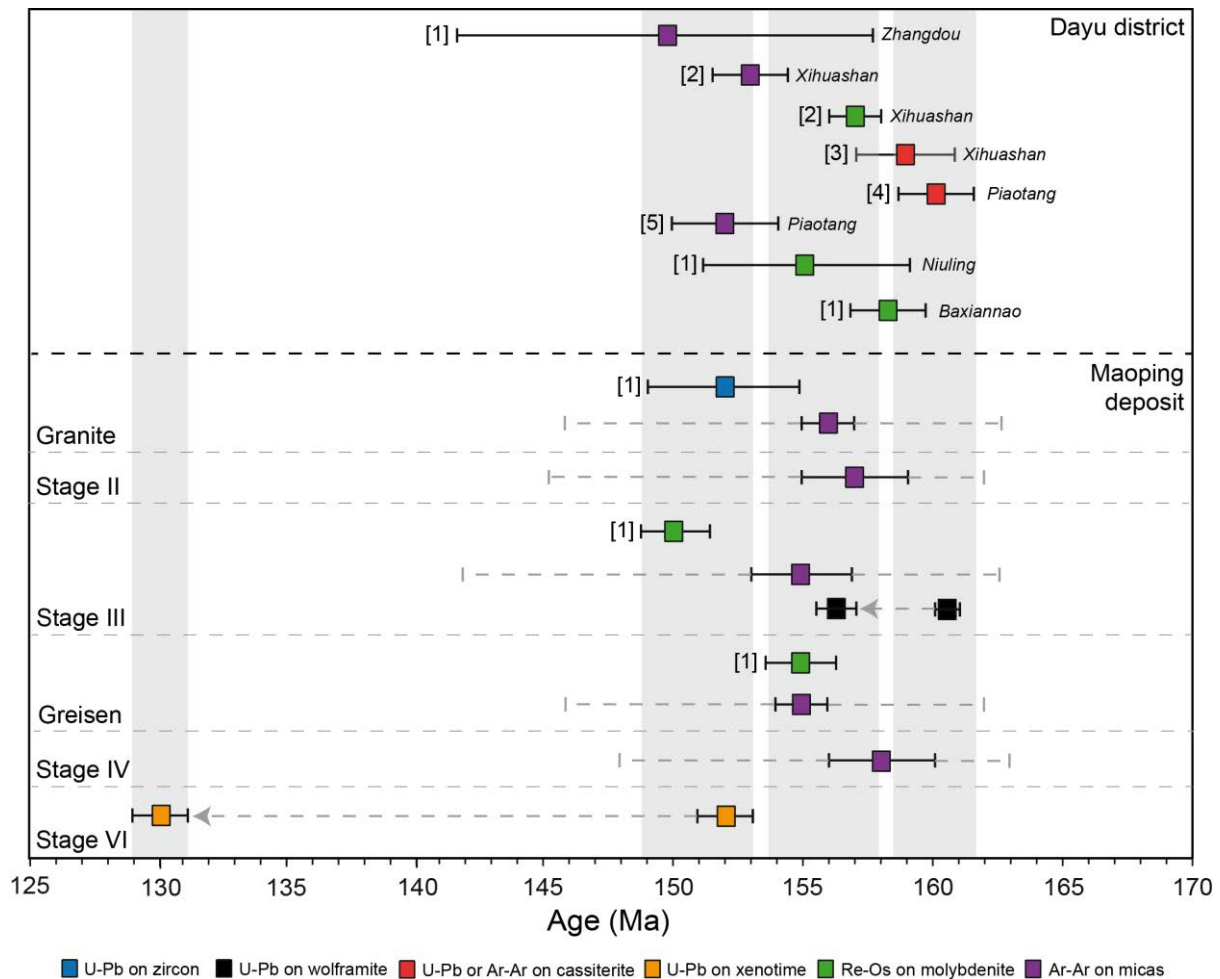


Figure 67. Comparison and compilation of multiple dating data from this study and Feng et al. (2011b) on the Maoping deposit and multiple authors in the Dayu district. [1] Feng et al., 2011b, [2] Hu et al., 2012a, [3] Bai et al., 2011, [4] Zhang et al., 2017, [5] Zhang et al., 2009.

The second stage is defined by U-Pb measurements on wolframite and is interpreted as the timing of wolframite alteration. This stage is constrained at ca. 156 Ma. This age corresponds to the Re-Os measurements performed on molybdenite from the greisen of the Maoping deposit by Feng et al. (2011). The alteration of the wolframite as well as micas could partially be due to the fluid circulations associated with the greisenization of the granite. Fluid circulation during this period is also observed by Re-Os measurements performed on molybdenite from the veins of the Baxiannao, Niuling and Xihuashan deposits from the same district (Feng et al., 2011b; Hu et al., 2012a).

The third stage reflects the timing of crystallization of xenotime crystals at ca. 152 Ma. This timing seems to be distinct from the W-Sn stage and agrees with the paragenetic sequence with a W-Sn-rich stage followed by a REE-rich circulations. This late stage was interpreted in Legros et al. (2016) as associated with Zr-Nb-Ta enrichment that could reflect the influence of a second, hidden, intrusion. In Feng et al. (2011b), the Maoping deposit is associated with the north-plunging extension of the Tianmenshan intrusion outcropping few kilometers away from the mine. U-Pb measurements on zircons of the Tianmenshan granite yield an age of 151.8 ± 2.9 Ma (Feng et al., 2011b). This age cannot be correlated to neither the W-Sn event of the Maoping deposit nor the emplacement of the related granitic intrusion. However, the Tianmenshan intrusion could be the supposed secondary intrusion affecting the Maoping deposit. No particular enrichment in Zr-Nb-Ta-REE has yet been described in the literature concerning the Tianmenshan intrusion but descriptions of this particular intrusion remain scarce. In the same timing range, Feng et al. (2011b) also yields an age of 150.2 ± 2.8 Ma measured by Re-Os on molybdenite and is interpreted as the age of the veins. In the paragenetic sequence of the Maoping deposit, molybdenite is described as an early phase crystallizing at the end of the W-Sn stage but is also described as poorly constrained (Legros et al., 2016). This age could reflect a much later crystallization of molybdenite, coevally to the main sulfide minerals, also associated with the crystallization of REE-minerals. This period correlates at the district scale with the Ar-Ar measurement performed on micas from the veins of the Zhangdou, Xihuashan and Piaotang deposits (Feng et al., 2011b; Hu et al., 2012a; Zhang et al., 2009).

The last stage is associated to the younger concordant ellipses yielded by xenotime measurement and giving an age of ca. 130 Ma. No similar ages are mentioned in the literature for deposits of the Dayu district.

6.2 Comparison to the Nanling Range

Multiple dating ages on W-Sn vein-type deposits have been synthesized on Figure 68 at the Nanling metallogenic belt scale (details in Appendix D and E). The probability repartition of ages between 50 and 250 Ma (Figure 68B) is consistent with previous published data interpreting three epochs of fluid circulation in the Nanling region in which the 150-160 Ma range was the most developed. However, the probability repartition of ages in the 130-170 Ma range (Figure 68C) meets our previous observations in the Dayu district of a multiple circulations in these deposits in the 150-160 Ma period. Similarly to observations made in the Dayu district, the repartition is developed in three peaks of fluid circulations although periods need to be adjusted. Moreover, another range of ages appears between 130 and 140 Ma as observed at Maoping, mostly controlled by the work of Wang et al. (2016) by U-Pb measurements of hydrothermal zircons. The significance and consistency of these ages with our study will be further discussed in the following.

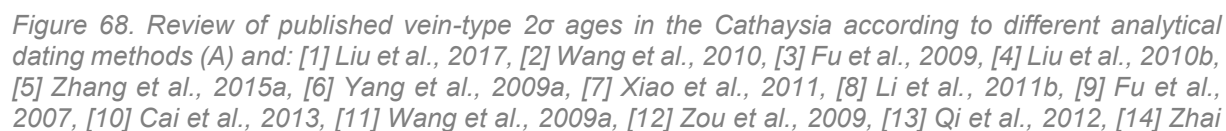
6.2.1 The tungsten mineralization

Within the Nanling Range and more specifically in the southern Jiangxi and Hunan provinces, multiple fluid inclusion studies have been carried on several deposits as Xihuashan (Wei et al., 2012), Yaogangxian (Hu et al., 2012b), Dajishan, Dangping, Piaotang, Pangushan (Ni et al., 2015), Taoxikeng (Wang et al., 2012b), Shimenshi (Gong et al., 2015; Wei et al., 2017), Baxiannao (Feng et al., 2012), Shizhuyuan and Yejiwei (Xuexin et al., 1990). Fluid inclusion studies have been performed on both transparent and opaque minerals and allow to distinguish different processes for gangue and W-Sn minerals. The commonly interpreted model invoke either two fluids (magmatic dominant and meteoric minor) mixing or a single fluid (magmatic) cooling, leading to the precipitation of ore minerals. In the Maoping and Piaotang deposits, fluid circulation posterior to the W-Sn mineralization has also been interpreted in association with the emplacement of a second intrusion. In conclusion, it is known that multiple fluid circulations exist in W-Sn deposits from the Nanling Range and that fluids associated to the W-Sn mineralization usually display different characteristics compared to gangue minerals.

Owing that, the timing of W-Sn deposition in the Nanling Range can only be constrained by the three ages on cassiterite and wolframite previously described in the Dayu district at ca. 160 Ma. Other ages are subjected to reflect either: (i) mixing ages of fluid circulations, (ii) reset ages or, (iii) the age of crystallization the analyzed gangue minerals that does not necessarily reflects the age of W-Sn mineralization.

6.2.2 The 130 Ma event

In the literature, very few geodynamic or metallogenic events have been described in the 130 Ma period in the Nanling range. However, Wang et al. (2016 and references therein) underlines a similar event around 130 Ma in the Nanling range as a new proxy for the tungsten mineralization, interpreted as disconnected from the granite crystallization. The event has been dated by U-Pb method on hydrothermal zircons under the principle that zircons coexist with the ore minerals and thus should form from the same hydrothermal fluids (Wang et al., 2016). In the Maoping deposit, hydrothermal zircons have also been described but coevally to the banded quartz veins on Stage IV and petrologically postdating the tungsten mineralization. The mineralogical assemblage presented by Wang et al. (2016) is very similar to our observations on the Maoping deposit associating hydrothermal zircons to REE minerals and fluorites. This event is not related to the crystallization of the ore minerals but is associated with another metallogenic assemblage enriched in Nb-Ta-Zr-REE. This signature could unravel another magmatic and mineralizing event in the Nanling metallogenic belt that would relate to a peralkaline source emplaced around 130 Ma as interpreted in Legros et al. (2016). Such a source could explain the renewal of fluid circulations at ca. 130 Ma in the Nanling Range and appears to affect the previously formed W-Sn deposits.



et al., 2010, [15] Fu et al., 2008, [16] Zhang et al., 2015b, [17] Zhang et al., 2013, [18] Yuan et al., 2007, [19] Yuan et al., 2012, [20] Liang et al., 2016, [21] Wang et al., 2016, [22] Wang et al., 2008, [23] Peng et al., 2006, [24] Zhai et al., 2011, [25] Feng et al., 2011b, [26] Zeng et al., 2011, [27] Mao et al., 2013 [28] Zhang et al., 2011, [29] Feng et al., 2015, [30] Huang et al., 2011, [31] Liu et al., 2011, [32] Feng et al., 2011a, [33] Liu et al., 2008, [34] Zhang et al., 2009, [35] Zhang et al., 2017, [36] Guo et al., 2011a, [37] Hu et al., 2012a, [38] Bai et al., 2011, [39] Lecumberri-Sanchez et al., 2014. (B) Probability diagram of all ages from W-Sn vein-type deposits collected in the literature and zoom (C) on the 130-170 Ma most productive period.

7. Conclusion

This study raises evidences of multiple fluid circulations in the Nanling Range and in particular in the Maoping deposit. These circulations induce perturbations of the ^{40}Ar - ^{39}Ar and U-Pb systems of both gangue and W-Sn minerals. The deconvolution of measurements performed on micas, wolframite and xenotime allow to propose four timing of fluid circulations in the Maoping deposit: (i) fluids responsible for the W-Sn mineralization at ca. 160 Ma, (ii) fluid circulation at ca. 156 Ma associated with the greisenization and responsible for the alteration wolframite samples, (iii) fluids crystallizing the late filling stages in association with REE-minerals and sulfides at ca. 152 Ma, and (iv) REE- and Zr-rich fluids circulating at ca. 130 Ma. These observations are corroborated by measurements performed at the district scale but also at the scale of the Nanling Range where cassiterite U-Pb dating confirm an early emplacement of W-Sn deposit at ca. 160 Ma. As fluids crystallizing gangue minerals do not necessarily reflect the ore-forming fluids, more direct dating analyses of ore minerals in the Nanling Range need to be performed.

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Appendix A – Detailed analytical ^{40}Ar - ^{39}Ar dating procedure on Fe-Li-micas

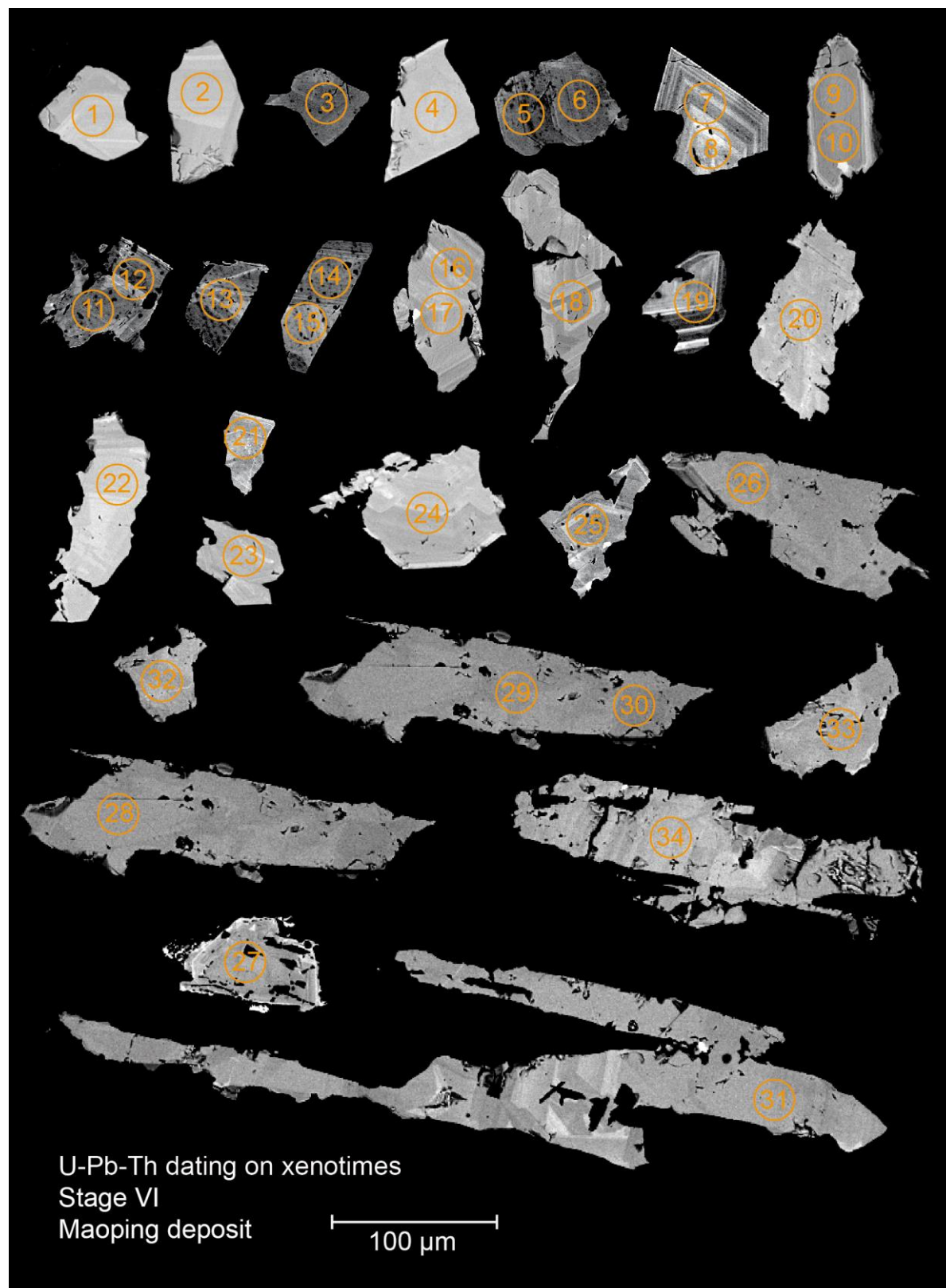
$^{40}\text{Ar}/^{39}\text{Ar}$ analytical work was performed at the University of Manitoba (Canada) using a multi-collector Thermo Fisher Scientific ARGUS VI mass spectrometer, linked to a stainless steel Thermo Fisher Scientific extraction/purification line, Photon Machines (55 W) Fusions 10.6 CO_2 laser, and Photon Machines (Analyte Excite) 193 nm laser. Argon isotopes (from mass 40 to 37) were measured using Faraday detectors with low noise $1 \times 10^{12} \Omega$ resistors and mass 36 was measured using a compact discrete dynode (CDD) detector. The sensitivity for argon measurements is $\sim 6.312 \times 10^{17}$ moles/fA as determined from measured aliquots of Fish Canyon Sanidine (Dazé et al., 2003; Kuiper et al., 2008). Standards and samples were placed in 2 mm deep wells in 18 mm diameter aluminium disks, with standards placed strategically so that the lateral neutron flux gradients across the disk could be evaluated. Planar regressions were fit to the standard data, and the $^{40}\text{Ar}/^{39}\text{Ar}$ neutron fluence parameter (J) interpolated for the unknowns. Uncertainties in J are estimated at 0.1 - 0.2% (1σ), based on Monte Carlo error analysis of the planar regressions (Best et al., 1995). All specimens were irradiated in the Cadmium-lined, in-core CLICIT facility of the TRIGA reactor at the Oregon State University (USA). The duration of irradiation was 17 hours. Standard used was the GA1550 biotite (Spell and McDougall, 2003). Standards for $^{40}\text{Ar}/^{39}\text{Ar}$ measurements were placed in a Cu sample tray, with a KBr cover slip, in a stainless steel chamber with a differentially pumped ZnS viewport attached to a Thermo Fisher Scientific extraction/purification line and baked with an infrared lamp for 24 hours. Single crystals were fused using the CO_2 laser. The samples selected for the $^{40}\text{Ar}/^{39}\text{Ar}$ dating were previously investigated by optical microscopy and SEM, in order to spot homogeneous areas within the analyzed minerals (mica or adularia) devoid of alteration, micro-inclusions or internal zoning. Discs of 5 mm in diameter and $\sim 150 \mu\text{m}$ thick were cut from the same polished thick section (150 - 200 μm thick). The discs were mounted using a ceramic adhesive (PELCO) on a quartz slide placed in a stainless steel chamber with a sapphire viewport attached to the same stainless steel high vacuum extraction system as

the CO₂ laser, and baked with an infrared lamp for 48 hours. For this study, a raster size of about 100 x 100 µm was used and ablation pits were excavated to an estimated depth of 50 µm. Reactive gases were removed for both the standard and unknown, after 3 minutes, by three GP-50 SAES getters (two at room temperature and one at 450 °C) prior to being admitted to an ARGUS VI mass spectrometer by expansion. Five argon isotopes were measured simultaneously over a period of 6 minutes. Measured isotope abundances were corrected for extraction-line blanks, which were determined before every sample analysis. Line blanks in both the Excimer and CO₂ system averaged ~3 fA for mass 40 and ~0.013 fA for mass 36. Mass discrimination was monitored by online analysis of air pipettes based on a power law relationship (Renne et al., 2009), which gave $D = 1.0081 \pm 0.0002$ per amu, based on 71 aliquots interspersed with the unknowns. A value of 295.5 was used for the atmospheric ⁴⁰Ar/³⁶Ar ratio (Steiger and Jäger, 1977) for the purposes of routine measurement of mass spectrometer discrimination using air aliquots, and correction for atmospheric argon in the ⁴⁰Ar/³⁹Ar age calculation. Corrections are made for neutron-induced ⁴⁰Ar from potassium, ³⁹Ar and ³⁶Ar from calcium, and ³⁶Ar from chlorine (Roddick, 1983; Renne et al., 1998; Renne and Norman, 2001).

Appendix B – LA-ICPMS analyses of wolframite samples

ppm	Maoping_0		Maoping_1		Maoping_2		Maoping_3		Maoping_4	
		±		±		±		±		±
Li	3.3	0.5	2.5	0.4	2.4	0.4	2.5	0.4	2.1	0.4
Mg	10.3	0.7	6.9	0.6	6.2	0.5	5.9	0.6	13.7	0.7
Sc	169	6	135	4	189	3	367	19	209	6
Ti	18.1	2.1	16.9	2.3	9.4	1.6	8.2	1.4	21.8	2.1
V	0.4	0.1	0.2	0.1	0.1	0.1	0.1	0.0	0.3	0.1
Mn	161300	3200	120300	1400	127400	1300	175700	2000	159400	3500
Fe	37300	1400	57940	590	52430	700	26710	330	34710	770
Co	0.3	0.1	0.4	0.1	0.3	0.1	0.2	0.1	0.3	0.1
Cu	5.0	0.5	5.3	0.5	2.3	0.4	8.9	0.7	5.0	0.5
Zn	122	4	150	4	150	5	178	5	163	6
Ga	0.1	0.0	0.2	0.1	0.3	0.1	0.2	0.1	0.1	0.0
Ge	0.3	0.2	1.0	0.3	0.3	0.2	0.4	0.2	0.6	0.2
Rb	2.0	0.2	1.5	0.2	1.9	0.2	1.5	0.2	1.3	0.1
Sr	0.8	0.1	1.0	0.1	0.7	0.1	0.5	0.1	1.2	0.1
Y	166	8	192	4	185	3	65	5	59	2
Zr	43	1	84	4	132	3	31	4	27	2
Nb	3005	72	2860	49	4111	47	2260	170	1917	52
Mo	46	7	157	3	109	3	78	2	66	2
Cd	0.2	0.1	0.2	0.1	0.2	0.1	0.6	0.2	0.4	0.2
In	19	0	25	1	33	1	83	5	52	1
Sn	70	2	46	2	75	3	70	9	59	3
Hf	7.6	0.3	20.6	1.0	33.5	1.0	4.6	0.6	6.2	0.5
Ta	637	8	779	20	1127	18	1120	100	1121	47
Pb	1.1	0.2	0.6	0.1	1.4	0.2	0.9	0.1	1.0	0.1
Bi	0.8	0.1	0.3	0.0	1.2	0.2	0.4	0.1	0.9	0.1
Th	0.6	0.1	0.5	0.0	0.4	0.0	0.4	0.1	0.4	0.0
U	17.5	0.4	12.8	0.5	14.8	0.4	17.0	2.3	12.7	0.5
La	0.0	0.0	0.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Ce	0.2	0.0	0.7	0.0	0.5	0.0	0.2	0.0	0.2	0.0
Pr	0.1	0.0	0.3	0.0	0.2	0.0	0.1	0.0	0.1	0.0
Nd	1.0	0.2	2.4	0.2	2.0	0.2	0.9	0.1	1.0	0.1
Sm	4.0	0.4	5.9	0.4	6.4	0.4	3.2	0.3	3.3	0.3
Eu	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Gd	11.3	0.7	12.6	0.6	12.3	0.5	6.7	0.6	6.5	0.4
Tb	7.4	0.3	7.1	0.2	7.8	0.2	4.0	0.3	3.6	0.1
Dy	91.1	2.5	80.1	1.9	88.3	1.3	45.3	3.5	37.7	1.2
Ho	25.5	0.7	21.2	0.4	22.8	0.3	11.8	1.0	9.4	0.3
Er	118	2.5	94.4	2.2	104	1.5	55.0	4.8	39.2	1.1
Tm	34.6	0.8	27.1	0.6	30.0	0.4	16.4	1.4	10.9	0.3
Yb	391	8.8	308	6.7	345	4.0	189	16.0	119	2.9
Lu	65.4	1.5	51.6	1.0	56.9	0.6	31.5	2.7	19.1	0.5

Appendix C – Back-scattered electron images of xenotime extracted from Stage VI fluorite of the Maoping deposit. Circles indicate location of isotopic U-Pb-Th analysis.

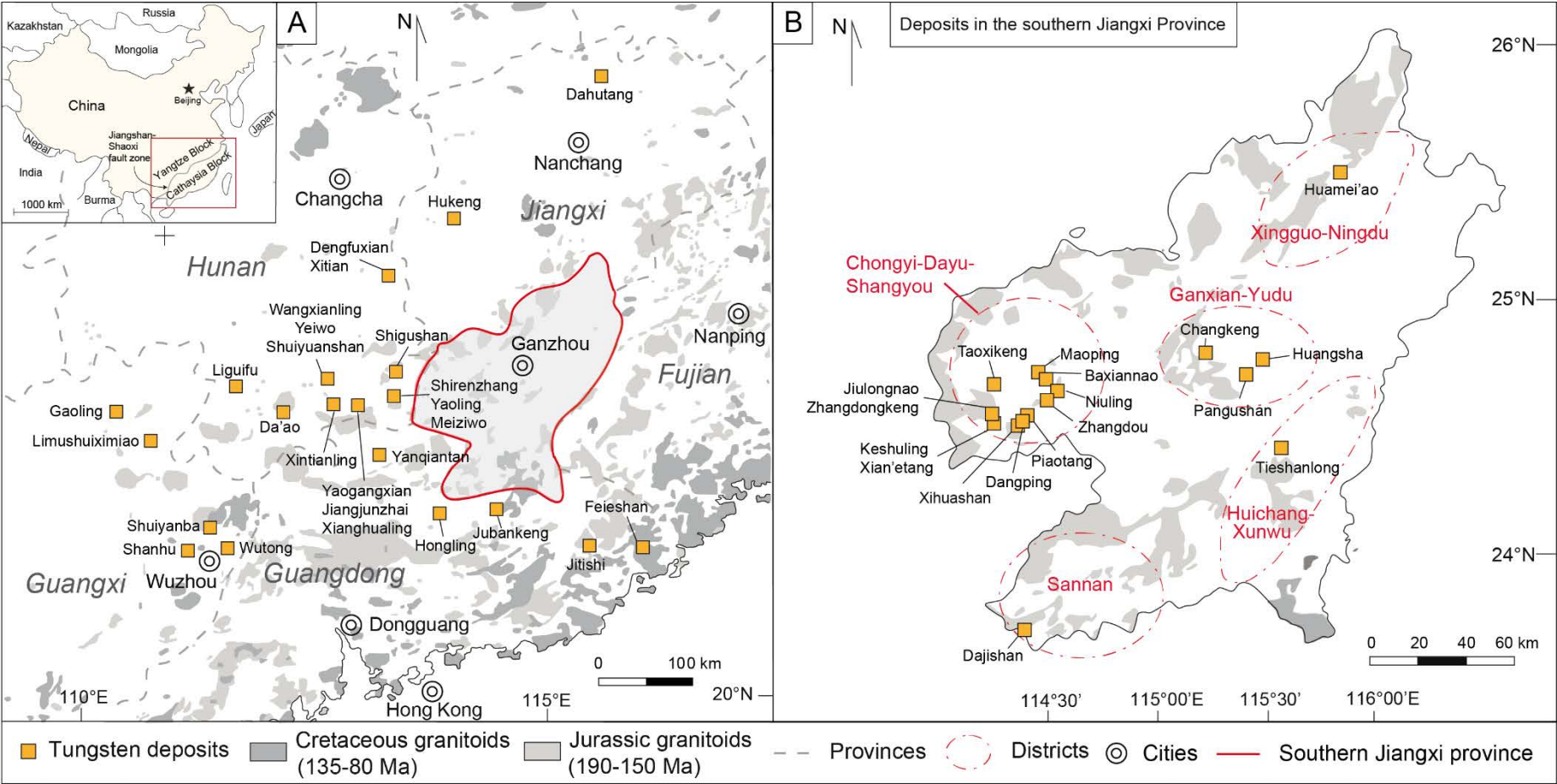


Appendix D – Table of dating data from the literature on W-Sn veins in the Cathaysia

Province	Deposit	Age	2 σ	Analytical method	Reference associated with figure 12
Guangdong	Feie'shan	135.1	0.8	^{40}Ar - ^{39}Ar on micas	1
		140.6	1.9	Re-Os on molybdenite	1
	Hongling	159.1	1.5	Re-Os on molybdenite	2
	Jitishi	155.4	5.1	Sm-Nd on quartz FI	3
	Jubankeng	143.6	3.9	^{40}Ar - ^{39}Ar on micas	3
	Yanqiantan	154.4	1.6	Re-Os on molybdenite	4
	Wutong	92.3 to 104.4		U-Pb on hubnerite	39
Guangxi	Gaoling	212.0	20	Sm-Nd on scheelite	5
	Limushuiximiao	214.1	1.9	^{40}Ar - ^{39}Ar on micas	6
	Shanhu	100.8	0.7	^{40}Ar - ^{39}Ar on micas	7
		102.7	1.7	^{40}Ar - ^{39}Ar on micas	7
	Shuiyanba	162.5	1.2	^{40}Ar - ^{39}Ar on micas	8
Hunan	Daao	151.3	2.4	Re-Os on molybdenite	9
	Dengfuxian	152.4	3.3	Re-Os on molybdenite	10
	Jiangjunzhai	169.6	2.7	Re-Os on molybdenite	11
	Liguifu	211.9	6.4	Re-Os on molybdenite	12
	Meiziwo	157.7	2.8	Re-Os on molybdenite	13
		155.9	0.6	^{40}Ar - ^{39}Ar on micas	14
	Shigushan	154.2	2.7	Re-Os on molybdenite	15
	Shirenzhang	159.1	2.2	Re-Os on molybdenite	15
	Shuiyuanshan	214.2	2.9	^{40}Ar - ^{39}Ar on micas	16
		220.7	4.1	Re-Os on molybdenite	16
	Wangxianling	220.7	4.1	Re-Os on molybdenite	17
		154.4	1.1	^{40}Ar - ^{39}Ar on micas	18
	Xianghualing	158.7	1.2	^{40}Ar - ^{39}Ar on micas	18
		161.3	1.1	^{40}Ar - ^{39}Ar on micas	18
	Xintianling	161.7	9.3	Re-Os on molybdenite	19
	Xitian	149.7	0.9	Re-Os on molybdenite	20
		133.7	1.3	U-Pb hydrothermal zircon	21
	Yaogangxian	153.0	7.0	Re-Os on molybdenite	22
		153.0	1.1	^{40}Ar - ^{39}Ar on micas	23
		154.9	2.6	Re-Os on molybdenite	23
		155.1	1.1	^{40}Ar - ^{39}Ar on micas	23
	Yaoling	159.5	2.8	Re-Os on molybdenite	13

		149.4	0.7	^{40}Ar - ^{39}Ar on micas	24
	Yeiwo	228.1	2.6	Re-Os on molybdenite	16
Jiangxi	Baxiannao	157.9	1.5	Re-Os on molybdenite	25
	Changkeng	158.1	1.2	Re-Os on molybdenite	26
	Dahutang	139.2	1.0	Re-Os on molybdenite	27
	Dajishan	161.0	1.3	Re-Os on molybdenite	28
	Dangping	133.6	1.0	U-Pb hydrothermal zircon	21
	Huamei'ao	158.5	3.3	Re-Os on molybdenite	29
	Huangsha	153.0	3.0	Re-Os on molybdenite	30
		132.9	1.5	U-Pb hydrothermal zircon	21
	Hukeng	147.2	1.4	^{40}Ar - ^{39}Ar on micas	31
		150.2	2.2	Re-Os on molybdenite	31
	Jiulongnao	151.5	1.1	Re-Os on molybdenite	32
	Keshuling	158.8	1.2	^{40}Ar - ^{39}Ar on micas	33
	Maoping	150.2	2.8	Re-Os on molybdenite	25
	Niuling	154.9	4.1	Re-Os on molybdenite	25
	Pangushan	157.8	0.8	Re-Os on molybdenite	26
		158.8	5.7	Re-Os on molybdenite	26
	Piaotang	152.0	1.9	^{40}Ar - ^{39}Ar on micas	34
		159.5	1.5	U-Pb on cassiterite	35
	Taoxikeng	134.4	1.9	U-Pb hydrothermal zircon	21
		152.7	1.5	^{40}Ar - ^{39}Ar on micas	36
		153.4	1.3	^{40}Ar - ^{39}Ar on micas	36
		154.4	3.8	Re-Os on molybdenite	36
	Tieshanlong	134.2	1.6	U-Pb hydrothermal zircon	21
	Xian'etang	231.4	2.4	^{40}Ar - ^{39}Ar on micas	33
		152.8	1.6	^{40}Ar - ^{39}Ar on micas	37
	Xihuashan	157.8	0.9	Re-Os on molybdenite	37
		158.6	2.9	^{40}Ar - ^{39}Ar on cassiterite	38
	Zhangdongkeng	150.9	5.3	Re-Os on molybdenite	32
	Zhangdou	149.1	7.1	Re-Os on molybdenite	25

Appendix E – Map of the W-Sn deposits associated with W-Sn vein dating in the Cathaysia (details in Appendix D and Figure 68)



Chapter 6 – New columbo-tantalite reference materials for SIMS U-Pb geochronology

Article in preparation for Chemical Geology.

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Abstract

Columbo-tantalite is the most common Nb-Ta mineral. It mostly occurs in granite and pegmatites but also in hydrothermal systems associated to other rare metals as W-Sn. This mineral is particularly suitable for U-Pb dating due to its high U and low Pb content. However, no reference material is, at this day, available for *in situ* dating. In order to evaluate the accuracy of our samples for *in situ* U-Pb dating, ID-TIMS was performed to determine the age of each sample. Correlation lines for SIMS calculations were drawn according to the chemical composition of the samples. It appears that ferrotapiolite, ferrocolumbite and ferrotantalite display three distinct lines of calibration and therefore that the Ta# ($Ta/(Ta+Nb)$) can induce a significant matrix effect during SIMS U-Pb dating. Therefore, a sample was selected among each range of chemical composition as an external standard: CT1 (Ivory Coast) for ferrotapiolite, Rongi (Rwanda) for ferrocolumbite and NP2 (China) for ferrotantalite. Ages are consistent with ID-TIMS calculations and confirm the absence of analytical bias. This study

proposes five samples as reference material for U-Pb SIMS dating: three ferrotapiolite, CT1 (2046.8 ± 1.1 Ma), CT3 (2053 ± 1.3 Ma) and CT4 (2044.5 ± 1.6 Ma) all originated from Ivory Coast, two ferrocolumbite, Rongi (931.5 ± 2.5 Ma) and Buranga (905.2 ± 3.2 Ma) both originated from Rwanda and two ferrotantalite, NP2 (380.3 ± 2.4 Ma) from China and NT-2 (372.0 ± 2.3 Ma) from Altai. Age calculation of younger samples still need testing to measure the possible bias induced.

Keywords

Columbite – Tantalite – U-Pb – SIMS – ID-TIMS - Geochronology

1. Introduction

Niobium and tantalum are considered as critical resources due to their wide applications in the electronic industry (Mineral profiles by BGS; www.bgs.ac.uk). These economic elements are mostly exploited under the form of columbo-tantalite minerals $[(\text{Fe},\text{Mn})(\text{Nb},\text{Ta})_2\text{O}_5]$ (termed coltan). Columbo-tantalite minerals are considered as promising material for U-Pb dating due to their high U and low Pb contents (Romer and Lehmann, 1995; Romer and Smeds, 1994; Romer and Wright, 1992; Smith et al., 2004). Columbo-tantalites are encountered in rare metal granites, alkaline and carbonatitic rocks, pegmatites and hydrothermal veins. Coltan U-Pb dating could help constrain the emplacement of these rocks as well as the possibly associated magmatic-hydrothermal transition that is often observed in these deposits and could not be defined by U-Pb zircon dating. Moreover, the Nb-Ta ratios are known to be good indicators of magmatic differentiation and the dating of several generations could constrain such magmatic evolutions.

Coltan U-Pb dating have already been reported using isotope dilution thermal ionisation mass-spectrometry (IDTIMS). *In situ* laser ablation inductively-coupled plasma mass spectrometry (LA-ICPMS) and secondary ion mass spectrometry (SIMS) techniques could

provide an alternative. However, no appropriate coltan standard has yet been published. Indeed, the columbo-tantalite group mineral has the particularity to own four chemical poles based on Nb-Ta and Mn-Fe ratios. Many studies by *in situ* laser ablation inductively-coupled plasma mass spectrometry (LA-ICPMS) and secondary ion mass spectrometry (SIMS) techniques have proven that standardisation with an inappropriate material that do not share the same matrix components could lead to so-called “matrix effects” and thus alter the subsequent dating results (e.i. Zhu et al., 1998; Che et al., 2015). The question of the need to identify four coltan potential standards remain.

This contribution tends to complement the results of U-Pb coltan dating by IDTIMS by providing new standards for *in situ* U-Pb dating methods as SIMS. In this view, several coltan samples from deposits worldwide have been collected and analysed by the two methods after identifying their chemical ratios. This paper discusses the potential matrix effects between the different coltan chemical poles and introduces seven new standards for *in situ* U-Pb dating methods by SIMS.

2. Sample location

2.1 CT and ISSIA, Ivory Coast

All columbo-tantalite products from Ivory Coast are associated with placer accumulations in soils in central-west of the country. Soils are directly associated to the weathering of subsequent granites and pegmatites under the surface. Most accumulations are interpreted as directly reflecting the composition of respective pegmatite at each location. Our samples are located next to the ISSIA granite, described as a non-oriented muscovite-dominated porphyroid peraluminous granite, associated to Nb-Ta-Be-Li pegmatites (Allou et al., 2005).

2.2 Rongi and Buranga, Rwanda

Rongi and Buranga samples were both sampled from pegmatites from the Karagwe-Ankole belt of Central Africa. Both pegmatites are located in the Gatumba, 50 km west of Kigali (Rwanda) and hosted in dolerites. These samples have been already studied by Dewaele et al. (2011) and Melcher et al. (2015) with multiple dating techniques.

2.4 NT-2 and A-1, Altai

These samples were collected from the Altai No. 3 pegmatite, located near Koktokay town in Xinjiang Province, China. This pegmatite is one of the most differentiated intrusions among more than 10 thousand pegmatites in the area. Petrographic details are given in Zhang et al. (2004) and Wang et al. (2012) and LA-ICPMS dating in Che et al. (2015).

2.5 NP2, Fujian, China

NP2 was sampled from the Nanping No. 31 pegmatite. This pegmatite is located in the Northern Fujian Province at 8 km to the west from the Nanping city. The No. 31 pegmatite is evolved and mineralized in Nb, Ta and Sn. More petrographic details are given in Rao et al. (2009) and LA-ICPMS dating in Che et al. (2015).

2.6 CC1716, France

CC1716 samples are grains extracted from the Epesses hercynian granite located in Vendée, not far from the Chateauroux and Chateliers cities (France). Several granitic facies are described in the sampling area (Guérangé et al., 1971) and all contain disseminated amount of columbo-tantalite.

3. Analytical techniques

Petrographic images were obtained from observations of polished sections using reflected light microscopy and a HITACHI FEG S4800 scanning electron microscope (SEM)

equipped with an energy dispersive spectrometer (EDS), using a Si(Li) semi-conductor detector.

3.1 EPMA

Electron microprobe analyses (EPMA) for Ti, Mn, Fe, Nb, Sn, Zr, Ta, W, Pb, Th, U were performed on columbotantalite samples, using a CAMECA SX100 instrument equipped with a wavelength dispersive spectrometer (WDS) and calibrated using natural and synthetic oxides (MnTiO₃, Fe₂O₃, LiNbO₃, cassiterite, zircon, LiTaO₃, scheelite, galena, ThO₂, UO₂). The analytical conditions were: current of 20 nA and accelerating voltage of 20 kV (20kV and 100nA for Pb, U and Th) and a counting time of 10 s per element. Total Fe is presented as FeO. W, Pb, Th and U were measured below the limit of detection and were removed from the EPMA results.

3.2 TIMS

Columbo-tantalite samples U-Pb ratios were analyzed at the GFZ German Research Centre for Geosciences (Potsdam), according to the analytical procedure described in Romer and Lüders (2006). Samples were crushed and picked carefully under the binocular microscope to avoid alteration, filled fractures or recrystallization zones. Grains were picked as small as possible in order to minimize the risk of nanoinclusions. Any contamination on surfaces of the grains were washed through 20% HF, 6N HCl, 7N HNO₃, H₂O and acetone. A second selection of the cleaner grains at that point was also performed. A ²⁰⁵Pb-²³⁵U tracer was also included before dissolving grains between 0.200 mg and 0.500 mg in 40% HF on a hot plate overnight. For each samples, a first batch of six analyses were performed before adjusting given the first analyses. Pb and U were separated by ion exchange chemistry (Romer et al., 2005) for distinct measurements for each samples. Each Pb and U samples were loaded on Re single filaments with H₃PO₄ and silicate gel. Isotopic ratios of Pb and U were analyzed on a Triton thermal ionization mass spectrometer (TIMS) using a Faraday collector or an ion counter given the intensity of the signal. Data reduction was performed according to Schmid et al. (2003). Mass fractionation, U-Pb blanks and tracer were corrected. All data are

summarized in Table 2. Uncertainties are given at the 2σ level. Data were plotted using ISOPLOT v.3.75 (Ludwig 1993).

3.3 SIMS

Analyses were performed at CRPG-CNRS (Nancy, France) using a Cameca IMS-1270 ion microprobe. The O^- primary ion beam was accelerated at 13kV, with an intensity of 20nA. The aperture illumination beam mode (Kohler illumination) was used with a 200 μm aperture and 20x30 μm spot size producing elliptical spots. Positive secondary ions were extracted by a 10-kV potential and the spectrometer slits set for a MRP of 13000 to separate isobaric interferences on ^{204}Pb isotope. The field aperture was set to 3000 μm , and the transfer optic magnification adjusted to 80. Rectangular lenses were activated in the secondary ion optics to improve the transmission at high mass resolution (de Chambost et al., 1996). The energy window was opened at 30 eV and its low energy side was positioned at 5 eV before the start of the energy distribution. A single collector was used in ion-counting mode, and the spectrum scanned by peak jumping. Each analysis consisted of 10 successive cycles. Each cycle consists in the measurement of the faraday cup background on the 195.8 mass and then measurement of ^{181}TaO (reference mass), followed by mass 203.5 for the electron multiplier background, $^{93}Nb_2O$, ^{204}Pb , ^{206}Pb , ^{207}Pb , ^{208}Pb , ^{238}U , ^{232}ThO , ^{238}UO and $^{238}UO_2$, with measurement times of 4, 4, 4, 4, 8, 4, 4, 12, 6, 8, 4 and, 3 s respectively (waiting time of 1 or 2 s). The mass and energy calibrations were checked before each measurements, after a 2 min pre-sputtering on a 20x20 raster to clean the surface from coating and contamination. To define the relative sensitivity factor for Pb and U, an empirical linear relationship (Compston et al., 1984) was defined between UO^+/U^+ and Pb^+/U^+ for all measurements performed on the standard of each sample mount. Correction for common lead was made by measuring the ^{204}Pb amount of the common lead composition was calculated at the $^{207}Pb/^{206}Pb$ measured age, using the model of Stacey and Kramers (1975).

4. Results and discussion

4.1 BSE imaging and reflected light

Investigation of millimetric chips of selected samples using reflected light and BSE imaging and qualitative energy dispersive spectrometry, shows that most of the selected materials are homogeneous and free of any inclusions. Inclusions at the micrometer scale were however observed in NT-2, A-1 and NP2 samples from China and CC1716 from France. However, most of inclusions are located in fractures and therefore can easily be removed or avoided. Moreover, no zoning was observed in the samples (Figure 69 and Figure 70).

4.2 EMPA geochemistry

A total of 232 analyses of columbo-tantalite were performed by EMPA (Table 14, Appendix A). Samples are plotted in the four quadrants of the columbo-tantalite diagram (Figure 71). All samples show chemical homogeneity.

Four groups can be distinguished according to their chemical compositions. Group I is composed of CT1, CT3 and CT4 (Ivory Coast) that show compositions in the ferrotapiolite field with Ta# ($Ta/(Nb+Ta)$) between 0.90 and 0.95 and Mn # ($Mn/(Fe+Mn)$) between 0.04 and 0.07.

Table 14. Representative EPMA analyses for columbo-tantalites

	CT1	CT3	CT4	Rongi	Buranga	NP2	NT-2	A-1	ISSIA2	CC1716
TiO ₂	0.79	0.71	0.95	0.17	0.77	0.37	0.22	0.38	0.24	1.76
MnO	0.73	0.97	0.62	6.84	9.78	6.54	6.88	14.3	9.72	6.11
FeO	14.8	14.5	14.8	13.8	11.1	10.5	11.6	2.87	6.46	15.4
Nb ₂ O ₅	7.19	6.44	7.51	66.2	64.7	42.7	43.6	27.7	18.4	70.5
SnO ₂	0.02	0.08	0.06	0.11	0.44	0.51	0.28	0.01	0.01	0.01
ZrO ₂	0.08	0.00	0.21	0.03	0.31	0.00	0.00	0.00	0.12	0.00
Ta ₂ O ₅	75.2	76.2	75.1	11.5	11.9	38.1	37.5	54.6	63.3	6.50
Total	98.94	98.91	99.17	98.66	99.12	98.69	100.1	99.86	98.39	100.3
Structural formula calculated on the basis of O = 6 atoms										
Ti	0.05	0.04	0.06	0.01	0.03	0.02	0.01	0.02	0.01	0.08
Mn	0.05	0.07	0.04	0.35	0.49	0.37	0.38	0.87	0.63	0.30
Fe	1.01	0.99	1.00	0.69	0.55	0.59	0.64	0.64	0.42	0.74
Nb	0.27	0.24	0.27	1.79	1.74	1.30	1.30	1.30	0.64	1.82
Sn	0.00	0.00	0.00	0.00	0.01	0.01	0.01	0.01	0.00	0.00
Zr	0.00	0.00	0.01	0.00	0.01	0.00	0.00	0.00	0.00	0.00
Ta	1.67	1.70	1.65	0.19	0.19	0.69	0.67	0.67	1.32	0.10
Mn#	0.05	0.06	0.04	0.33	0.47	0.39	0.37	0.83	0.60	0.28
Ta#	0.86	0.88	0.86	0.09	0.10	0.35	0.50	0.70	0.67	0.10

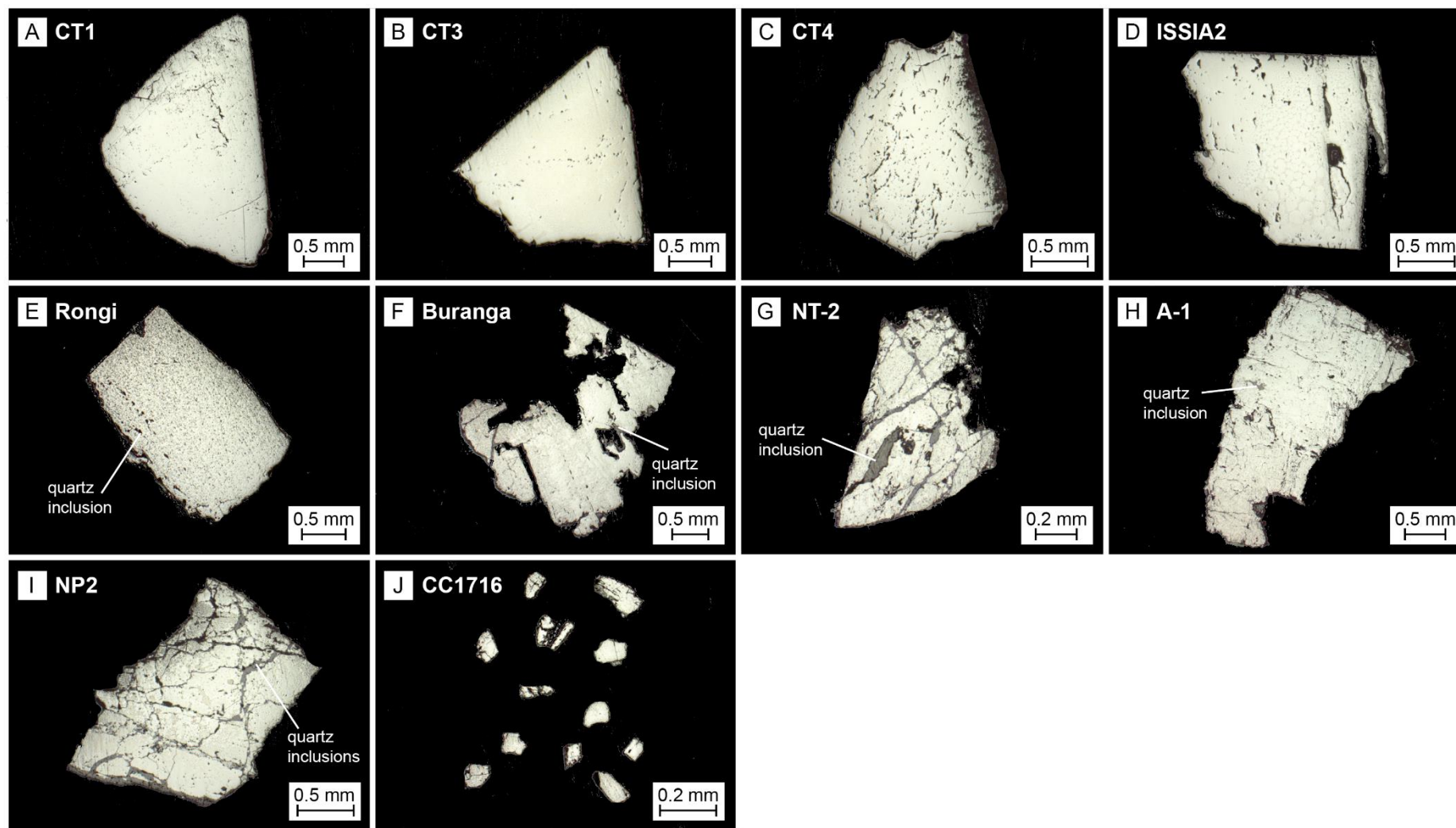


Figure 69. Imaging in reflected light of grains of columbo-tantalite analyzed in this study.

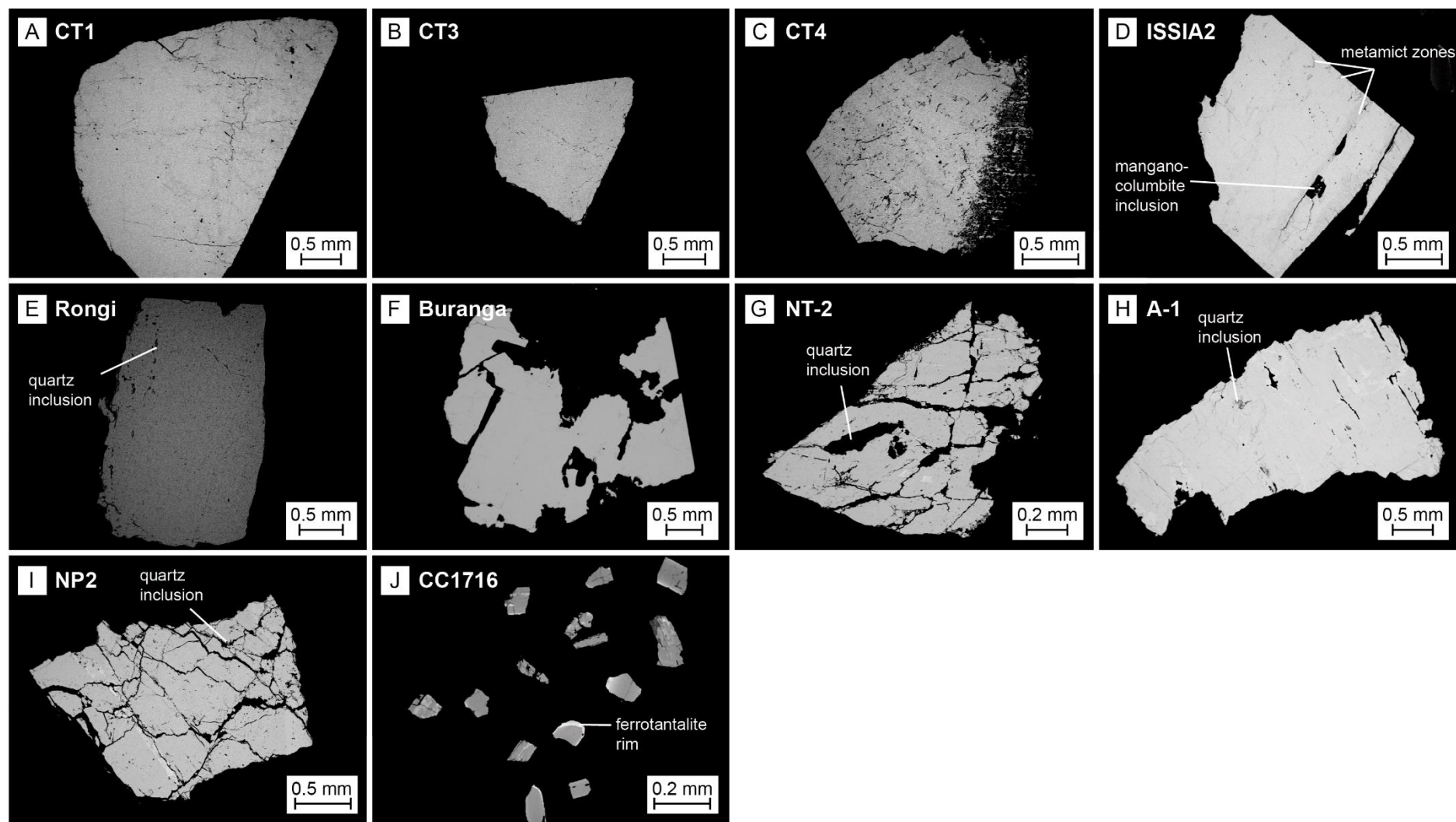


Figure 70. SEM imaging of grains of columbo-tantalite analyzed in this study.

Group II is composed of NP2 (China) and NT-2 (Altai) samples that show intermediate compositions at the edge of the ferrocolumbite and ferrotantalite field. NP2 yields Ta# between 0.50 and 0.53 and Mn # between 0.35 and 0.40 while NT-2 shows Ta# between 0.48 and 0.50 and Mn# between 0.37 and 0.39. One analysis of NP2 shows a higher Mn# of 0.48 and is likely representing a rare inclusion enriched in manganese.

Group III is composed of ISSIA2 (Ivory Coast) and A-1 (Altai) samples that show compositions in the manganotantalite field. ISSIA2 composition is at the edge of the miscibility gap, with Ta# between 0.73 and 0.86 and Mn # between 0.57 and 0.63 while A-1 contains higher Mn content (Mn# between 0.81 and 0.84) and lower Ta content (Ta# between 0.69 and 0.71).

Group IV is composed of Rongi and Buranga (Rwanda) and, CC1716 (France) samples that show compositions in the ferrocolumbite field. Rongi samples are grouped in compositions with Ta# between 0.16 and 0.18 and Mn # between 0.28 and 0.34. Buranga samples display higher content in Mn with Mn# between 0.46 and 0.49 while CC1716 shows lower Ta with Ta# between 0.06 and 0.11.

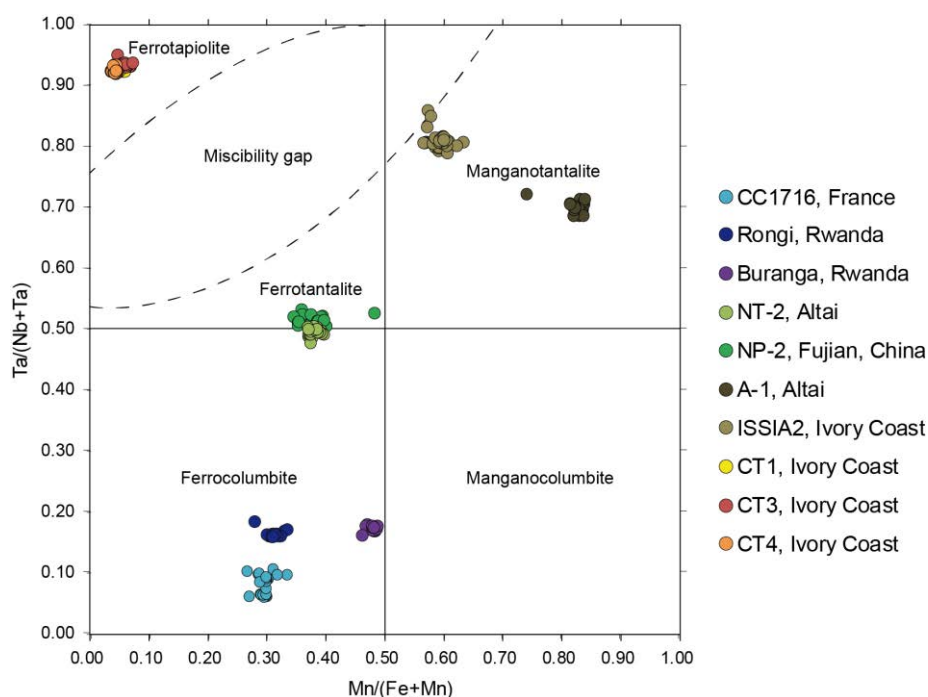


Figure 71. Electron Micro-Probe analysis of columbo-tantalite samples plotted in the four quadrant of the columbite diagram.

4.3 U-Pb ID-TIMS dating

4.3.1 CT1 (Ivory Coast)

In a $^{206}\text{Pb}/^{238}\text{U}$ vs. $^{207}\text{Pb}/^{235}\text{U}$ diagram, all data from the CT1 ferrotapiolite are concordant or very close to the concordia line. Two samples only are displayed along the discordia line (samples 1 and 7) with variable $^{207}\text{Pb}/^{235}\text{U}$ ratios. 6 samples yield an age of 2046.8 ± 1.1 Ma, calculated using a ^{207}Pb - ^{206}Pb isochron (2σ , MSWD = 2.2).

The samples have homogeneous U contents of ca. 200 ppm and variable Pb contents from 70 to 314 ppm due to variable contribution of common lead (0 to 216 ppm). The variable contributions of common lead account for the large range of measured $^{206}\text{Pb}/^{204}\text{Pb}$ ratios from 38 to 47700. However, $^{207}\text{Pb}/^{206}\text{Pb}$ are homogeneous at ca. 0.126. The samples show essentially no variation in U and Pb contents at the exception of one extreme value (sample 4) with very high common lead (216 ppm), correlated to lower amounts of ^{206}Pb (78 at%) and ^{207}Pb (9.5 at%) and higher content in ^{208}Pb (13 at%) associated with high Th content (105 ppm) that could indicate sulphide inclusions. This sample has not been taken into account for age calculations.

4.3.2 CT3 (Ivory Coast)

In a $^{206}\text{Pb}/^{238}\text{U}$ vs. $^{207}\text{Pb}/^{235}\text{U}$ diagram, all data from the CT3 ferrotapiolite are concordant or very close to the concordia line as previously observed with CT1 sample. One sample only is displayed along the discordia line (samples 9) with a higher $^{207}\text{Pb}/^{235}\text{U}$ ratio. 6 samples yield an age of 2053.2 ± 1.3 Ma, calculated using a ^{207}Pb - ^{206}Pb isochron (2σ , MSWD = 2.2).

The samples have elevated homogeneous U content (294 to 321 ppm). Same applies to Pb values (107 to 118 ppm), which show variable contributions of common lead (0.08 to 2.40 ppm). The large range of $^{206}\text{Pb}/^{204}\text{Pb}$ ratios (2770 to 83700) is associated to the variable contribution of common lead. However, $^{207}\text{Pb}/^{206}\text{Pb}$ are homogeneous at ca. 0.127. One sample (sample 9) shows elevated ratios associated to higher $^{206}\text{Pb}/^{204}\text{Pb}$ (84000),

$^{207}\text{Pb}_{\text{rad}}/^{204}\text{Pb}_{\text{rad}}$ (11000), $^{208}\text{Pb}/^{204}\text{Pb}$ (40) ratios that might indicate the presence of micro-inclusions of different nature than CT1 samples that could not be identified by SEM. CT3 sample was sampled in the same area as CT1 and thus could display the same type of micro-inclusions.

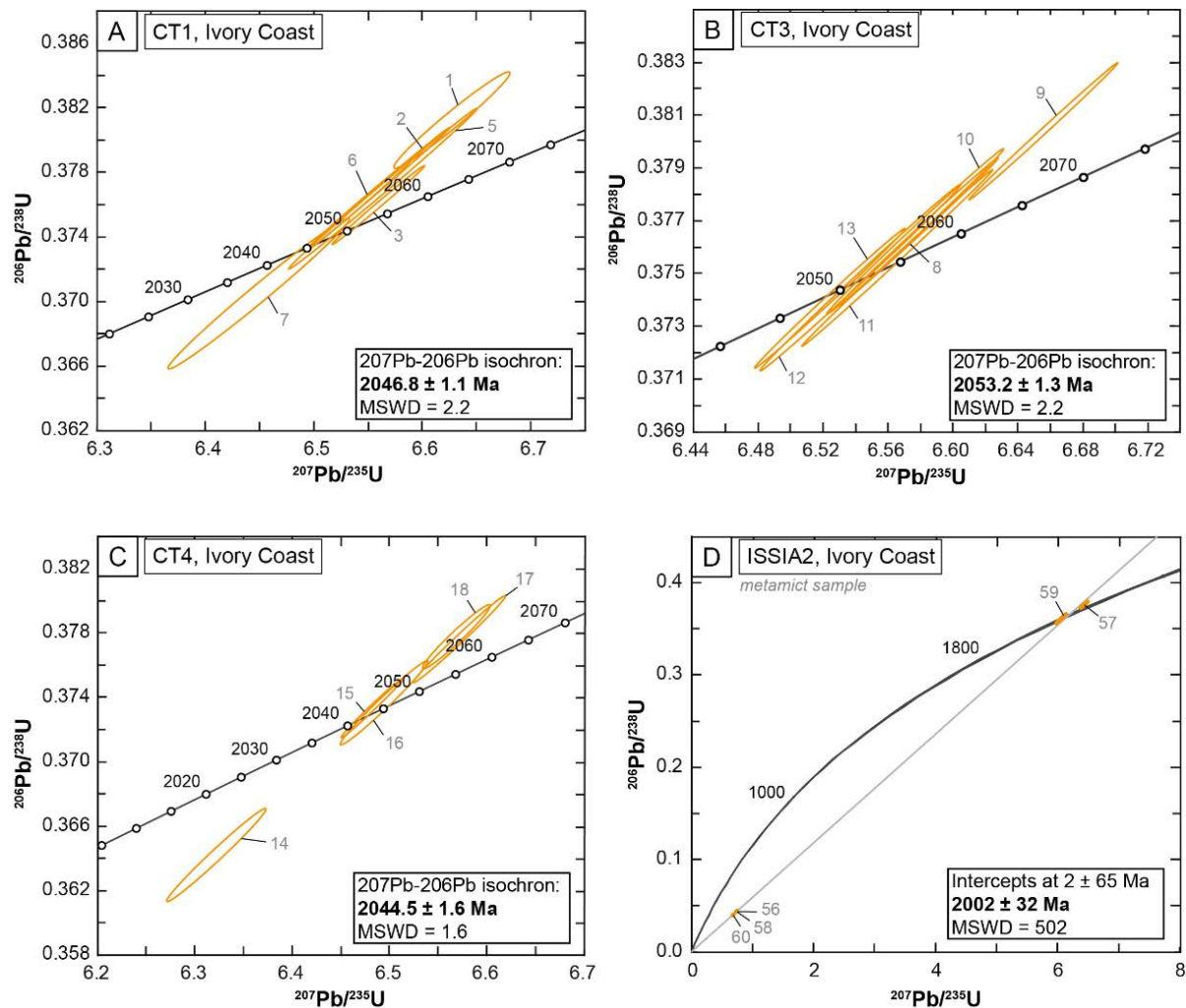


Figure 72. Concordia diagrams for four samples of columbo-tantalite from the Ivory Coast dated by ID-TIMS. (A) Six samples from the CT1 ferrotapiolite sample yield an age of $2046.8 \pm 1.1 \text{ Ma}$ (2σ). (B) Six samples from the CT3 ferrotapiolite sample yield an age of $2053.2 \pm 1.3 \text{ Ma}$ (2σ). (C) Five samples from the CT4 ferrotapiolite sample yield an age of $2044.5 \pm 1.6 \text{ Ma}$ (2σ). (D) Five samples from the ISSIA2 ferrotapiolite sample show the example of a metamict sample.

4.3.3 CT4 (Ivory Coast)

In a $^{206}\text{Pb}/^{238}\text{U}$ vs. $^{207}\text{Pb}/^{235}\text{U}$ diagram, all data from the CT4 ferrotapiolite are on the discordia line which inclination is influenced by the anomalous sample 14. 5 samples yield an age of 2044 ± 1.6 Ma, calculated using a ^{207}Pb - ^{206}Pb isochron (2σ , MSWD = 1.6).

The samples have homogeneous U contents (169 to 181 ppm) as well as Pb contents (62 to 68 ppm) with variable contents in common lead (0.3 to 4.2 ppm). The large range of common lead values is correlated to the variable $^{206}\text{Pb}/^{204}\text{Pb}$ ratios (910 to 11100). All samples are clustered while sample 14 shows elevated common lead (4.2 ppm). However, $^{207}\text{Pb}/^{206}\text{Pb}$ ratios are homogeneous at ca. 0.126.

4.3.4 ISSIA2 (Ivory Coast)

The samples have highly variable U and Pb contents (172 to 990 ppm and 10 to 469 ppm respectively). These variations in Pb are correlated to a variable common lead contribution between 2 to 17 ppm. $^{206}\text{Pb}/^{204}\text{Pb}$ ratios are also very variable but anticorrelated to previous parameters (141 to 1290). Moreover, samples display a variable enrichment in Th (3 to 890 ppm). This sample is an example of metamictization.

In a $^{206}\text{Pb}/^{238}\text{U}$ vs. $^{207}\text{Pb}/^{235}\text{U}$ diagram, the samples display two groups: two samples (samples 57 and 59) close to the concordia line and three samples (samples 56, 58 and 60) on the discordia line, closer to the origin. The discordia is rooted at the origin of the diagram. The high variability of content correlates to the poor metallic aspect of grains after cleaning. Grains clearly reflect metamict zones in the grains in the form of alteration and overgrowths of manganocolumbite. The metamictization allows lead to migrate, increasing common lead content. It has already been observed in columbo-tantalite but also wolframite and zircons (Mezger et al., 1997; Harlaux et al., 2017). Due to the mobilization of lead, the MSWD is very high and the intercept is probably too low. This sample comes from the same area as CT1, CT3 and CT4. The metamictization and the associated uncertainties might involve the calculation of an age younger than reality.

4.3.5 Rongi (Rwanda)

In a $^{206}\text{Pb}/^{238}\text{U}$ vs. $^{207}\text{Pb}/^{235}\text{U}$ diagram, all selected data from the Rongi ferrocolumbite are sub-concordant and clustered close to the concordia line. Samples 19, 22 and 25 have been removed from calculations to avoid an overly large effect on the discordia intercept. Given the selected data that are whether concordant or sub-concordant, the intercept varies from -950 to -800 with a meaningless error as large as the value of the intercept. The elevated $^{207}\text{Pb}/^{235}\text{U}$ ratios might be due to an underestimation of the initial $^{207}\text{Pb}/^{204}\text{Pb}$ ratio. However, four samples yield a discordant age of 931.5 ± 2.5 Ma (2σ) with a very low MSWD of 0.82.

The samples have low and variable U and Pb contents of 60 to 100 ppm and 8 to 236 ppm respectively. Samples show variable contributions of common lead from 0.3 to 232 ppm that account for the variable $^{206}\text{Pb}/^{204}\text{Pb}$ ratios (22 to 2740). Compared to samples from Ivory Coast, samples from Rongi display lower $^{206}\text{Pb}/^{204}\text{Pb}$ ratios due to higher values in common lead (0.3 to 15.8 ppm). Sample 20 displays a very high common Pb contribution and low $^{206}\text{Pb}/^{204}\text{Pb}$ ratio and therefore, was used to estimate initial lead ratios. Sample 20, as samples 22 and 25 show high Th content (2 to 546 ppm) that are not usual in columbo-tantalites and might reflect the presence of remaining micro-inclusions of sulphides.

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Table 15. U-Pb ID-TIMS analytical results for columbo-tantalites

Sample ^a	Weight (mg)	Concentrations (ppm)		²⁰⁶ Pb	Common lead (pg)	Radiogenic Pb (at%) ^c			Th/U ^d	Atomic ratios ^c							Apparent ages (Ma) ^e			
				²⁰⁴ Pb		²⁰⁶ Pb	²⁰⁷ Pb	²⁰⁸ Pb		²⁰⁶ Pb		²⁰⁷ Pb	²⁰⁷ Pb		Error corr		²⁰⁶ Pb	²⁰⁷ Pb	²⁰⁷ Pb	
		U	Pb								Err (2σ)		Err (2σ)							
				Measured ratios ^b						²³⁸ U		²³⁵ U		²⁰⁶ Pb			²³⁸ U	²³⁵ U	²⁰⁶ Pb	
CT1, Ivory Coast, Ferrotapiolite																				
1	0.200	181	70.5	1100	13500	88.55	11.16	0.282	0.0100	.38121	0.64	6.6273	0.66	0.12609	0.14	0.9774	2082	2063	2044	
2	0.210	201	73.3	33700	17	88.76	11.23	0.004	0.0001	.37597	0.53	6.5594	0.53	0.12654	0.04	0.9965	2057	2054	2050	
3	0.292	200	72.8	47700	8	88.80	11.20	0.002	0.0001	.37590	0.62	6.5406	0.61	0.12620	0.05	0.9970	2057	2051	2046	
4	0.213	202	314	38.84	4670000	f	f	f												
5	0.267	191	70.1	13200	99	88.76	11.21	0.034	0.0012	.37699	1.08	6.5638	1.08	0.12628	0.07	0.9977	2062	2054	2047	
6	0.560	194	71.1	23890	31	88.79	11.20	0.009	0.0004	.37691	0.84	6.5584	0.84	0.12620	0.05	0.9985	2062	2054	2046	
7	0.585	208	77.4	1770	6400	88.65	11.19	0.158	0.0056	.37046	1.03	6.4493	1.06	0.12626	0.16	0.9890	2032	2039	2046	
CT3, Ivory Coast, Ferrotapiolite																				
8	0.306	309	113	49100	19	88.75	11.24	0.005	0.0002	.37658	0.61	6.5786	0.61	0.12670	0.04	0.9983	2060	2057	2053	
9	0.458	321	118	83700	7	88.73	11.26	0.005	0.0002	.38039	0.56	6.6558	0.56	0.12690	0.03	0.9984	2078	2067	2056	
10	0.504	304	111	65000	10	88.76	11.24	0.002	0.0001	.37660	0.68	6.5769	0.68	0.12666	0.05	0.9976	2060	2056	2052	
11	0.182	312	113	14100	226	88.73	11.25	0.230	0.0008	.37559	0.73	6.5659	0.73	0.12679	0.04	0.9987	2056	2055	2054	
12	0.158	306	114	2770	5740	88.66	11.22	0.118	0.0042	.37484	0.77	6.5431	0.77	0.12660	0.05	0.9978	2052	2052	2051	
13	0.212	294	107	11900	286	88.74	11.23	0.290	0.0010	.37406	0.58	6.5244	0.58	0.12650	0.04	0.9975	2049	2049	2050	
CT4, Ivory Coast, Ferrotapiolite																				
14	0.376	181	68.2	909	18000	88.51	11.14	0.349	0.0124	.36418	0.65	6.3222	0.66	0.12591	0.11	0.9866	2002	2022	2042	
15	0.141	169	62.3	3360	1180	88.73	11.17	0.910	0.0032	.37330	0.40	6.4817	0.40	0.12593	0.05	0.9920	2045	2043	2042	
16	0.168	176	63.9	11100	116	88.78	11.19	0.230	0.0008	.37365	0.57	6.4946	0.57	0.12606	0.10	0.9834	2047	2045	2044	
17	0.189	177	65.1	4010	924	89.47	11.29	0.761	0.0267	.37763	0.59	6.5722	0.59	0.12622	0.07	0.9919	2065	2056	2046	
18	0.238	175	64.4	8190	216	88.77	11.19	0.034	0.0012	.37778	0.43	6.5688	0.43	0.12611	0.10	0.9708	2066	2055	2044	
Rongi, Rwanda, Ferrocolumbite																				
19	0.227	59.5	8.25	1090	275	93.54	6.433	0.031	0.0010	.14127	0.66	1.33961	0.76	0.06877	0.04	0.8678	852	863	892	
20	0.239	74.0	236	22.10	5416000	g	g	g												
21	0.122	99.8	14.8	1710	367	93.40	6.561	0.040	0.0013	.15422	0.63	1.49381	0.70	0.07025	0.32	0.8915	925	928	936	
22	0.112	91.4	27.8	83.04	225000	92.64	6.845	0.519	0.0173	.15152	0.84	1.54364	2.63	0.07389	2.48	0.33	910	948	1038	
23	0.231	74.2	11.3	862	275	93.43	6.555	0.017	0.0005	.15294	1.18	1.47956	1.27	0.07016	0.44	0.9382	917	922	933	
24	0.308	85.4	14.8	328	8000	93.25	6.576	0.171	0.0057	.15263	2.09	1.48410	2.12	0.07052	0.32	0.9885	916	924	944	
25	0.210	77.6	26.1	68.37	249000	91.99	6.607	1.407	0.0476	.14271	1.15	1.41333	2.16	0.07183	1.90	0.47	860	895	981	
26	0.231	80.5	11.8	2740	94	93.41	6.548	0.040	0.0013	.15559	0.72	1.50388	0.73	0.07010	0.13	0.9836	932	932	931	

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NP2, Fujian, China, Ferrocolumbite

27	0.216	248	14.7	904	1130	94.78	5.141	0.080	0.0026	.06032	0.60	0.45112	0.77	0.05424	0.47	0.7888	378	378	381
28	0.194	272	16.2	716	2160	94.84	5.121	0.040	0.0013	.06003	0.65	0.44693	1.68	0.05399	1.54	0.40	376	375	371
29	0.295	211	12.0	1010	615	95.37	5.029	0.399	0.0131	.05865	1.10	0.42643	7.23	0.05273	7.13	0.18	367	361	317
30	0.316	136	12.3	1730	229	94.83	5.136	0.030	0.0010	.09566	1.05	0.71432	1.07	0.05416	0.21	0.9800	589	547	378
31	0.271	322	15.6	1310	634	94.83	5.128	0.040	0.0013	.05085	0.78	0.37916	0.89	0.05408	0.42	0.8800	320	326	374
32	0.193	235	14.3	730	1600	94.64	5.195	0.164	0.0051	.06089	0.64	0.46079	0.73	0.05489	0.34	0.8831	381	385	408
33	0.206	276	15.9	1220	751	94.77	5.139	0.092	0.0028	.05999	0.70	0.44852	0.72	0.05423	0.16	0.9757	376	376	380
34	0.201	241	13.9	1390	446	94.78	5.145	0.075	0.0026	.06052	0.71	0.45300	0.74	0.05428	0.22	0.9558	379	379	383

Buranga, Rwanda, Ferrocolumbite

35	0.234	84.4	12.1	1610	278	93.49	6.480	0.025	0.0010	.14946	0.71	1.42838	0.86	0.06931	0.49	0.8254	898	901	908
36	0.196	163	76.5	48.22	310400	91.66	6.503	1.835	0.0058	.13569	1.73	1.32727	3.97	0.07095	3.73	0.35	820	858	956
37	0.232	80.3	11.3	5350	23	93.54	6.457	0.001	0.0000	.15135	1.18	1.44057	1.24	0.06903	0.39	0.9500	909	906	900
38	0.265	89.8	16.0	262	13700	93.40	6.581	0.015	0.0004	.14927	1.35	1.45021	1.58	0.07046	0.79	0.8660	870	910	942
39	0.348	74.5	10.6	4430	30	93.49	6.475	0.039	0.0013	.15218	1.04	1.45326	1.06	0.06926	0.21	0.9795	913	911	906
40	0.379	67.6	9.49	6450	11	93.46	6.478	0.061	0.0021	.15108	1.16	1.44379	1.18	0.06931	0.18	0.9886	907	907	908
41	0.378	84.0	11.7	7800	11	93.45	6.493	0.056	0.0019	.14442	1.20	1.38367	1.20	0.06949	0.11	0.9959	870	882	913

NT-2, Altai, Ferrotantalite

42	0.319	218	13.2	534	2380	94.87	5.131	0.002	0.2316	.05885	0.89	0.43878	0.94	0.05408	0.27	0.9571	369	369	374
43	0.243	142	8.86	341	2390	94.80	5.124	0.074	0.3719	.05665	0.82	0.42216	1.02	0.05405	0.56	0.8352	355	358	373
44	0.434	149	9.86	314	3400	94.78	5.136	0.081	0.4052	.05948	1.28	0.44438	1.36	0.05418	0.42	0.9504	373	373	379
45	0.242	154	8.99	713	648	94.84	5.134	0.029	0.1729	.05851	0.98	0.43673	1.04	0.05413	0.31	0.9533	367	368	377
46	0.208	231	40.9	49.01	765000	92.90	5.122	1.979	3.8079	.06174	1.24	0.46931	3.93	0.05514	3.77	0.28	386	391	418
47	0.265	174	10.2	923	519	94.81	5.147	0.043	0.0014	.06037	1.63	0.45190	1.63	0.05429	0.15	0.9957	378	379	383
48	0.250	166	10.3	479	1750	94.78	5.174	0.049	0.0016	.05957	0.57	0.44837	0.69	0.05459	0.38	0.8364	373	376	396

A-1, Altai, Ferrotantalite

49	0.207	81.3	2.92	274	393	94.88	4.741	0.381	0.0127	.03117	1.24	0.21477	1.68	0.04997	1.07	0.7696	198	198	194
50	0.233	304	9.96	454	1900	95.02	4.758	0.220	0.0073	.03120	1.62	0.21544	1.70	0.05008	0.40	0.9713	198	198	199
51	0.199	78.3	15.9	1220	756	95.04	4.752	0.204	0.0067	.21185	1.96	1.46057	1.98	0.05000	0.23	0.9934	1239	914	195
52	0.199	274	11.3	1530	253	95.05	4.752	0.201	0.0066	.04370	0.68	0.30122	0.70	0.05000	0.17	0.9691	276	267	195
53	0.184	213	13.0	305	6500	95.01	4.740	0.251	0.0083	.05436	0.70	0.37390	0.92	0.04989	0.56	0.7979	341	323	190
54	0.202	42.1	17.8	1010	1400	95.04	4.750	0.206	0.0068	.04363	0.72	0.30070	0.75	0.04998	0.18	0.9695	275	267	194
55	0.145	515	16.0	703	2210	95.05	4.765	0.184	0.0061	.03129	1.00	0.21624	1.06	0.05013	0.32	0.9522	199	199	201

ISSIA2, Ivory Coast, Manganocolumbite

56	0.275	344	21.9	141	49100	87.97	10.82	1.218	0.0433	.04440	0.92	0.75280	1.00	0.12297	0.40	0.9178	280	570	2000
57	0.191	990	469	1290	284000	70.89	8.810	20.29	0.8976	.37592	0.69	6.44125	0.70	0.12427	0.08	0.9936	2057	2038	2018
58	0.204	172	9.61	199	5500	88.44	10.80	0.761	0.0269	.04360	0.87	0.73410	0.92	0.12211	0.30	0.9463	275	559	1987

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59	0.200	436	160	1130	66400	88.97	10.84	0.19	0.0067	.36082	1.09	6.06039	1.09	0.12182	0.06	0.9982	1986	1985	1983
60	0.194	575	24.5	634	4600	88.76	10.92	0.319	0.0112	.04013	1.02	0.68079	1.03	0.12303	0.11	0.9946	254	527	2001

CC1716, France, Ferrocolumbite

61	0.151	9.66	24.3	7520	49	94.44	4.999	0.566	0.0188	2.7446	1.10	20.0333	1.10	0.05294	0.07	0.9978	8511	3093	326
62	0.122	262	23.6	9620	28	94.46	5.002	0.541	0.0179	.04996	0.85	0.36473	0.85	0.05295	0.10	0.9937	314	316	327
63	0.126	54500	2523	327000	270	92.11	4.877	3.011	0.1025	.04961	2.51	0.36216	2.51	0.05295	0.03	0.9999	312	314	326
64	0.121	532	30.5	394	22400	94.31	4.995	0.698	0.0232	.05322	0.66	0.38861	0.80	0.05296	0.43	0.8463	334	333	327
65	0.115	14800	748	1840	702000	91.75	4.853	3.402	0.1163	.05185	0.85	0.37817	0.86	0.05290	0.09	0.9943	326	326	324

- a** Small fragments from single columbite grains. Fragments were selected to show only fresh fracture surfaces. All samples were leached with 20% HF, 6N HCl and 7N HNO₃ before sample dissolution (Romer and Smeds, 1996). After leaching, most grains had shiny metallic surfaces no traces of sulphides, silicate inclusions and metamict domains. Rare grains developed rusty stains during the HCl and HNO₃ washing stages, indicating that metamict domains were not completely removed. As such grains typically yield variably discordant data, they were removed from the sample. Although the analyzed fragments eventually had perfect surfaces, it is unclear whether the metamict domains had been removed completely also from interior part accessible to leaching. The high discordance of some samples indicates that this apparently was not always possible.
- b** Lead isotope ratios corrected for fractionation, blank and isotopic tracer. Samples were analyzed at GFZ German Research Centre for Geosciences, Potsdam, Germany, using a ²⁰⁵Pb-²³⁵U mixed isotopic tracer. Analytical details are given in Baumgartner et al. (2006). During the measurement period total blanks were less than 15 pg, for lead and less than 1 pg for uranium.
- c** Lead corrected for fractionation, blank, isotopic tracer, and initial lead with the composition according to Stacey and Kramers (1975).
- d** ²³²Th/²³⁸U calculated from radiogenic ²⁰⁸Pb/²⁰⁶Pb and the age of the sample.
- e** Apparent ages were calculated using the constants recommended by IUGS. $\lambda_{238} = 1.55125 \text{ E-10 y}^{-1}$, $\lambda_{235} = 9.848 \text{ E-10 y}^{-1}$.
- f** Sample dominated by common lead; measured ²⁰⁶Pb/²⁰⁴Pb = 38.84, ²⁰⁷Pb/²⁰⁴Pb = 18.21, ²⁰⁸Pb/²⁰⁴Pb = 38.89.
- g** Sample dominated by common lead; measured ²⁰⁶Pb/²⁰⁴Pb = 22.10, ²⁰⁷Pb/²⁰⁴Pb = 15.92, ²⁰⁸Pb/²⁰⁴Pb = 38.91.
- Data in italic were removed for age calculation.

4.3.6 Buranga (Rwanda)

In a $^{206}\text{Pb}/^{238}\text{U}$ vs. $^{207}\text{Pb}/^{235}\text{U}$ diagram, four samples from the Buranga ferrocolumbite are concordant and one (sample 41) is on the discordia line. The four samples yield a concordant age of 905.2 ± 3.2 Ma (2σ , MSWD = 0.41).

The samples have rather homogeneous U and Pb contents of 74 to 84 ppm and 10 to 12 ppm respectively with a variable common lead contribution from 0.1 to 56 ppm. Overall, the common lead contribution is lower than in other samples of this study. Two samples (samples 36 and 38) display these extreme common lead contributions, associated to higher contents in U and ^{208}Pb , lower $^{206}\text{Pb}/^{204}\text{Pb}$ but no Th enrichment. Therefore, these samples do not seem to reflect the occurrence of remaining micro-inclusions but maybe metamictization. However, samples 36 and 38 were not included in age calculations.

4.3.7 NT-2 (Altai)

In a $^{206}\text{Pb}/^{238}\text{U}$ vs. $^{207}\text{Pb}/^{235}\text{U}$ diagram, six samples from the NT-2 ferrotantalite are aligned on the discordia line which five are subconcordant. The discordia is sub-parallel to the concordia line. Therefore, the extreme sample 43 controls the inclination of the discordia and may induce a little error in the age calculation. No concordant age could be calculated but the homogeneous ^{207}Pb - ^{206}Pb ratios display an isochron age of 372.0 ± 2.3 Ma (2σ , MSWD = 2.1).

The samples have variable U contents from 142 to 218 ppm and low Pb content from 9 to 13 ppm with high common lead contributions between 0.7 to 1.8 ppm. One sample (sample 46) has higher abnormal U and Pb contents of 231 and 71 ppm respectively with a common lead contribution of 28 ppm associated to higher ^{208}Pb content. This sample does not seem to reflect the presence of micro-inclusions but maybe occurrence of metamictization. This sample has been removed from age calculations.

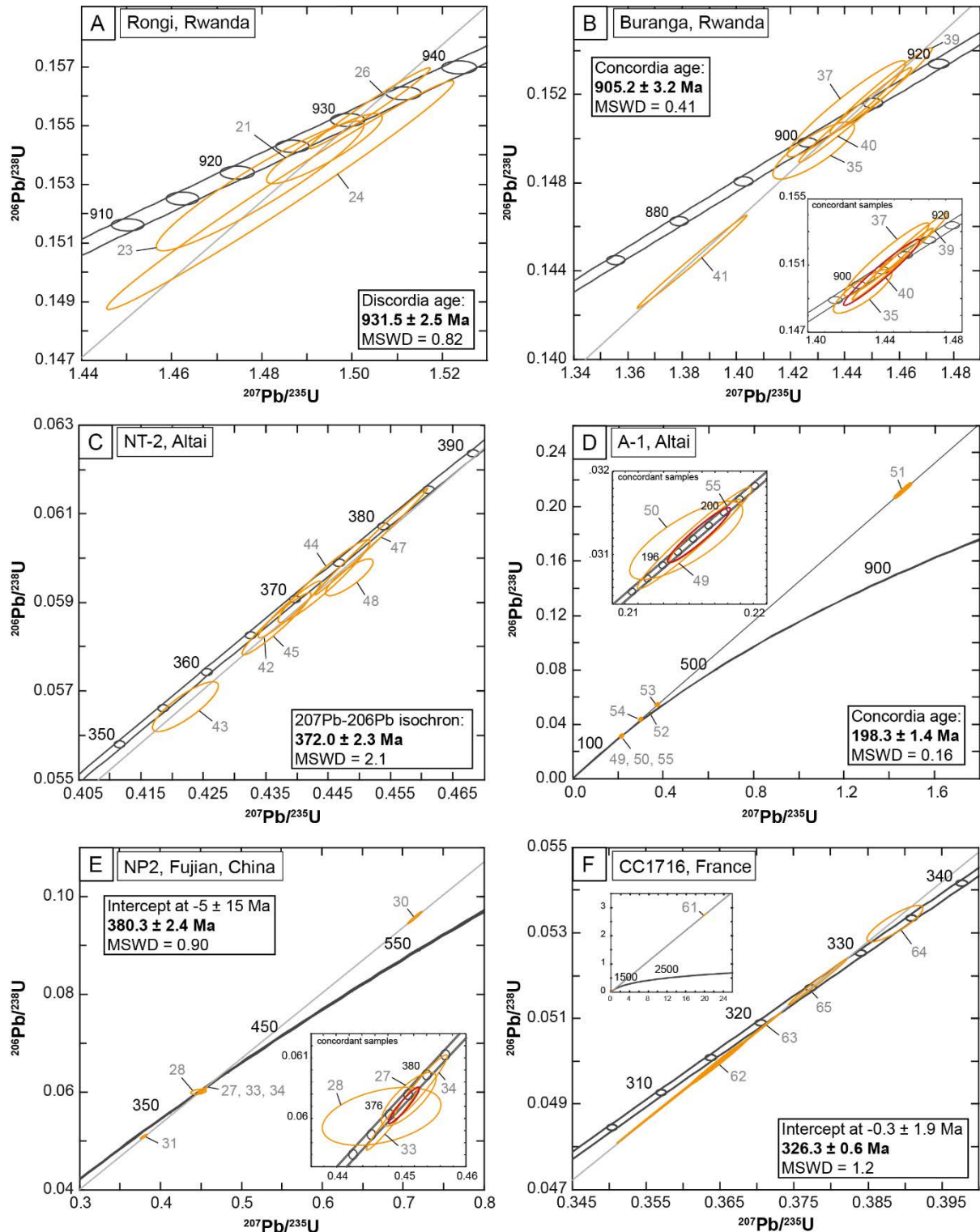


Figure 73. Concordia diagrams for six samples of columbo-tantalite from Rwanda, Altai, China and France dated by ID-TIMS. (A) Four samples from the Rongi ferrocolumbite sample yield an age of 931.5 ± 2.5 Ma (2σ). (B) Four samples from the Buranga ferrocolumbite sample yield an age of 905.2 ± 3.2 Ma (2σ). (C) Six samples from the NT-2 ferrotantalite sample yield an age of 372.0 ± 2.3 Ma (2σ). (D) Three samples from the A-1 ferrotantalite sample yield an age of 198.3 ± 1.4 Ma (2σ). (E) Six samples from the NP2 ferrotantalite sample yield an age of 380.3 ± 2.4 Ma (2σ). (F) Four samples from the CC1716 ferrocolumbite sample yield an age of 326.3 ± 0.6 Ma (2σ).

4.3.8 A-1 (*Altai*)

In a $^{206}\text{Pb}/^{238}\text{U}$ vs. $^{207}\text{Pb}/^{235}\text{U}$ diagram, all samples from the A-1 manganotantalite display a reverse discordia rooted at the origin. Three samples give a concordant age of 198.3 ± 1.4 Ma (2σ , MSWD = 0.16).

The samples have variable contents in U (42 to 515 ppm) and Pb (3 to 18 ppm) with variable but not excessive contribution of common lead (0.5 to 2.5 ppm). This variability is clearly associated to a uranium fractionation during a remobilizing process. Uranium was remobilized and created metamict zones in the grains. However, Th/U ratios are low and show little variability (<0.013).

4.3.9 NP2 (*Fujian, China*)

In a $^{206}\text{Pb}/^{238}\text{U}$ vs. $^{207}\text{Pb}/^{235}\text{U}$ diagram, four samples from the NP2 ferrotantalite are clustered on the concordia and two samples are respectively directing the discordia line at lower (sample 31) and higher (sample 30) values. These samples have, respectively, the highest and lowest overall Pb and U contents. The discordia intercept is interpreted as rooted at the origin. Six samples yield a discordia age of 380.3 ± 2.4 Ma (2σ , MSWD = 0.9).

The samples have variable U contents from 136 to 322 ppm. Pb content is less variable and low from 12 to 16 ppm with variable common lead contributions. Two samples (samples 29 and 32) have abnormally elevated ^{208}Pb contents that are not related to Th content or any other parameter. These samples have been removed from age calculations.

4.3.10 CC1716 (*France*)

In a $^{206}\text{Pb}/^{238}\text{U}$ vs. $^{207}\text{Pb}/^{235}\text{U}$ diagram, all samples from the CC1716 ferrocolumbite display a discordia line rooted at the origin. Samples are dispersed on the discordia line. This line is subparallel to the Concordia but is however well constraint with an age (over all samples) of 326.3 ± 0.6 Ma (2σ , MSWD = 1.2).

The samples have highly variable U (10 to 54500 ppm) and Pb (24 to 2500 ppm) contents. The common lead contribution is also highly variable from 0.1 to 26 ppm anti-

correlated to variations of $^{206}\text{Pb}/^{204}\text{Pb}$ ratios. Extreme values in U and Pb contents are anti-correlated to extreme values of common lead contributions. Common lead contribution is also anticorrelated to enrichments in Th which is high in samples 63 and 65 (5600 and 1700 ppm respectively). These samples show the presence of heterogeneous rims around the grains and the isotopic results seem to suggest the occurrence of inclusions.

4.4 SIMS dating and calibration

4.4.1 Matrix effects

The ^{206}Pb , ^{238}U and ^{254}UO species are chosen for the $^{206}\text{Pb}/^{238}\text{U}$ calibration. Depending on their chemical composition, each sample yield a linear relationship between UO/U and $^{206}\text{Pb}/^{238}\text{U}$ with the following equations (Figure 74):

Ferrotapiolite: $^{206}\text{Pb}/^{238}\text{U} = 1.5474 (^{254}\text{UO}/^{238}\text{U}) - 7.6670$

Ferrocolumbite: $^{206}\text{Pb}/^{238}\text{U} = 1.4493 (^{254}\text{UO}/^{238}\text{U}) - 23.835$

Ferrotantalite: $^{206}\text{Pb}/^{238}\text{U} = 0.2735 (^{254}\text{UO}/^{238}\text{U}) - 0.8888$

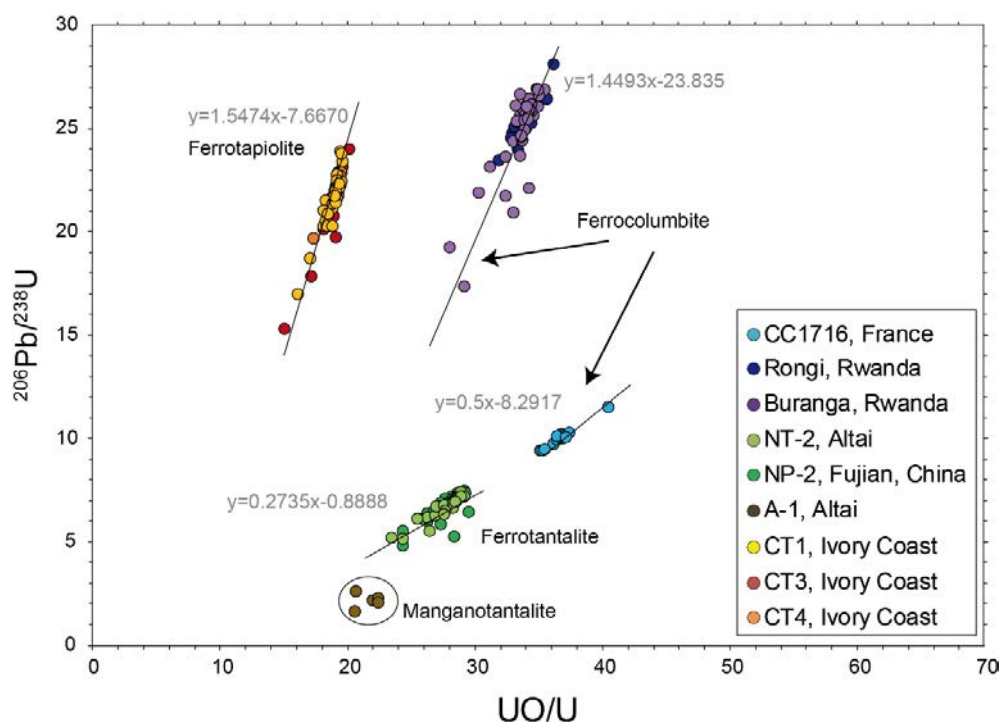


Figure 74. $^{206}\text{Pb}/^{238}\text{U}$ vs UO/U pairs of measured samples according to their chemical composition. Each group can be defined by a linear relation corresponding to the SIMS calibration line. This line depends on the composition and of age of the samples.

These equations can be repeatedly obtained from columbo-tantalites from the same compositional group. The $^{206}\text{Pb}/^{238}\text{U}$ ratio of the potential standard known, the U/Pb calibration can be defined from the appropriate equation. Thus, any $^{206}\text{Pb}/^{238}\text{U}$ ratio from a sample of the same composition can be calculated from measured $^{206}\text{Pb}/^{238}\text{U}$ and $^{254}\text{UO}/^{238}\text{U}$ pairs (Zhu et al., 1998). However, the equation of correlation is also function of the age of the analyzed sample according to the following equation:

$$(^{206}\text{Pb}/^{238}\text{U})_{\text{sample}} = k a (^{254}\text{UO}/^{238}\text{U})_{\text{sample}} + k b$$

Where $k = (^{206}\text{Pb}/^{238}\text{U})_{\text{sample}} / (^{206}\text{Pb}/^{238}\text{U})_{\text{standard}}$ and a and b the intercept and slope of the standard calibration line.

This factors explains the different line of correlation between on one side, Rongi and Buranga samples which yield ages around 900 Ma and on the other side, CC1716 sample which is dated round 330 Ma.

If the Ta # seems critical in the calibration of samples, the Mn #, which is the second compositional factor of columbo-tantalite, does not induce matrix effects. Indeed, the Rongi and Buranga samples have a Mn # of 0.33 and 0.47 respectively and the same Ta # and yield the same line of calibration.

4.4.2 Ferrotapiolite dating results (CT1, CT3, CT4)

A total of eighty-five analyses were performed on each sample. The results are listed in the Appendix B and the age calculation diagrams are shown on Figure 75. Three samples of ferrotapiolite were measured: CT1, CT3 and CT4, all sampled in Ivory Coast. CT1 sample was used as an external standard to measure CT3 and CT4 U-Pb isotopic ratios. Ratios have been plotted in concordia diagrams and yields two discordia lines with sub-concordant ellipses. Thirty-six measurements on CT3 sample define a discordia line with concordia intercept at 2050.8 ± 3.4 Ma (1σ , MSWD = 0.95). Three measurements only are located on the discordia line while other measurement are all on the concordia. This age is consistent with the ID-TIMS age of 2053.2 ± 1.3 Ma.

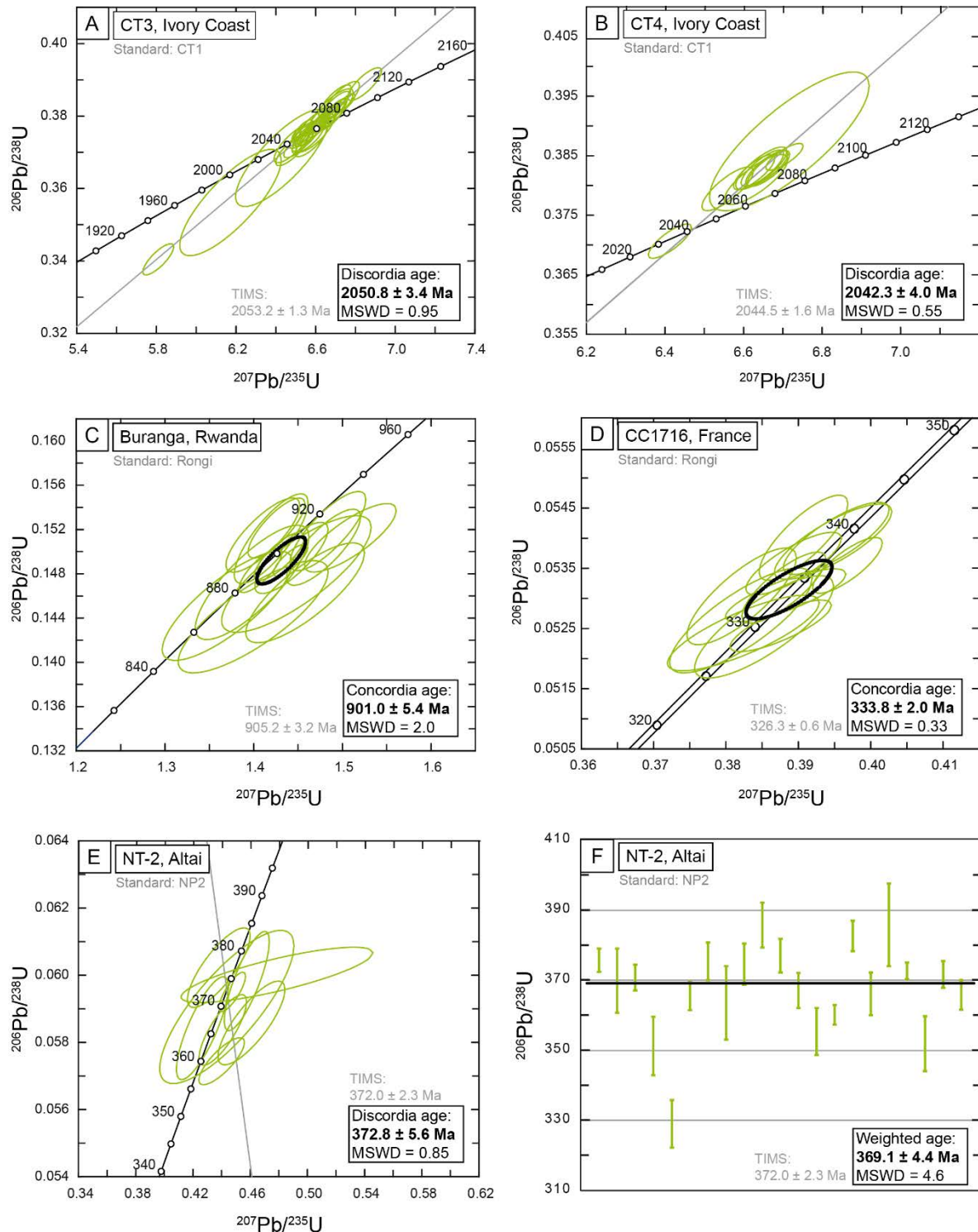


Figure 75. Concordia diagrams for two samples of Ferrotapiolite CT3 and CT4 calculated with CT1 as external standard. (A) Thirty-six measurements of the CT3 ferrotapiolite sample yield an age of $2050.8 \pm 3.4 \text{ Ma}$ (1 σ). (B) Ten measurements of the CT4 ferrotapiolite sample yield an age of $2042.3 \pm 4.0 \text{ Ma}$ (1 σ). Concordia diagrams for two samples of Ferrocolumbite Buranga and CC1716 calculated with Rongi as external standard. (C) Fifteen measurements of the Buranga ferrocolumbite sample yield an age of $901.0 \pm 5.4 \text{ Ma}$ (1 σ). (D) Twelve measurements of the CC1716 ferrocolumbite sample yield an age of $333.8 \pm 2.0 \text{ Ma}$ (1 σ). (E) Concordia diagram of the NT-2 Ferrotantalite calculated with NP2 as external standard and yielding an age of $372.8 \pm 5.6 \text{ Ma}$ (1 σ). (F) Weighted age diagram of the NT-2 Ferrotantalite calculated from all analyses with NP2 as external standard and yielding an age of $369.1 \pm 4.4 \text{ Ma}$ (1 σ).

Ten measurements were performed on the CT4 sample and are displayed along a discordia line which intercepts the concordia at 2042 ± 4.0 (1σ , MSWD = 0.55). One measurement is located on the concordia line while the nine others are sub-concordant, above the concordia line. This age matches the ID-TIMS measurements that yielded an age of 2044.5 ± 1.6 Ma.

4.4.3 Ferrocolumbite dating results

A total of sixty-three analyses were performed on each sample. The results are listed in the Appendix B and the age calculation diagrams are shown on Figure 75. Three samples of ferrocolumbite were analysed: Rongi, Burang and CC1716. The Rongi sample was used as an external standard to calibrate and calculate the isotopic ratios of Buranga and CC1716 samples. Both samples yield concordia ages.

Fifteen measurement of the Buranga sample (over seventeen) yield an age of 901.0 ± 5.4 Ma (1σ , MSWD = 2.0) in accordance with ID-TIMS measurements (905.2 ± 3.2 Ma). The two discriminated measurement did not permit to calculate a concordia age but are subconcordant. The totality of the measurements also yield a discordia age of 901.0 ± 5.4 Ma (1σ , MSWD = 0.90).

Twelve measurements (over fifteen) of the CC1716 sample yield an age of 333.8 ± 2.0 Ma (1σ , MSWD = 0.33). All measurement display sub-concordant ellipses. Among the two discriminated measurements, one was too low under the concordia line to calculate a concordant age and the second had a very large error. However, the totality of the measurement also yield a discordia age of 334.7 ± 1.7 Ma (1σ , MSWD = 1.3). This age is a little older than the ID-TIMS calculated age (326.3 ± 0.6 Ma). This error can be due to younger age of this sample compared to the Rongi standard.

4.4.2 Ferrotantalite dating results

A total of forty-five analyses were performed on each sample. The results are listed in the Appendix B and the age calculation diagrams are shown on Figure 75. Two samples of ferrotantalite were measured: NP2 and NT-2. NP2 was used as external standard to calculate

the NT-2 isotopic ratios. Both sample have low ^{207}Pb content which induces a large error in a concordia diagram. Therefore, nearly half measurement could not be used for age calculation in this kind of diagram.

Nine measurements (over twenty) of the NT-2 sample are subconcordant and yield an age of 372.8 ± 5.6 Ma (1σ , MSWD = 0.85). This age is in accordance with ID-TIMS measurements (372.0 ± 2.3 Ma). Nevertheless, a weighted age calculation including all analyses seems more adequate for this sample and yields an age of 369.1 ± 4.4 Ma (1σ , MSWD = 4.6) which is also consistent with ID-TIMS measurements.

5. Conclusion

We conducted comprehensive U-Pb measurement with multiple techniques (ID-TIMS and SIMS) to evaluate columbo-tantalites from multiple locations. The data show age and compositional homogeneity in the scale of centimetric grains. Columbo-tantalite isotopic ratios are subjected to matrix effects which limit the accuracy of SIMS calibration. These matrix effects are controlled by the $\text{Ta}/(\text{Nb}+\text{Ta})$ ratio of the samples and the range of age calculated. Therefore, the CT1, CT3 and CT4 chemical and isotopically homogeneous ferrotapiolite are considered as suitable natural reference material for SIMS U-Pb analysis as well as Rongi and Buranga samples in the range of ferrocolumbite. NP2 and NT-2 ferrotantalite samples can also be considered as valuable reference materials with mean $^{206}\text{Pb}/^{238}\text{U}$ age calculations.

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Appendix A – EMPA analytical results for columbo-tantalites

Sample	TiO ₂	MnO	FeO	Nb ₂ O ₅	SnO ₂	ZrO ₂	Ta ₂ O ₅	Total	Mn#	Ta#
CT1, Côte d'Ivoire, Ferrotapiolite										
1	0.83	0.86	14.52	7.35	0.00	0.00	74.88	98.45	0.06	0.92
2	0.82	0.75	14.58	7.16	0.02	0.00	74.04	97.37	0.05	0.92
3	0.83	0.89	14.46	6.88	0.12	0.00	75.03	98.27	0.06	0.93
4	0.81	0.95	14.60	7.02	0.00	0.18	74.13	97.69	0.06	0.93
5	0.77	0.81	14.54	7.16	0.00	0.00	74.41	97.70	0.05	0.92
6	0.79	0.82	14.55	6.89	0.00	0.00	74.24	97.29	0.05	0.93
7	0.79	0.73	14.81	7.19	0.02	0.08	75.25	98.94	0.05	0.92
8	0.79	0.62	14.63	7.32	0.00	0.01	74.68	98.04	0.04	0.92
9	0.77	0.87	14.47	6.82	0.00	0.20	75.28	98.41	0.06	0.93
10	0.80	0.90	14.82	7.07	0.10	0.11	75.14	98.95	0.06	0.93
11	0.80	0.88	14.41	7.10	0.02	0.18	74.49	97.87	0.06	0.92
12	0.76	0.80	14.65	7.05	0.00	0.13	74.92	98.31	0.05	0.93
13	0.79	0.85	14.60	7.06	0.04	0.00	74.70	98.05	0.05	0.93
14	0.77	0.86	14.53	6.94	0.03	0.06	74.79	97.99	0.06	0.93
15	0.79	0.91	14.64	6.95	0.09	0.09	75.08	98.54	0.06	0.93
16	0.83	0.94	14.60	7.24	0.12	0.00	74.29	98.00	0.06	0.92
17	0.76	0.92	14.39	6.90	0.00	0.06	74.85	97.87	0.06	0.93
18	0.76	0.80	14.35	7.01	0.00	0.19	75.41	98.52	0.05	0.93
19	0.84	0.94	14.69	7.32	0.04	0.01	74.30	98.13	0.06	0.92
20	0.83	0.79	14.53	7.15	0.00	0.08	74.53	97.90	0.05	0.92
CT3, Côte d'Ivoire, Ferrotapiolite										
21	0.63	0.71	14.26	4.75	0.03	0.00	76.64	97.02	0.04	0.95
22	0.69	0.63	14.41	6.34	0.05	0.12	75.58	97.81	0.04	0.93
23	0.56	1.07	14.31	6.45	0.00	0.08	74.78	97.25	0.05	0.93
24	0.58	0.98	14.46	6.33	0.00	0.03	74.72	97.10	0.05	0.93
25	0.59	1.05	14.16	6.47	0.04	0.00	74.96	97.26	0.05	0.93
26	0.61	0.84	14.38	6.13	0.10	0.03	76.07	98.16	0.04	0.94
27	0.59	0.90	14.35	6.44	0.01	0.02	75.21	97.52	0.04	0.93
28	0.59	0.99	14.26	6.48	0.00	0.09	75.23	97.66	0.05	0.93
29	0.67	0.95	14.64	6.38	0.09	0.03	75.07	97.84	0.04	0.93
30	0.67	1.01	14.07	6.30	0.00	0.08	74.74	96.87	0.04	0.93
31	0.68	1.04	14.51	6.29	0.04	0.12	75.45	98.18	0.04	0.93
32	0.69	0.83	14.41	6.43	0.08	0.05	75.92	98.46	0.05	0.93
33	0.70	0.98	14.55	6.63	0.06	0.04	75.20	98.15	0.04	0.93
34	0.71	0.97	14.50	6.44	0.08	0.00	76.21	98.91	0.05	0.93
35	0.72	0.87	14.50	6.20	0.06	0.11	76.25	98.70	0.05	0.94
36	0.69	0.99	14.37	6.29	0.00	0.00	75.29	97.68	0.05	0.93
37	0.71	0.90	14.47	6.32	0.00	0.02	74.83	97.27	0.04	0.93
38	0.67	0.99	14.30	6.33	0.00	0.16	75.56	98.01	0.04	0.93
39	0.71	0.98	14.25	6.26	0.00	0.10	75.20	97.50	0.04	0.93
40	0.67	0.90	14.27	5.99	0.00	0.03	75.15	97.01	0.04	0.94
41	0.62	0.93	14.19	6.21	0.00	0.00	75.51	97.46	0.05	0.93
42	0.66	1.12	14.15	6.00	0.03	0.04	76.53	98.57	0.04	0.94
CT4, Côte d'Ivoire, Ferrotapiolite										
43	1.02	0.56	14.78	7.05	0.15	0.03	73.47	97.05	0.04	0.92
44	0.95	0.66	14.71	7.18	0.02	0.12	74.81	98.46	0.05	0.92

45	0.93	0.69	14.40	7.20	0.00	0.00	74.04	97.27	0.04	0.92
46	0.90	0.70	14.35	7.39	0.00	0.00	73.87	97.21	0.04	0.92
47	0.96	0.72	14.49	7.28	0.12	0.05	73.55	97.17	0.05	0.92
48	1.02	0.65	14.44	7.16	0.20	0.00	73.64	97.11	0.05	0.92
49	1.05	0.66	14.60	7.19	0.00	0.00	74.71	98.21	0.05	0.92
50	1.06	0.71	14.73	7.46	0.02	0.00	74.12	98.18	0.04	0.92
51	0.87	0.61	14.65	6.95	0.00	0.00	73.59	96.67	0.04	0.93
52	0.81	0.66	14.69	7.10	0.04	0.09	74.53	97.92	0.05	0.92
53	0.84	0.66	14.84	7.06	0.02	0.00	74.54	97.95	0.04	0.93
54	0.80	0.71	14.50	7.38	0.21	0.13	74.33	98.06	0.04	0.92
55	0.92	0.57	14.83	7.27	0.19	0.23	74.13	98.14	0.04	0.92
56	0.95	0.70	14.76	7.63	0.00	0.00	74.20	98.24	0.05	0.92
57	1.04	0.71	14.61	7.48	0.00	0.00	73.64	97.48	0.04	0.92
58	1.01	0.72	14.65	7.34	0.00	0.03	74.46	98.23	0.05	0.92
59	0.95	0.62	14.76	7.51	0.06	0.21	75.06	99.17	0.05	0.92
60	0.94	0.58	14.65	7.31	0.06	0.00	74.71	98.25	0.05	0.92
61	1.15	0.69	14.69	7.65	0.15	0.15	73.92	98.40	0.04	0.92
62	0.96	0.63	14.61	7.36	0.07	0.19	73.45	97.30	0.04	0.92
63	0.89	0.68	14.43	6.29	0.10	0.22	75.47	98.07	0.04	0.93
64	0.86	0.66	14.35	7.12	0.00	0.00	75.64	98.64	0.04	0.93
65	0.83	0.56	13.14	6.52	0.05	0.00	75.88	97.03	0.05	0.93
66	0.93	0.64	14.46	7.52	0.12	0.00	74.22	97.89	0.04	0.92
67	0.95	0.70	14.50	7.31	0.10	0.00	75.08	98.63	0.04	0.92
68	0.62	0.58	14.49	5.81	0.28	0.07	76.50	98.34	0.04	0.92

Rongi, Rwanda, Ferrocolumbite

69	0.17	6.84	13.80	66.22	0.11	0.03	11.49	98.66	0.33	0.17
70	0.22	5.68	14.53	57.76	0.00	0.10	11.12	89.41	0.28	0.18
71	0.16	6.88	13.60	66.16	0.00	0.00	11.69	98.49	0.34	0.17
72	0.20	6.63	13.92	66.48	0.09	0.00	10.85	98.19	0.32	0.16
73	0.21	6.59	14.14	65.72	0.15	0.00	11.02	97.84	0.32	0.16
74	0.19	6.30	14.31	66.17	0.09	0.00	10.84	97.89	0.30	0.16
75	0.22	6.38	14.05	66.23	0.02	0.08	10.97	97.95	0.31	0.16
76	0.23	6.45	14.31	66.15	0.03	0.00	11.03	98.20	0.31	0.16
78	0.22	6.41	14.26	65.69	0.13	0.00	10.77	97.48	0.31	0.16
79	0.21	6.35	14.08	66.30	0.07	0.00	10.80	97.82	0.31	0.16
80	0.24	6.25	14.06	66.14	0.05	0.03	10.88	97.66	0.31	0.16
81	0.23	6.24	14.00	66.20	0.07	0.05	10.84	97.62	0.31	0.16
82	0.25	6.25	14.45	65.52	0.08	0.11	10.87	97.53	0.30	0.16
83	0.26	6.67	13.85	66.43	0.11	0.00	10.84	98.19	0.32	0.16
84	0.23	6.40	14.24	65.96	0.00	0.00	10.66	97.49	0.31	0.16
85	0.25	6.36	14.08	66.07	0.09	0.00	11.12	97.97	0.31	0.16
86	0.22	6.49	14.24	66.15	0.06	0.07	10.95	98.20	0.31	0.16
87	0.22	6.54	13.98	66.15	0.18	0.00	10.83	97.92	0.32	0.16
88	0.24	6.59	14.35	65.78	0.12	0.00	10.93	98.01	0.31	0.16
89	0.22	6.63	14.64	66.31	0.02	0.04	10.73	98.59	0.31	0.16

Buranga, Rwanda, Ferrocolumbite

90	0.75	9.63	10.73	64.29	0.30	0.15	11.82	97.71	0.47	0.18
91	0.70	9.90	10.43	64.82	0.25	0.07	11.84	98.17	0.49	0.18
92	0.70	9.81	10.48	63.07	0.24	0.18	11.34	95.87	0.48	0.17
93	0.73	9.90	10.47	64.44	0.34	0.30	11.57	97.79	0.49	0.17
94	0.68	9.92	10.68	64.97	0.28	0.22	11.85	98.73	0.48	0.18
95	0.74	10.00	10.60	64.34	0.35	0.17	11.19	97.47	0.48	0.17
96	0.71	9.78	10.49	64.36	0.30	0.21	11.18	97.12	0.48	0.17

97	0.71	9.61	10.72	64.31	0.56	0.25	12.05	98.29	0.47	0.18
98	0.73	9.88	10.69	64.44	0.43	0.27	11.31	97.84	0.48	0.17
99	0.72	10.07	10.57	64.34	0.29	0.31	11.46	97.79	0.49	0.17
100	0.73	9.99	10.45	64.53	0.23	0.10	11.90	98.03	0.49	0.18
101	0.77	9.78	11.06	64.72	0.44	0.31	11.93	99.12	0.47	0.18
102	0.74	9.99	10.83	64.31	0.44	0.09	11.60	98.05	0.48	0.17
103	0.74	9.80	10.61	64.11	0.46	0.22	11.76	97.74	0.48	0.18
104	0.75	9.82	10.54	64.22	0.36	0.21	11.60	97.53	0.48	0.17
105	0.65	9.53	11.04	66.08	0.03	0.15	10.91	98.41	0.46	0.16
106	0.75	9.63	10.73	64.29	0.30	0.15	11.82	97.71	0.47	0.18
107	0.70	9.90	10.43	64.82	0.25	0.07	11.84	98.17	0.49	0.18
108	0.70	9.81	10.48	63.07	0.24	0.18	11.34	95.87	0.48	0.17
109	0.73	9.90	10.47	64.44	0.34	0.30	11.57	97.79	0.49	0.17

NP2, Fujian, China, Ferrotantalite

110	0.37	6.54	10.47	42.74	0.51	0.00	38.06	98.69	0.38	0.51
111	0.33	6.41	10.99	41.43	0.40	0.00	36.36	95.93	0.37	0.51
112	0.30	6.34	11.25	39.74	0.07	0.00	38.65	96.36	0.36	0.53
113	0.24	6.38	11.28	41.22	0.10	0.00	38.71	97.94	0.36	0.52
114	0.44	6.88	10.50	41.03	0.34	0.00	38.18	97.37	0.40	0.52
115	0.35	6.84	10.65	41.48	0.34	0.00	36.29	95.95	0.39	0.51
116	0.37	6.55	11.01	41.43	0.46	0.00	36.30	96.12	0.37	0.51
117	0.30	6.03	11.36	41.24	0.08	0.00	38.21	97.21	0.35	0.52
118	0.38	6.75	10.39	40.80	0.51	0.00	37.83	96.66	0.39	0.52
119	0.35	6.94	10.69	41.91	0.55	0.00	37.44	97.88	0.39	0.51
120	0.40	8.86	9.42	39.49	0.32	0.00	37.45	95.95	0.48	0.53
121	0.38	6.85	10.50	41.43	0.44	0.00	36.89	96.49	0.39	0.51
122	0.38	6.04	11.02	41.01	0.41	0.00	36.52	95.39	0.35	0.51
123	0.36	6.75	10.38	41.85	0.53	0.00	36.28	96.16	0.39	0.50
124	0.35	7.04	10.61	41.34	0.59	0.00	37.20	97.13	0.40	0.51
125	0.32	6.22	11.33	41.94	0.33	0.00	36.76	96.89	0.35	0.51
126	0.37	7.04	10.48	41.65	0.47	0.00	36.33	96.34	0.40	0.51
127	0.40	6.35	10.53	40.31	0.50	0.00	37.89	95.98	0.38	0.52
128	0.41	6.18	11.21	41.32	0.19	0.00	37.02	96.32	0.35	0.51
129	0.38	6.87	10.72	42.74	0.48	0.00	35.83	97.04	0.39	0.50
130	0.40	6.60	10.41	41.22	0.46	0.00	37.04	96.14	0.39	0.51
131	0.46	6.62	10.41	41.30	0.34	0.00	37.13	96.26	0.39	0.51
132	0.40	6.64	10.70	42.07	0.45	0.00	36.40	96.67	0.38	0.50
133	0.38	7.06	10.68	40.12	0.50	0.00	36.34	95.07	0.40	0.51

NT-2, Altai, Ferrotantalite

134	0.21	6.90	11.46	45.30	0.28	0.00	36.57	100.77	0.38	0.49
135	0.23	7.03	11.69	44.54	0.19	0.00	37.67	101.35	0.37	0.50
136	0.22	7.00	11.22	44.32	0.20	0.00	37.43	100.39	0.38	0.50
137	0.22	6.85	11.72	45.28	0.22	0.00	36.90	101.20	0.37	0.49
138	0.25	6.99	11.85	43.93	0.24	0.00	36.41	99.69	0.37	0.49
139	0.19	7.33	11.42	43.90	0.27	0.00	37.13	100.25	0.39	0.50
140	0.25	7.26	11.10	44.84	0.33	0.00	37.04	100.85	0.39	0.49
141	0.22	6.88	11.57	43.63	0.28	0.00	37.51	100.09	0.37	0.50
142	0.21	7.19	11.53	43.40	0.18	0.00	37.66	100.16	0.38	0.50
143	0.19	7.53	11.69	44.28	0.26	0.00	36.84	100.81	0.39	0.49
144	0.24	7.03	11.53	43.02	0.15	0.00	37.44	99.51	0.38	0.50
145	0.28	7.02	12.06	43.26	0.09	0.00	37.44	100.18	0.37	0.50
146	0.27	6.91	11.00	43.29	0.17	0.00	36.88	98.52	0.38	0.50
147	0.18	6.79	11.41	45.48	0.27	0.00	37.68	101.80	0.37	0.49

148	0.22	7.11	11.18	43.79	0.32	0.00	36.70	99.72	0.39	0.50
149	0.27	6.76	11.53	45.04	0.21	0.00	37.23	101.05	0.37	0.49
150	0.18	6.93	11.26	44.82	0.25	0.00	37.53	100.97	0.38	0.50
151	0.30	6.89	11.59	44.79	0.29	0.00	34.93	98.80	0.37	0.48
152	0.27	6.77	11.44	43.78	0.26	0.00	36.18	98.76	0.37	0.49
153	0.26	6.86	11.53	43.55	0.25	0.00	35.92	98.58	0.37	0.49
154	0.17	6.82	11.44	44.11	0.22	0.00	37.13	99.95	0.37	0.50
155	0.22	7.05	11.28	43.85	0.25	0.00	36.82	99.51	0.38	0.50
156	0.25	7.16	11.71	44.11	0.28	0.00	37.46	100.98	0.38	0.50
157	0.23	7.01	11.24	43.80	0.23	0.00	37.49	100.11	0.38	0.50

A-1, Altai, Manganocolumbite

158	0.42	14.09	2.99	27.02	0.00	0.00	54.85	99.48	0.82	0.70
159	0.38	14.34	2.87	27.75	0.01	0.00	54.64	100.01	0.83	0.70
160	0.44	14.26	2.92	27.41	0.02	0.00	55.17	100.21	0.83	0.70
161	0.39	14.33	2.87	26.61	0.02	0.00	53.89	98.19	0.83	0.70
162	0.42	14.24	3.08	27.37	0.00	0.00	55.81	101.00	0.82	0.70
163	0.41	13.70	2.80	26.97	0.00	0.00	53.30	97.20	0.83	0.70
164	0.44	13.71	3.17	26.85	0.02	0.00	55.35	99.66	0.81	0.71
165	0.43	13.82	2.97	27.75	0.00	0.00	54.08	99.05	0.82	0.70
166	0.46	14.07	2.92	28.17	0.00	0.00	53.97	99.60	0.83	0.69
167	0.11	12.47	4.41	25.79	0.00	0.00	57.53	100.33	0.74	0.72
168	0.54	13.83	2.88	27.01	0.00	0.00	54.27	98.57	0.83	0.70
169	0.63	13.74	2.85	26.36	0.00	0.00	56.44	100.18	0.83	0.71
170	0.40	13.41	2.64	26.14	0.05	0.00	53.50	96.40	0.84	0.71
171	0.23	14.19	2.80	27.02	0.01	0.00	56.60	100.86	0.83	0.71
172	0.26	14.30	2.89	28.08	0.00	0.00	53.53	99.05	0.83	0.69
173	0.26	13.64	2.86	29.03	0.04	0.00	54.43	100.27	0.83	0.69
174	0.29	13.97	2.73	26.43	0.02	0.00	56.56	100.05	0.84	0.71
175	0.49	13.95	3.11	28.13	0.01	0.00	52.67	98.40	0.82	0.69
176	0.45	13.32	2.81	26.90	0.02	0.00	53.30	96.99	0.83	0.70
177	0.40	13.95	2.78	28.99	0.00	0.00	54.29	100.42	0.83	0.69
178	0.45	13.64	3.03	28.75	0.01	0.00	54.06	100.07	0.82	0.69
179	0.42	13.83	3.04	27.25	0.01	0.00	54.82	99.55	0.82	0.70
180	0.52	13.96	3.10	27.85	0.03	0.00	54.73	100.27	0.82	0.70
181	0.42	13.66	3.02	27.36	0.01	0.00	54.57	99.07	0.82	0.70
182	0.48	13.54	3.14	26.72	0.00	0.00	54.72	98.70	0.81	0.71

ISSIA2, Côte d'Ivoire, Manganotantalite

182	0.26	9.42	6.66	18.38	0.06	0.07	62.33	97.20	0.59	0.80
183	0.26	9.38	6.39	17.83	0.12	0.24	62.50	96.73	0.59	0.80
184	0.16	9.56	6.56	18.94	0.07	0.09	61.76	97.15	0.59	0.79
185	0.29	9.42	6.42	18.58	0.02	0.35	62.83	97.92	0.59	0.80
186	0.25	9.40	6.47	18.45	0.01	0.45	61.71	96.75	0.59	0.80
187	0.24	9.72	6.46	18.43	0.01	0.12	63.34	98.39	0.60	0.80
188	0.25	9.50	6.43	18.59	0.04	0.15	62.23	97.21	0.60	0.80
189	0.25	8.69	6.00	16.52	0.00	0.25	58.37	90.08	0.59	0.81
190	0.19	10.18	6.15	18.31	0.01	0.00	62.75	97.59	0.62	0.80
191	0.26	9.51	6.41	18.33	0.28	0.09	61.46	96.36	0.60	0.80
192	0.28	9.72	6.46	18.39	0.00	0.23	63.25	98.37	0.60	0.80
193	0.27	9.32	6.40	18.49	0.14	0.40	63.12	98.15	0.59	0.80
194	0.27	9.52	6.54	18.36	0.09	0.52	62.21	97.57	0.59	0.80
195	0.26	8.97	6.84	17.96	0.11	0.33	63.58	98.15	0.57	0.81
196	0.22	9.60	6.20	19.46	0.02	0.05	61.91	97.44	0.61	0.79
197	0.29	9.45	6.31	18.02	0.18	0.33	63.26	97.89	0.60	0.80

198	0.28	9.80	6.36	18.32	0.22	0.31	62.84	98.17	0.61	0.80
199	0.26	9.54	6.37	17.43	0.05	0.25	62.89	96.86	0.60	0.81
200	0.27	9.58	6.35	18.00	0.16	0.49	63.41	98.35	0.60	0.80
201	0.29	8.40	5.88	17.55	0.13	0.18	62.31	94.74	0.59	0.81
202	0.28	9.36	6.69	18.11	0.00	0.18	63.72	98.33	0.58	0.80
203	0.28	9.36	6.43	17.55	0.12	0.44	62.68	96.87	0.59	0.81
204	0.26	9.25	6.32	17.23	0.18	0.29	64.13	97.69	0.59	0.81
205	0.27	9.44	6.32	17.48	0.10	0.09	63.38	97.11	0.60	0.81
206	0.26	9.71	6.29	17.18	0.11	0.46	62.68	96.76	0.61	0.81
207	0.29	8.74	6.16	17.16	0.00	0.27	63.98	96.66	0.59	0.81
208	0.24	9.61	6.54	17.24	0.19	0.46	63.44	97.72	0.59	0.81
209	0.26	9.36	6.41	17.35	0.14	0.31	62.96	96.82	0.59	0.81
210	0.23	8.39	5.57	17.00	0.02	0.18	64.12	95.50	0.60	0.82
211	0.27	9.53	6.32	17.25	0.08	0.29	62.84	96.69	0.60	0.81

CC1716, France, Ferrocolumbite

212	1.93	6.10	15.10	71.73	0.02	0.00	4.26	99.51	0.29	0.07
213	1.69	6.08	14.30	72.42	0.00	0.00	4.02	98.55	0.30	0.06
214	1.79	6.78	13.57	69.39	0.00	0.00	6.38	98.07	0.33	0.10
215	2.19	6.09	14.98	70.13	0.00	0.00	4.12	97.72	0.29	0.06
216	1.89	6.56	14.64	70.04	0.10	0.00	7.12	100.48	0.31	0.11
217	1.89	6.05	14.26	71.75	0.05	0.00	4.09	98.83	0.30	0.06
218	1.66	6.12	14.72	72.93	0.06	0.00	3.99	99.88	0.29	0.06
219	1.77	6.11	14.74	71.22	0.08	0.00	4.12	98.81	0.29	0.06
220	1.57	6.67	14.39	70.57	0.00	0.00	6.45	99.75	0.32	0.10
221	1.49	6.24	14.74	72.55	0.06	0.00	4.27	99.55	0.30	0.06
222	1.94	6.35	14.81	69.76	0.00	0.00	5.90	98.93	0.30	0.09
223	1.76	6.11	15.41	70.53	0.01	0.00	6.50	100.44	0.28	0.10
224	1.70	6.16	14.47	70.66	0.04	0.00	5.82	98.94	0.30	0.09
225	1.85	5.55	15.34	68.46	0.04	0.00	6.72	97.99	0.26	0.10
226	1.56	6.28	14.82	68.22	0.00	0.00	4.71	95.90	0.30	0.07
227	1.74	5.61	15.30	71.95	0.05	0.00	4.02	98.74	0.27	0.06
228	1.61	6.37	15.04	69.58	0.05	0.00	5.58	98.32	0.30	0.09
229	1.71	6.07	15.24	70.04	0.10	0.00	6.56	99.72	0.28	0.10
230	1.61	6.22	14.70	70.42	0.02	0.00	6.00	99.00	0.30	0.09
231	1.61	5.94	14.82	70.07	0.02	0.00	5.56	98.06	0.29	0.09
232	1.66	6.35	15.02	69.97	0.01	0.00	6.15	99.57	0.30	0.09

Appendix B – SIMS analytical results for columbo-tantalites. Data in italic were not used for age calculation.

Measured ratios								Calibrated ratios					U-Pb ages (Ma)			
²⁰⁷ Pb/ ²⁰⁶ Pb	err.	²⁰⁴ Pb/ ²⁰⁶ Pb	err.	²⁰⁶ Pb/U	err.	UO/U	err.	²⁰⁶ Pb/ ²³⁸ U	err.	²⁰⁷ Pb/ ²³⁵ U	err.	Cor.	²⁰⁶ Pb/ ²³⁸ U	err.	²⁰⁷ Pb/ ²³⁵ U	err.
CT1 - Ivory Coast - Ferrotapiolite standard																
0.12651	0.00031	1.4E-05	1.9E-06	21.9834	0.1225	19.214	0.078	0.3748	0.0062	6.546	0.007	0.928	-	-	-	-
0.12664	0.00034	3.1E-05	3.6E-06	21.9490	0.1224	19.007	0.085	0.3796	0.0063	6.625	0.007	0.915	-	-	-	-
0.12771	0.00037	1.7E-05	1.7E-06	22.0246	0.1577	18.990	0.096	0.3815	0.0077	6.724	0.008	0.935	-	-	-	-
0.12631	0.00016	2.2E-05	4.3E-06	21.5041	0.0741	18.830	0.093	0.3767	0.0045	6.565	0.005	0.951	-	-	-	-
0.12634	0.00023	3.4E-05	1.1E-06	21.7042	0.0549	18.992	0.109	0.3758	0.0037	6.540	0.004	0.897	-	-	-	-
0.12657	0.00027	1.4E-05	2.5E-06	22.0039	0.1235	19.151	0.117	0.3768	0.0063	6.584	0.007	0.944	-	-	-	-
0.12628	0.00015	2.0E-05	3.4E-06	21.7620	0.0881	19.221	0.046	0.3708	0.0049	6.461	0.005	0.967	-	-	-	-
0.12629	0.00025	2.0E-05	4.3E-06	22.2501	0.1505	19.199	0.093	0.3797	0.0074	6.616	0.008	0.960	-	-	-	-
0.12615	0.00020	2.1E-05	2.7E-06	20.7816	0.0825	18.460	0.089	0.3740	0.0049	6.510	0.005	0.946	-	-	-	-
0.12567	0.00040	2.0E-05	2.7E-06	22.1244	0.1138	19.350	0.071	0.3736	0.0059	6.478	0.007	0.875	-	-	-	-
0.12569	0.00046	1.9E-05	3.0E-06	21.3502	0.0953	18.871	0.088	0.3729	0.0053	6.468	0.006	0.815	-	-	-	-
0.12626	0.00034	1.4E-05	2.5E-06	21.6659	0.0925	19.159	0.048	0.3708	0.0051	6.463	0.006	0.883	-	-	-	-
0.12555	0.00012	2.2E-05	4.1E-06	22.0014	0.1099	19.116	0.083	0.3776	0.0058	6.540	0.006	0.982	-	-	-	-
0.12476	0.00034	1.3E-05	3.1E-06	21.8988	0.0830	19.077	0.076	0.3770	0.0047	6.494	0.005	0.862	-	-	-	-
0.12581	0.00017	4.4E-05	5.9E-06	21.1872	0.0406	18.790	0.080	0.3721	0.0034	6.443	0.004	0.899	-	-	-	-
0.12488	0.00051	3.9E-05	5.4E-06	20.9640	0.0976	18.269	0.169	0.3826	0.0055	6.579	0.007	0.793	-	-	-	-
0.12780	0.00042	1.2E-04	1.1E-05	18.7472	0.4023	17.019	0.264	0.3771	0.0219	6.579	0.022	0.986	-	-	-	-
0.12811	0.00044	6.1E-05	1.2E-05	21.5453	0.1103	18.246	0.294	0.3938	0.0060	6.931	0.007	0.834	-	-	-	-
0.12698	0.00028	2.5E-05	2.0E-06	20.2969	0.2332	18.114	0.151	0.3749	0.0119	6.565	0.012	0.982	-	-	-	-
0.12680	0.00047	2.0E-05	3.5E-06	21.4025	0.0899	18.991	0.066	0.3706	0.0051	6.485	0.006	0.800	-	-	-	-

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0.12743	0.00023	6.2E-05	7.7E-06	21.4004	0.1768	18.787	0.174	0.3758	0.0088	6.579	0.009	0.971	-	-	-	-
0.12722	0.00075	1.5E-04	1.4E-05	21.1059	0.1549	18.077	0.199	0.3901	0.0081	6.755	0.010	0.782	-	-	-	-
0.12823	0.00020	1.0E-04	5.5E-06	20.6060	0.1380	18.293	0.149	0.3750	0.0073	6.579	0.008	0.971	-	-	-	-
0.12698	0.00027	1.4E-05	3.0E-06	21.9882	0.0805	19.202	0.079	0.3752	0.0046	6.578	0.005	0.901	-	-	-	-
0.12631	0.00021	1.7E-05	3.8E-06	22.4916	0.0983	19.297	0.076	0.3812	0.0052	6.646	0.006	0.945	-	-	-	-
0.12618	0.00029	1.4E-05	2.5E-06	22.4612	0.0913	19.511	0.101	0.3751	0.0049	6.534	0.005	0.900	-	-	-	-
0.12659	0.00024	2.6E-05	4.3E-06	21.5110	0.1559	18.978	0.095	0.3728	0.0078	6.509	0.008	0.969	-	-	-	-
0.12669	0.00032	2.8E-05	4.5E-06	20.8929	0.0864	18.505	0.087	0.3747	0.0050	6.545	0.006	0.882	-	-	-	-
0.12446	0.00029	1.7E-05	2.2E-06	20.2824	0.1197	18.371	0.126	0.3675	0.0065	6.313	0.007	0.940	-	-	-	-
0.12396	0.00066	1.6E-05	4.4E-06	20.2718	0.2073	18.783	0.093	0.3564	0.0107	6.098	0.012	0.891	-	-	-	-
0.12536	0.00023	1.9E-05	4.8E-06	22.0456	0.1156	19.178	0.078	0.3768	0.0060	6.517	0.006	0.948	-	-	-	-
0.12592	0.00031	1.8E-05	4.0E-06	21.4301	0.1041	19.119	0.104	0.3678	0.0056	6.392	0.006	0.910	-	-	-	-
0.12817	0.00041	1.1E-04	8.3E-06	22.8215	0.1824	19.201	0.036	0.3888	0.0086	6.812	0.009	0.926	-	-	-	-
0.12687	0.00054	5.1E-05	3.5E-06	22.1248	0.1491	19.305	0.091	0.3746	0.0073	6.536	0.009	0.859	-	-	-	-
0.12691	0.00025	7.8E-05	5.4E-06	22.5398	0.0945	19.123	0.124	0.3863	0.0051	6.724	0.006	0.920	-	-	-	-
0.12748	0.00046	7.5E-05	5.2E-06	17.0253	0.4425	16.133	0.253	0.3699	0.0263	6.469	0.027	0.990	-	-	-	-
0.12681	0.00021	1.2E-05	2.2E-06	22.0028	0.0420	19.115	0.048	0.3777	0.0034	6.615	0.004	0.890	-	-	-	-
0.12557	0.00031	2.0E-05	2.9E-06	22.3363	0.0707	19.355	0.074	0.3770	0.0042	6.533	0.005	0.860	-	-	-	-
0.12634	0.00018	1.7E-05	3.5E-06	21.7509	0.0616	18.951	0.059	0.3778	0.0040	6.588	0.004	0.932	-	-	-	-

CT3 - Ivory Coast

0.12612	0.00021	7.0E-06	2.2E-06	22.1016	0.1143	19.178	0.048	0.3778	0.0059	6.582	0.006	0.960	2066	10	2057	5
0.12670	0.00023	1.2E-05	2.4E-06	22.2478	0.0731	19.239	0.044	0.3786	0.0043	6.624	0.005	0.921	2070	8	2063	4
0.12730	0.00029	1.3E-05	1.2E-06	22.2771	0.1977	19.383	0.101	0.3754	0.0093	6.596	0.010	0.971	2055	16	2059	8
0.12584	0.00023	3.6E-05	3.9E-06	17.8493	0.4826	17.135	0.305	0.3561	0.0272	6.172	0.027	0.998	1964	46	2001	24
0.12568	0.00019	7.4E-06	9.0E-07	21.8121	0.0379	19.081	0.044	0.3754	0.0033	6.517	0.004	0.906	2055	6	2048	3

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0.12624	0.00013	6.3E-06	1.3E-06	21.9119	0.0964	18.983	0.080	0.3798	0.0052	6.624	0.005	0.981	2075	9	2062	5
0.12598	0.00021	9.4E-06	2.3E-06	21.6332	0.0370	18.959	0.067	0.3756	0.0033	6.535	0.004	0.889	2055	6	2051	3
0.12625	0.00011	9.9E-06	1.6E-06	21.8099	0.1157	19.115	0.070	0.3745	0.0060	6.529	0.006	0.989	2050	11	2050	5
0.12583	0.00018	1.7E-05	2.8E-06	21.8584	0.0720	19.067	0.041	0.3765	0.0043	6.538	0.005	0.949	2060	8	2051	4
0.12637	0.00020	6.4E-06	1.5E-06	15.2913	0.3649	15.045	0.199	0.3685	0.0240	6.433	0.024	0.998	2022	42	2037	21
0.12740	0.00023	1.1E-05	2.1E-06	22.4424	0.0807	19.181	0.053	0.3835	0.0046	6.747	0.005	0.929	2093	8	2079	4
0.12678	0.00013	6.9E-06	1.7E-06	22.6794	0.1098	19.466	0.058	0.3800	0.0056	6.655	0.006	0.984	2076	10	2067	5
0.12702	0.00021	7.7E-06	2.1E-06	22.6683	0.0701	19.375	0.034	0.3822	0.0042	6.706	0.004	0.927	2086	7	2073	4
0.12730	0.00012	1.2E-05	1.8E-06	22.4396	0.1349	19.315	0.082	0.3799	0.0066	6.677	0.007	0.990	2076	12	2070	6
0.12666	0.00049	5.1E-06	7.0E-07	21.6311	0.2100	18.735	0.157	0.3817	0.0101	6.680	0.011	0.935	2084	18	2070	10
0.12368	0.00035	2.0E-05	2.8E-06	19.7558	0.1547	19.061	0.078	0.3404	0.0083	5.809	0.009	0.948	1889	14	1948	8
0.12751	0.00036	1.3E-05	1.7E-06	22.8978	0.1628	19.271	0.077	0.3888	0.0076	6.846	0.008	0.938	2117	14	2092	7
0.12699	0.00027	4.6E-06	1.7E-06	22.2191	0.0905	19.098	0.062	0.3820	0.0049	6.703	0.005	0.919	2085	9	2073	5
0.12674	0.00026	1.2E-05	2.1E-06	23.1580	0.1960	19.602	0.103	0.3844	0.0089	6.727	0.009	0.974	2097	16	2076	8
0.12625	0.00018	1.0E-05	2.0E-06	23.9915	0.1760	20.142	0.073	0.3841	0.0079	6.697	0.008	0.984	2095	14	2072	7
0.12698	0.00018	8.9E-06	1.5E-06	21.7602	0.0861	18.992	0.035	0.3769	0.0048	6.610	0.005	0.962	2062	9	2061	4
0.12669	0.00044	8.4E-06	2.1E-06	20.1090	0.1192	18.090	0.060	0.3722	0.0066	6.513	0.007	0.882	2040	11	2048	7
0.12712	0.00048	1.0E-05	2.1E-06	21.7446	0.1379	19.102	0.046	0.3737	0.0069	6.560	0.008	0.879	2047	12	2054	7
0.12663	0.00025	7.5E-06	1.7E-06	21.6901	0.0771	18.930	0.064	0.3773	0.0045	6.601	0.005	0.916	2064	8	2059	4
0.12679	0.00017	1.1E-05	1.9E-06	21.9707	0.0910	19.087	0.135	0.3779	0.0050	6.618	0.005	0.967	2067	9	2062	5
0.12623	0.00032	1.9E-05	2.1E-06	21.2331	0.1876	18.816	0.089	0.3723	0.0093	6.485	0.010	0.964	2040	16	2044	8
0.12674	0.00033	2.1E-05	3.2E-06	20.5110	0.1487	18.405	0.144	0.3707	0.0078	6.481	0.008	0.948	2033	14	2043	7
0.12683	0.00011	5.9E-06	1.2E-06	21.5918	0.0497	18.994	0.060	0.3739	0.0036	6.553	0.004	0.971	2048	6	2053	3
0.12697	0.00039	1.5E-05	2.2E-06	21.7179	0.0986	19.098	0.075	0.3733	0.0053	6.543	0.006	0.867	2045	9	2052	5
0.12623	0.00056	7.9E-06	2.1E-06	22.1547	0.1174	19.201	0.087	0.3781	0.0060	6.593	0.007	0.804	2067	11	2058	7
0.12762	0.00033	8.9E-06	1.7E-06	22.2458	0.1575	19.451	0.088	0.3731	0.0076	6.577	0.008	0.946	2044	13	2056	7

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0.12681	0.00047	1.6E-05	3.9E-06	22.0946	0.1225	19.272	0.162	0.3751	0.0062	6.566	0.007	0.858	2054	11	2055	6
0.12631	0.00024	1.3E-05	3.8E-06	21.4710	0.1042	18.860	0.065	0.3754	0.0056	6.546	0.006	0.947	2055	10	2052	5
0.12702	0.00033	9.9E-06	1.4E-06	22.2885	0.1201	19.252	0.068	0.3790	0.0061	6.649	0.007	0.920	2072	11	2066	6
0.12655	0.00040	1.1E-05	1.4E-06	22.5952	0.0970	19.265	0.088	0.3839	0.0051	6.709	0.006	0.854	2094	9	2074	5

CT4 - Ivory Coast

0.12610	0.00014	4.0E-05	6.7E-06	22.8041	0.1007	19.428	0.028	0.3829	0.0052	6.648	0.005	0.975	2090	9	2066	5
0.12571	0.00029	4.4E-05	6.0E-06	21.8633	0.0932	19.288	0.080	0.3707	0.0051	6.413	0.006	0.910	2032	9	2034	5
0.12633	0.00033	2.5E-05	1.8E-06	22.8141	0.0608	19.365	0.037	0.3848	0.0039	6.704	0.005	0.829	2099	7	2073	4
0.12564	0.00016	1.9E-05	3.6E-06	22.5940	0.0952	19.323	0.053	0.3822	0.0051	6.627	0.005	0.970	2087	9	2063	5
0.12605	0.00021	2.1E-05	2.2E-06	22.9053	0.0961	19.495	0.152	0.3829	0.0050	6.659	0.005	0.948	2090	9	2067	5
0.12596	0.00026	4.4E-05	6.6E-06	19.7019	0.3867	17.312	0.232	0.3874	0.0199	6.717	0.020	0.995	2111	36	2075	17
0.12626	0.00044	2.8E-05	1.6E-06	21.7188	0.1452	18.810	0.067	0.3810	0.0073	6.632	0.008	0.901	2081	13	2064	7
0.12540	0.00020	2.8E-05	2.8E-06	22.3663	0.1280	19.282	0.084	0.3794	0.0064	6.559	0.007	0.970	2074	11	2054	6
0.12587	0.00026	2.7E-05	4.9E-06	22.2000	0.1210	19.047	0.061	0.3829	0.0061	6.645	0.006	0.948	2090	11	2065	6
0.12613	0.00017	1.9E-05	1.9E-06	22.7337	0.0985	19.390	0.064	0.3828	0.0052	6.663	0.005	0.967	2090	9	2068	5

Rongi - Rwanda - Ferrocolumbite standard

0.07040	0.00018	3.3E-05	4.6E-06	25.3000	0.1400	33.270	0.180	0.1521	0.0069	1.467	0.007	0.930	-	-	-	-
0.07010	0.00020	2.4E-05	5.1E-06	24.3800	0.0600	32.980	0.110	0.1483	0.0049	1.429	0.006	0.840	-	-	-	-
0.07060	0.00025	2.1E-05	3.3E-06	23.6700	0.1000	32.420	0.080	0.1473	0.0062	1.430	0.007	0.850	-	-	-	-
0.07050	0.00010	5.7E-05	4.9E-06	25.6700	0.2100	33.510	0.270	0.1528	0.0092	1.470	0.009	0.980	-	-	-	-
0.07310	0.00021	2.8E-04	1.8E-05	24.5600	0.1400	33.710	0.220	0.1444	0.0075	1.379	0.009	0.810	-	-	-	-
0.07080	0.00023	3.6E-05	6.1E-06	24.6200	0.1700	33.580	0.150	0.1462	0.0081	1.418	0.009	0.910	-	-	-	-
0.07090	0.00020	6.0E-05	0.0E+00	26.4710	0.1927	34.271	0.136	0.1529	0.0085	1.478	0.009	0.930	-	-	-	-
0.07080	0.00030	4.0E-05	0.0E+00	23.6890	0.1012	33.511	0.103	0.1410	0.0061	1.368	0.008	0.780	-	-	-	-

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0.07080	0.00020	3.0E-05	0.0E+00	26.0582	0.2249	34.950	0.117	0.1468	0.0097	1.427	0.010	0.930	-	-	-	-
0.07330	0.00010	1.5E-04	0.0E+00	25.4491	0.3434	33.270	0.120	0.1527	0.0144	1.500	0.015	0.980	-	-	-	-
0.07160	0.00030	4.0E-05	0.0E+00	26.9221	0.1100	35.414	0.151	0.1490	0.0060	1.460	0.008	0.770	-	-	-	-
0.07130	0.00030	3.0E-05	0.0E+00	26.1635	0.1452	34.394	0.188	0.1505	0.0071	1.473	0.008	0.870	-	-	-	-
0.07390	0.00030	3.0E-04	0.0E+00	25.7662	0.1351	34.615	0.283	0.1463	0.0070	1.407	0.009	0.790	-	-	-	-
0.08130	0.00020	7.8E-04	0.0E+00	26.2407	0.1035	33.759	0.163	0.1528	0.0061	1.481	0.009	0.680	-	-	-	-
0.07190	0.00030	1.2E-04	0.0E+00	25.8780	0.0854	34.407	0.250	0.1486	0.0056	1.440	0.008	0.690	-	-	-	-
0.07080	0.00010	2.0E-05	0.0E+00	26.6344	0.1456	35.013	0.181	0.1497	0.0070	1.457	0.007	0.950	-	-	-	-
0.07030	0.00020	4.0E-05	0.0E+00	26.9062	0.1177	34.920	0.207	0.1517	0.0062	1.461	0.007	0.900	-	-	-	-
0.07240	0.00020	7.0E-05	0.0E+00	26.1654	0.1969	33.177	0.140	0.1578	0.0088	1.556	0.009	0.940	-	-	-	-
0.07090	0.00020	6.0E-05	0.0E+00	25.9055	0.0809	34.172	0.100	0.1502	0.0053	1.454	0.006	0.890	-	-	-	-
0.07030	0.00030	3.0E-05	0.0E+00	25.6752	0.0989	33.977	0.131	0.1501	0.0058	1.449	0.007	0.770	-	-	-	-
0.07030	0.00020	5.0E-05	0.0E+00	23.1659	0.1552	31.124	0.280	0.1523	0.0081	1.462	0.009	0.920	-	-	-	-
0.07000	0.00030	2.0E-05	0.0E+00	25.8623	0.2084	34.246	0.248	0.1496	0.0092	1.440	0.010	0.900	-	-	-	-
0.07060	0.00040	1.0E-05	0.0E+00	25.4072	0.1934	33.899	0.113	0.1490	0.0088	1.448	0.011	0.820	-	-	-	-
0.07020	0.00030	2.0E-05	0.0E+00	24.9531	0.1704	33.888	0.214	0.1464	0.0081	1.413	0.009	0.880	-	-	-	-
0.07470	0.00050	3.4E-04	0.0E+00	26.8388	0.2016	33.495	0.187	0.1590	0.0089	1.533	0.012	0.740	-	-	-	-
0.07050	0.00025	3.4E-05	2.6E-06	24.6629	0.2395	33.612	0.203	0.1462	0.0107	1.414	0.011	0.950	-	-	-	-
0.07010	0.00019	1.6E-05	5.2E-06	25.4624	0.1111	34.113	0.175	0.1481	0.0062	1.429	0.007	0.900	-	-	-	-
0.07030	0.00020	1.7E-05	3.6E-06	25.9599	0.2170	34.233	0.185	0.1503	0.0094	1.454	0.010	0.950	-	-	-	-
0.07010	0.00024	2.5E-05	2.3E-06	21.9248	0.3118	30.261	0.423	0.1499	0.0149	1.443	0.015	0.970	-	-	-	-
0.07100	0.00033	5.9E-05	7.9E-06	19.2577	0.0623	27.984	0.127	0.1468	0.0054	1.422	0.008	0.720	-	-	-	-
0.07050	0.00032	4.3E-05	6.4E-06	26.0807	0.0492	34.039	0.127	0.1521	0.0047	1.467	0.007	0.700	-	-	-	-
Buranga - Rwanda																
0.07519	0.00059	4.3E-04	3.2E-05	26.0861	0.2166	34.303	0.404	0.1496	0.0095	1.429	0.013	0.730	899	8	901	8

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0.07749	0.00081	5.4E-04	6.1E-05	23.6782	0.3108	31.878	0.341	0.1494	0.0142	1.443	0.019	0.750	898	12	907	11
<i>0.07673</i>	<i>0.00051</i>	<i>6.1E-04</i>	<i>3.9E-05</i>	<i>26.3340</i>	<i>0.4728</i>	<i>33.885</i>	<i>0.467</i>	<i>0.1530</i>	<i>0.0188</i>	<i>1.439</i>	<i>0.020</i>	<i>0.920</i>	<i>918</i>	<i>16</i>	<i>905</i>	<i>12</i>
0.07759	0.00077	7.2E-04	8.4E-05	25.6993	0.3442	33.466	0.327	0.1515	0.0146	1.410	0.020	0.750	909	12	893	12
0.07456	0.00055	4.8E-04	5.7E-05	27.1233	0.2869	34.860	0.552	0.1521	0.0117	1.426	0.015	0.790	913	10	900	9
0.07430	0.00039	3.2E-04	3.8E-05	25.3330	0.1121	33.660	0.291	0.1492	0.0063	1.442	0.009	0.720	897	5	906	5
0.07390	0.00040	3.7E-04	5.9E-05	24.8648	0.1834	33.325	0.376	0.1483	0.0088	1.408	0.011	0.780	891	7	892	7
0.07260	0.00050	3.3E-04	3.8E-05	24.9008	0.4201	32.899	0.237	0.1512	0.0177	1.420	0.019	0.910	908	15	897	11
0.07400	0.00075	2.1E-04	3.2E-05	24.6211	0.3511	32.801	0.339	0.1504	0.0151	1.479	0.019	0.810	903	13	922	11
0.07550	0.00074	3.4E-04	4.9E-05	25.2419	0.3684	33.119	0.393	0.1519	0.0155	1.484	0.019	0.810	911	13	924	11
0.08240	0.00152	7.5E-04	1.0E-04	25.2861	0.4850	33.940	0.395	0.1463	0.0203	1.458	0.030	0.670	880	17	913	18
0.08420	0.00209	1.0E-03	1.7E-04	24.4094	0.5300	33.311	0.811	0.1441	0.0232	1.399	0.040	0.580	868	19	889	23
0.08040	0.00126	5.9E-04	5.7E-05	28.3732	0.3522	36.197	0.513	0.1513	0.0135	1.509	0.023	0.600	908	11	934	14
<i>0.07840</i>	<i>0.00146</i>	<i>4.6E-04</i>	<i>7.9E-05</i>	<i>25.4201</i>	<i>0.3647</i>	<i>34.443</i>	<i>0.712</i>	<i>0.1449</i>	<i>0.0154</i>	<i>1.443</i>	<i>0.026</i>	<i>0.590</i>	<i>872</i>	<i>13</i>	<i>907</i>	<i>16</i>
0.08070	0.00202	7.1E-04	1.4E-04	26.5324	0.5894	34.908	0.667	0.1480	0.0235	1.448	0.038	0.610	890	19	909	23
0.07990	0.00118	8.0E-04	1.3E-04	25.9027	0.5668	34.370	0.384	0.1472	0.0232	1.398	0.030	0.770	885	19	888	18
0.07640	0.00178	5.8E-04	9.9E-05	26.6942	0.5710	35.662	0.661	0.1451	0.0225	1.370	0.035	0.640	874	18	876	21

CC1716 - France

0.05700	0.00045	3.0E-04	3.3E-05	10.1794	0.0461	36.679	0.148	0.0536	0.0064	0.389	0.011	0.573	336	2	334	3
0.05690	0.00082	2.8E-04	5.0E-05	9.7742	0.0620	36.158	0.267	0.0524	0.0079	0.382	0.018	0.436	329	3	329	5
<i>0.05570</i>	<i>0.00034</i>	<i>1.1E-04</i>	<i>1.4E-05</i>	<i>9.4716</i>	<i>0.0454</i>	<i>35.087</i>	<i>0.231</i>	<i>0.0530</i>	<i>0.0065</i>	<i>0.396</i>	<i>0.009</i>	<i>0.711</i>	<i>333</i>	<i>2</i>	<i>339</i>	<i>3</i>
0.05550	0.00059	1.8E-04	1.5E-05	10.2640	0.0637	36.788	0.203	0.0539	0.0076	0.395	0.014	0.564	339	3	338	4
0.05600	0.00065	2.4E-04	4.4E-05	9.4818	0.0928	35.239	0.225	0.0526	0.0109	0.382	0.017	0.643	331	4	329	5
0.05590	0.00050	2.0E-04	3.0E-05	11.5872	0.0663	40.413	0.205	0.0540	0.0073	0.395	0.012	0.600	339	2	338	4
0.05690	0.00054	2.5E-04	3.3E-05	10.0501	0.0300	36.901	0.243	0.0525	0.0054	0.388	0.012	0.450	330	2	333	3
0.05530	0.00029	1.2E-04	1.5E-05	10.2039	0.0370	36.860	0.240	0.0535	0.0057	0.397	0.008	0.714	336	2	339	2

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0.05580	0.00067	2.0E-04	3.5E-05	9.9751	0.0316	36.440	0.073	0.0531	0.0055	0.388	0.014	0.387	333	2	333	4
0.05400	0.00034	8.5E-05	1.2E-05	10.0629	0.0587	36.678	0.169	0.0532	0.0073	0.388	0.010	0.743	334	2	333	3
<i>0.05370</i>	<i>0.00029</i>	<i>4.4E-05</i>	<i>9.6E-06</i>	<i>24.8420</i>	<i>0.2041</i>	<i>66.090</i>	<i>0.719</i>	<i>0.0645</i>	<i>0.0093</i>	<i>0.473</i>	<i>0.011</i>	<i>0.861</i>	<i>403</i>	<i>4</i>	<i>393</i>	<i>4</i>
0.05340	0.00029	7.4E-05	1.1E-05	10.1228	0.0749	36.414	0.392	0.0540	0.0086	0.391	0.010	0.834	339	3	335	3
0.05500	0.00042	2.0E-04	2.7E-05	10.3061	0.0911	37.384	0.221	0.0530	0.0100	0.382	0.013	0.765	333	3	329	4
<i>0.05900</i>	<i>0.00146</i>	<i>4.6E-04</i>	<i>9.5E-05</i>	<i>9.5663</i>	<i>0.1072</i>	<i>35.473</i>	<i>0.256</i>	<i>0.0524</i>	<i>0.0125</i>	<i>0.379</i>	<i>0.032</i>	<i>0.392</i>	<i>329</i>	<i>4</i>	<i>327</i>	<i>9</i>
0.05570	0.00063	1.9E-04	4.2E-05	10.1039	0.0965	37.096	0.243	0.0525	0.0107	0.385	0.016	0.650	330	3	331	5

NP2 – Fujian China - Ferrotantalite standard

0.05993	0.00071	3.0E-04	6.9E-05	7.0804	0.0756	27.958	0.290	0.0610	0.0126	0.467	0.028	0.457	-	-	-	-
0.06661	0.00065	6.7E-04	3.8E-05	7.3258	0.0424	28.191	0.218	0.0619	0.0077	0.485	0.018	0.430	-	-	-	-
0.07396	0.00199	1.0E-03	1.1E-04	6.5220	0.1836	26.218	0.487	0.0614	0.0308	0.499	0.056	0.549	-	-	-	-
0.07846	0.00154	1.2E-03	6.0E-05	5.6770	0.2024	24.312	0.520	0.0603	0.0377	0.503	0.049	0.768	-	-	-	-
0.07900	0.00054	1.6E-03	6.1E-05	7.5638	0.1920	28.638	0.329	0.0614	0.0275	0.474	0.035	0.794	-	-	-	-
0.08870	0.00179	1.9E-03	1.6E-04	7.3643	0.1152	27.685	0.315	0.0626	0.0194	0.528	0.058	0.336	-	-	-	-
0.07930	0.00153	1.6E-03	1.1E-04	7.6902	0.0564	29.194	0.238	0.0606	0.0103	0.463	0.045	0.229	-	-	-	-
0.11627	0.00338	4.6E-03	3.6E-04	6.5616	0.1003	26.219	0.199	0.0580	0.0235	0.393	0.152	0.155	-	-	-	-
0.08780	0.00191	2.5E-03	2.7E-04	6.8786	0.0989	27.383	0.131	0.0589	0.0203	0.411	0.103	0.198	-	-	-	-
0.08010	0.00167	1.6E-03	7.9E-05	7.2391	0.1044	28.168	0.190	0.0602	0.0167	0.468	0.043	0.393	-	-	-	-
0.06610	0.00092	7.5E-04	1.2E-04	7.4841	0.0601	29.274	0.137	0.0596	0.0110	0.454	0.042	0.263	-	-	-	-
0.07430	0.00068	1.4E-03	1.3E-04	7.0632	0.0706	27.270	0.342	0.0621	0.0131	0.465	0.044	0.299	-	-	-	-
0.10239	0.00148	2.9E-03	2.3E-04	7.3032	0.1555	28.145	0.364	0.0595	0.0268	0.496	0.076	0.353	-	-	-	-
0.06850	0.00072	1.1E-03	1.3E-04	6.8034	0.0738	27.874	0.359	0.0580	0.0140	0.415	0.047	0.295	-	-	-	-
0.13431	0.00211	5.4E-03	1.7E-04	7.2398	0.1354	26.745	0.247	0.0610	0.0240	0.469	0.073	0.330	-	-	-	-
0.12428	0.00077	4.4E-03	2.4E-04	7.0983	0.1608	27.243	0.409	0.0593	0.0292	0.502	0.078	0.375	-	-	-	-
0.10801	0.00113	3.5E-03	9.4E-05	7.2085	0.1702	27.369	0.425	0.0606	0.0271	0.473	0.045	0.598	-	-	-	-

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0.06740	0.00034	1.1E-03	5.1E-05	7.3588	0.0651	28.513	0.132	0.0607	0.0107	0.434	0.021	0.519	-	-	-	-
0.07560	0.00098	1.6E-03	9.6E-05	7.2574	0.0540	28.546	0.243	0.0592	0.0102	0.432	0.038	0.271	-	-	-	-
0.06410	0.00096	5.7E-04	1.2E-04	6.1808	0.1799	25.884	0.565	0.0599	0.0316	0.461	0.050	0.631	-	-	-	-
0.08790	0.00104	2.2E-03	1.2E-04	7.5480	0.0631	28.744	0.137	0.0603	0.0115	0.470	0.042	0.273	-	-	-	-
0.07810	0.00105	1.5E-03	8.4E-05	7.1739	0.1150	28.538	0.313	0.0586	0.0184	0.451	0.037	0.496	-	-	-	-
0.09730	0.00117	2.8E-03	1.8E-04	7.2256	0.1166	27.945	0.581	0.0597	0.0205	0.471	0.061	0.334	-	-	-	-
0.08670	0.00135	2.2E-03	1.5E-04	7.1145	0.0348	28.039	0.235	0.0591	0.0088	0.447	0.055	0.160	-	-	-	-

NT2 - Altai

0.10073	0.00489	2.9E-03	2.4E-04	5.4920	0.0357	23.439	0.121	0.0600	0.0091	0.479	0.093	0.098	376	3	397	30
0.11744	0.00311	4.3E-03	2.1E-04	6.6873	0.1488	26.221	0.371	0.0591	0.0255	0.444	0.070	0.365	370	9	373	22
0.05672	0.00049	2.1E-04	2.8E-05	6.6742	0.0607	27.462	0.182	0.0592	0.0102	0.438	0.014	0.728	371	4	369	4
0.41897	0.00449	2.3E-02	5.2E-04	8.8966	0.1033	24.358	0.377	0.0560	0.0244	0.783	0.091	0.268	351	8	587	40
0.11403	0.00172	4.4E-03	1.9E-04	6.0084	0.1099	26.411	0.479	0.0524	0.0212	0.356	0.047	0.451	329	7	309	12
0.05772	0.00039	2.4E-04	4.8E-05	6.9065	0.0714	28.335	0.240	0.0583	0.0114	0.436	0.014	0.810	365	4	367	4
0.06553	0.00102	1.0E-03	7.7E-05	6.8381	0.0926	27.409	0.235	0.0600	0.0148	0.420	0.026	0.562	375	5	356	8
0.06680	0.00121	9.1E-04	6.7E-05	6.3942	0.1823	26.851	0.458	0.0580	0.0296	0.428	0.038	0.779	363	10	362	11
0.06050	0.00082	3.4E-04	6.1E-05	7.2211	0.1109	28.654	0.284	0.0598	0.0163	0.458	0.023	0.719	375	6	383	7
0.06570	0.00103	8.4E-04	9.4E-05	6.8356	0.1081	26.968	0.375	0.0617	0.0170	0.454	0.027	0.626	386	6	380	9
0.07750	0.00136	1.7E-03	1.7E-04	7.0115	0.0780	27.544	0.201	0.0602	0.0130	0.438	0.033	0.393	377	5	369	10
0.07760	0.00161	1.4E-03	1.1E-04	7.0777	0.0889	28.308	0.187	0.0586	0.0140	0.461	0.034	0.419	367	5	385	11
0.07130	0.00090	1.5E-03	1.6E-04	6.8404	0.1201	28.255	0.207	0.0567	0.0193	0.382	0.031	0.631	355	7	329	9
0.07220	0.00105	1.1E-03	1.4E-04	6.6598	0.0403	27.653	0.205	0.0574	0.0080	0.440	0.024	0.337	360	3	370	7
0.06180	0.00099	4.7E-04	8.2E-05	7.4986	0.0795	28.912	0.189	0.0611	0.0118	0.463	0.023	0.519	383	4	386	7
0.06950	0.00092	1.1E-03	1.6E-04	7.3459	0.1153	29.158	0.218	0.0584	0.0173	0.427	0.028	0.615	366	6	361	8
0.09870	0.00435	2.8E-03	3.1E-04	6.4697	0.1828	25.477	0.357	0.0617	0.0315	0.498	0.088	0.356	386	12	410	29

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0.06430	0.00056	6.7E-04	7.4E-05	7.2344	0.0329	28.680	0.149	0.0595	0.0065	0.447	0.014	0.475	373	2	375	4
<i>0.10911</i>	<i>0.00155</i>	<i>3.9E-03</i>	<i>2.3E-04</i>	<i>6.8114</i>	<i>0.1348</i>	<i>27.560</i>	<i>0.458</i>	<i>0.0561</i>	<i>0.0228</i>	<i>0.408</i>	<i>0.044</i>	<i>0.520</i>	<i>352</i>	<i>8</i>	<i>347</i>	<i>13</i>
<i>0.06060</i>	<i>0.00058</i>	<i>5.8E-04</i>	<i>4.7E-05</i>	<i>7.2751</i>	<i>0.0685</i>	<i>28.873</i>	<i>0.277</i>	<i>0.0593</i>	<i>0.0106</i>	<i>0.427</i>	<i>0.016</i>	<i>0.664</i>	<i>372</i>	<i>4</i>	<i>361</i>	<i>5</i>
0.07230	0.00112	1.2E-03	7.3E-05	7.0751	0.0744	28.440	0.175	0.0584	0.0119	0.441	0.025	0.476	366	4	371	8

A-1 - Altai - Manganocolumbite

0.21548	0.00326	1.1E-02	5.1E-04	2.7732	0.0454	21.937	0.149	-	-	-	-	-	-	-	-	-
0.54560	0.00640	3.2E-02	9.6E-04	6.3654	0.2061	20.667	0.287	-	-	-	-	-	-	-	-	-
0.15297	0.00227	7.1E-03	1.8E-04	2.6356	0.0355	22.428	0.178	-	-	-	-	-	-	-	-	-
0.06548	0.00035	9.7E-04	2.1E-05	2.1440	0.0139	22.427	0.127	-	-	-	-	-	-	-	-	-
0.48134	0.01104	2.9E-02	4.8E-04	3.5668	0.0778	20.606	0.270	-	-	-	-	-	-	-	-	-

General discussion, conclusions and perspectives

This discussion aims to involve all interpretations and thoughts that were developed during this thesis. Three main questions are addressed: (i) How do W-Sn deposits formed in the Nanling Range?, (ii) Why is there that much deposits in the Nanling Range at this period? and, (iii) Comparing to the FMC, why is the Nanling Range richer in W-Sn resources?

These three questions are successively addressed in each of the three parts of this discussion. Each part firstly develops the current models invoked in the literature and then proposes a discussion in the light of the results of this thesis.

This discussion tries to integrate this work in a broader perspective which is the common understanding of W-Sn hydrothermal deposits and the settings that could have been involved in the Nanling Range compared to other provinces.

1. Integrated model for W-Sn hydrothermal deposits

In the W-Sn literature, two models are proposed to explain the source of metals and processes able to transport and concentrate them.

The first and widely accepted model involves a magmatic source from which metal-enriched magmatic fluids are exsolved from a granitic intrusion. This model is supported by a wide range of fluid inclusion studies performed on W-Sn deposits worldwide (e.g. Beuchat et al., 2004; Carruzzo et al., 2004; Luders et al., 2009; Wei et al., 2012). The main arguments in favor of a magmatic origin of W-Sn are: $\delta^{18}\text{O}$ and δD values on quartz yielding in the commonly defined “magmatic” box (e.g. Chicharro et al., 2016; Wei et al., 2017), $\delta^{34}\text{S}$ values of pyrite and arsenopyrite yielding magmatic values (e.g. Wang et al., 2012) and the evidence of coexisting melt and fluid inclusions in quartz crystals (e.g. Hulsbosch et al., 2016). However, very few of these studies clearly demonstrated that these gangue minerals were strictly coeval with the

W-Sn minerals. The recent advances in infrared light measurements permitted to compare microthermometric observations between gangue and W-Sn minerals. Most papers dedicated to this topic were dealing with Chinese deposits located in the Nanling range (e.g. Wei et al., 2012, Ni et al., 2015). These studies agree on the inability to use gangue minerals as a proxy to describe ore-forming processes by microthermometry (P-T conditions).

The second model was mainly approached in the case of the Panasqueira deposit in Portugal. Polya et al. (2000) first raised the idea of meteoric fluids involved in the ore-forming processes. This idea was mainly developed from low δD measurements of fluid inclusions in quartz that could only be explained by an equilibration of the ore-forming fluid with the surrounding schist that also displayed low δD isotopic ratios. This observation was then renewed by Lecumberri-Sanchez et al. (2017) and it was concluded that fluid-rock interaction was a decisive process for the formation of tungsten deposits as presented in Figure 76.

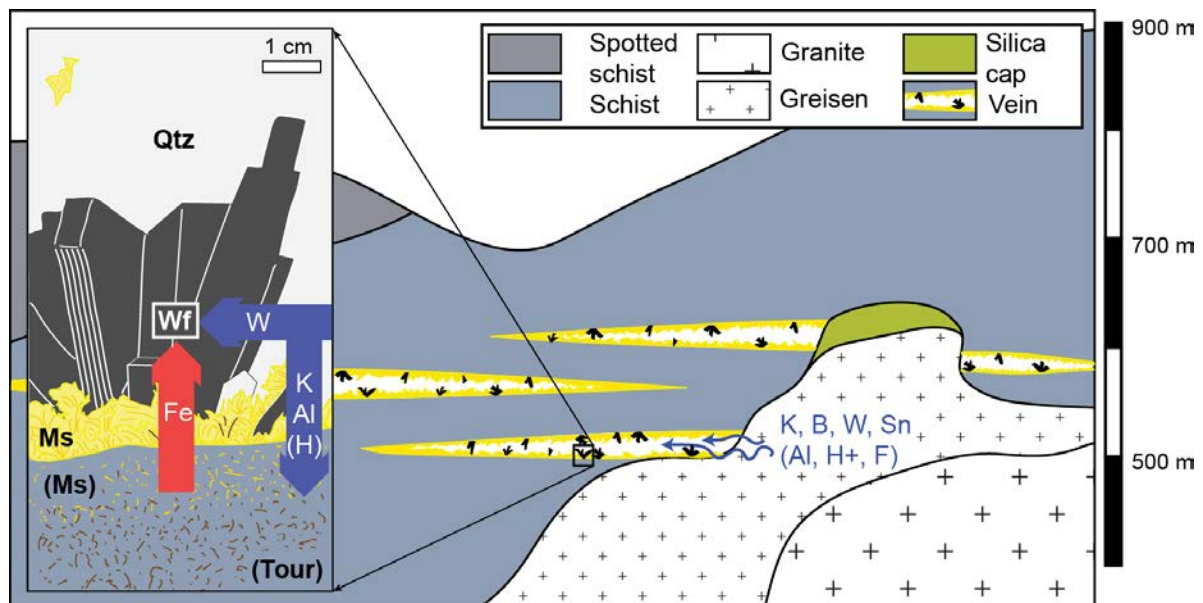


Figure 76. Schematic representation of fluid-rock interaction processes involved at Panasqueira deposit (Portugal). Wf = wolframite, Ms = muscovite, Tour = tourmaline, Qtz = quartz. (From Lecumberri-Sanchez et al., 2017)

This interpretation started to question the implication of granitic intrusions in the W-Sn ore-forming processes. The magmatic origin of W-Sn is not yet questioned but some studies raised the following alternative model: the granitic intrusion first yields magmatic fluids

enriching the perigranitic cover, then local metamorphic settings activate the circulation of external fluids in the crust in the shape of convective cells and finally leach the rare metals from the cover to concentrate them within fractures to form the veins (e.g. Wilkinson, 1990; Blamart, 1991; Zhao and Jiang, 2004; Vallance et al., 2001; Harlaux, 2017). This model is particularly developed in the case of Variscan deposits.

In both models, the prevailing process invoked for the W-Sn minerals precipitation is the mixing of the rare-metal-enriched fluid with a meteoric fluid (e.g. Wilkinson, 1990; Luders et al., 2009; Dolnicek et al., 2012). Other processes such as unmixing and boiling were also observed but interpreted as non-decisive for the ore mineral precipitation (e.g. Wei et al., 2012; Wei et al., 2017). However, such processes were interpreted from the observation of heterogeneous gas contents that were never observed in wolframite fluid inclusions (e.g. Wei et al., 2012; Ni et al., 2015).

This thesis agrees with the first model in the case of the Maoping and Piaotang deposits by multiple and integrated approaches performed on both gangue and ore minerals (see Chapter 2, 3 and 4). The differences between gangue and ore minerals could be enlighten and confirmed by other approaches than microthermometry such as stable isotopes (O, H) and LA-ICPMS. These complementary approaches permitted to propose an explanation for the inconsistency between measurements in gangue and ore minerals. These differences include, from the quartz to ore minerals crystallization: an increase in temperature, salinity, incompatible elements including metals and a decrease of the δD value. These characteristics were interpreted in this thesis as characteristic of two magmatic fluid pulses exsolved at different grades of differentiation of the granitic intrusion. The low δD isotopic ratio was interpreted as a possible H_2O degassing from the magma (see Chapter 4). Lecumberri-Sanchez et al. (2017) recently proposed an explanation for the wide interaction of ore-forming fluids with the surrounding rocks in Panasqueira as a necessary process providing for the iron contained in ferberite and explaining the heterogeneous filling in the veins. In the case of Maoping and Piaotang, wolframite crystallizes in the form of hubnerite. Due to the lack of fluid-

rock observations in the respective mines, this theory could not be inferred in our case. However, both the peraluminous granite and the schists in Maoping are manganese-depleted and could not have provided the necessary manganese to form hubnerite. Moreover, multiple deposits in the Nanling Range show veins crosscutting very differentiated granites that could not have provided for the iron and manganese content necessary to the crystallization of wolframite.

2. The W-Sn Jurassic-Cretaceous event in the Nanling Range

2.1 The timing of W-Sn mineralizations

W-Sn deposits are widely emplaced in the Nanling Range during the Trias-Cretaceous period with multiple pulses. However, it is believed that one great episode accounted for the emplacement of most of these deposits. Two models concerning this peak of emplacement granites and related W-Sn deposits in the Nanling Range are proposed at this day.

Most authors agree on a timing between 150 and 160 Ma. These range is associated with multiple dating analysis performed on gangue minerals as Re-Os on molybdenite (e.g. Feng et al., 2011; Zeng et al., 2011; Zhang et al., 2011), Ar-Ar on micas (e.g. Peng et al., 2006; Yuan et al., 2007; Zhai et al., 2010) and Sm-Nd on quartz fluid inclusions (Fu et al., 2009). Few studies also proposed a similar age by measuring U-Pb isotopic ratios in cassiterite (Bai et al., 2011; Zhang et al., 2017) but in the absence of appropriate external standard, the accuracy of this technique is still debated. This timing of emplacement is dated coevally to the associated biotite granites that are believed to be the source of tungsten and therefore, is commonly accepted as accurate.

Recently, Wang et al., (2016) raised some questions and inconsistencies about the current model adopted in the Nanling Range. This author proposes that the prevailing dating methods have some limitations such as low closure temperatures, low isotopic contents and dispersed initial isotopic values that induced errors in interpretation. Therefore, this author

provides new ages by dating hydrothermal zircons (U-Pb) as a new proxy to evaluate the timing of hydrothermal fluids transporting tungsten and proposes a new range of emplacement between 130 and 135 Ma. In the light of these findings, a new source of tungsten is proposed: the muscovite granites (Wang et al., 2017).

This thesis proposes (i) tangible arguments permitting to increment this discussion by the first direct dating measurements of wolframite in the Nanling range and yielding ages of ca. 160 Ma (see Chapter 5). This age is consistent with very recent attempts of U-Pb dating of cassiterite that also suggest a very early emplacement of the mineralization comparing to most dating results performed on gangue minerals. (ii) Multiple dating associated to the detailed paragenesis of the Maoping deposit raised evidences of a major resetting and/or alteration of isotopic systems in gangue minerals of these deposits due the multiple and overlapping fluid circulations. These major fluid events were dated at ca. 155 Ma as many studies before but clearly postdate the W-Sn mineralizing events. (iii) Dating of late filling phases such as xenotime yield an age of ca. 130 Ma, similarly to the hydrothermal zircons analyzed by Wang et al. (2016). At the Maoping deposit, a secondary event was identified as postponing the W-Sn event and crystallizing Nb-Ta-Zr-REE minerals in association with a supposed buried peralkaline intrusion. In their study, Wang et al. (2016) shows hydrothermal zircons pictures in association with fluorite, xenotime, monazite and sulphides. These phases and hydrothermal zircons are clearly identified in the Maoping deposit and are interpreted as posterior to the W-Sn mineralizing event (Legros et al., 2016). It is clear that two episodes are defined: a W-Sn episode at ca. 160 Ma in association with biotite granites and a second episode where mainly Rare Earth Elements mineralizations occur in association with Zr-Nb-Ta affinities that define another pulse of magmatism that is not of peraluminous affinity.

Considering a ca. 160 Ma timing for the W-Sn mineralizing event, the question raises of the consistency of the current orthomagmatic models in association with the emplacement of biotite granitoids. As suggested by Wang et al. (2016), these models are mostly based on dating results where gangue minerals are dated coevally to these granites. This thesis does

not argue on the magmatic origin of W-Sn but agrees on the possible inconsistency of such sources.

2.2 Why is it located in the Nanling Range?

Numerous models have proposed to relate the giant mineralization event of South China with the collision between the South China Craton and Indochina in the Triassic and the subduction of the Paleo-Pacific plate below the South China Block continental margin in the Triassic-Cretaceous period (e.g. Zhou and Li, 2000; Li and Li, 2007). However, models are still debated and the significance of the Jurassic-Cretaceous plutons is still controversial in the absence of clear evidence of a Jurassic subduction below the South China Block SE margin (Li et al., 2007; Chen et al., 2008; Jiang et al., 2009; Huang et al., 2013). The most recent work was pursued by Zhao et al. (2017) and reviewed all present hypothesis to propose a new model for the Jurassic-Cretaceous event emplacing the majority of W-Sn deposits. This model proposes that an E-W-trending mid-ocean ridge was subducted and then broke under the Nanling region. The model of flat-slab subduction followed by a break-off of the slab in the Early Jurassic and the slab roll-back and foundering during the Middle Jurassic was particularly developed by Li and Li (2007). This regime involved massive anorogenic magmatism as well as arc-related and bi-modal magmatism in the Nanling Range (Li and Li, 2007). The ridge would also have been under the Yangtze River region during the Early Cretaceous. From the Late Jurassic (ca. 160 Ma), the South China Block rotated clockwise (Zhao et al., 2017). This author suggests that the rotation of the ridge segment took place under the Nanling region and widespread magmatism associated to tungsten deposits of ca. 165-150 Ma before flat slab delamination

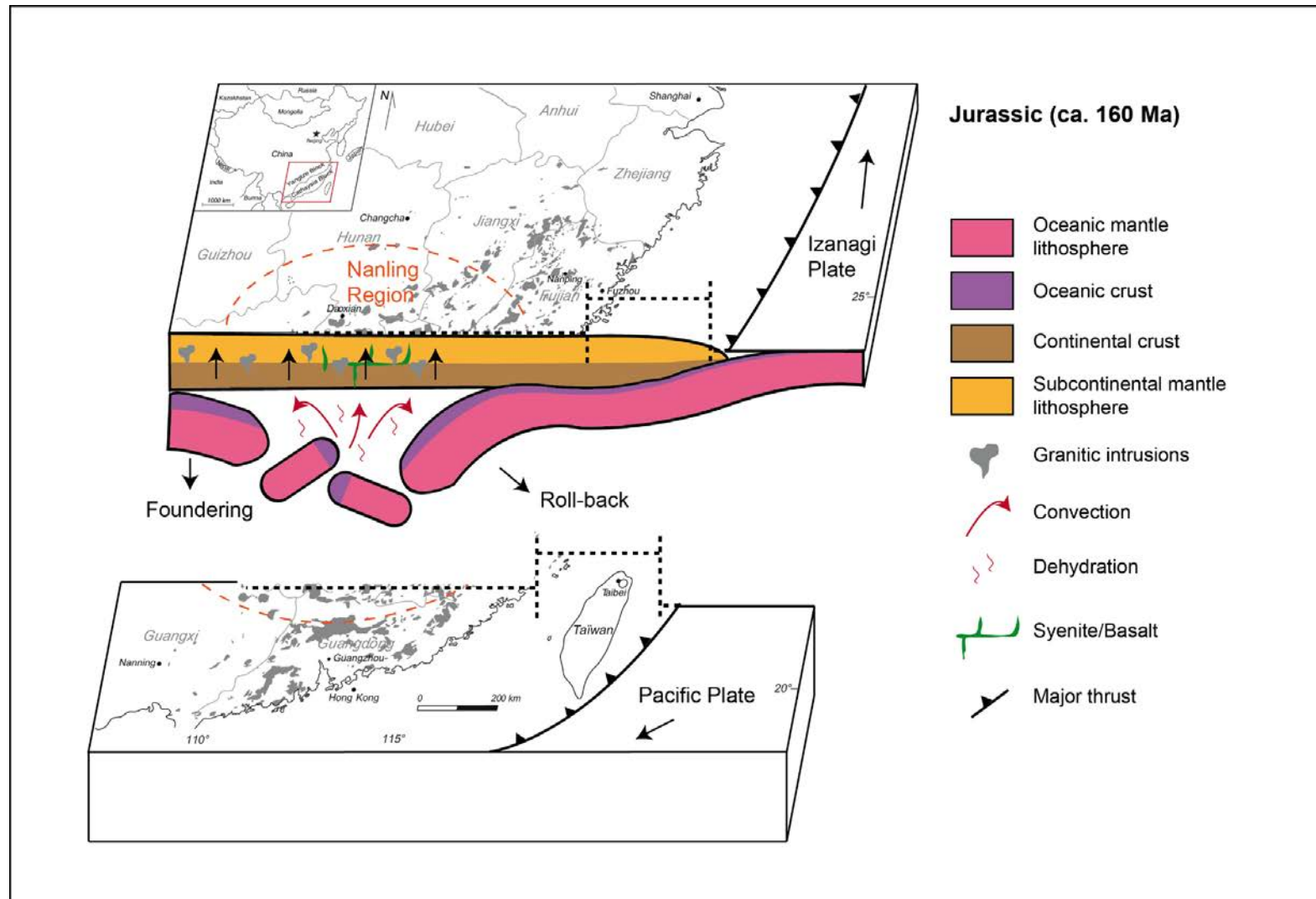


Figure 77. 3D diagram of the geodynamical settings during the Jurassic period and more precisely at ca. 160 Ma, in the South China Block (modified after Li and Li, 2007; Legros et al., 2016 and Zhao et al., 2017)

3. Comparison to the French Massif Central, an example from the Variscan belt

The French Massif Central (FMC) is part of the Variscan belt and hosts many W-Sn hydrothermal deposits that are specially associated to Carboniferous granitoids emplaced through the syn-orogenic compression and post-orogenic extension of the West European Variscan belt. Resources of at least 45,000 t WO₃ are estimated (Audion and Labbé, 2012).

2.1 Geodynamic settings

The Variscan belt extends from the Bohemian Massif to the east to Iberia and Morocco to west. The complex shape of the Variscan belt is due to the interaction of low-strain and high-strain domains of continental crust during the Laurussia and Gondwana collision. Low-strain domains are composed of thick continental crust poorly deformed during the Variscan orogeny while high-strain domain composed of thin continental crust endured extensive Variscan deformation (Kroner and Romer, 2013). The collision of thick continental crust induced new subduction settings emplaced behind the colliding block. Both the thinned continental crust and the sedimentary cover were involved in this intra-continental subduction. Part of the rocks involved were exhumed and preserved in high-strained domains such as the FMC (Romer and Kroner, 2010 and 2013). The transition from compressional to extensional tectonics possibly started around 300-310 Ma (e.g. Wilson et al., 2004; Kroner and Romer, 2013). Magmatism is associated to the emplacement of the high-strain domains, the late crustal melts emplaced posteriorly to the metamorphic units and to the post-variscan crustal extension (Kroner and Romer, 2013). These magmatic events respectively yielded peraluminous cordierite-biotite granites (Gueret-type, Margeride), two-peraluminous leucogranites (Limousin-type), the Velay granitic and migmatitic dome and highly fractionated peraluminous rare metal granites (e.g. Cuney et al., 2001; Melleton et al., 2015; Figure 78).

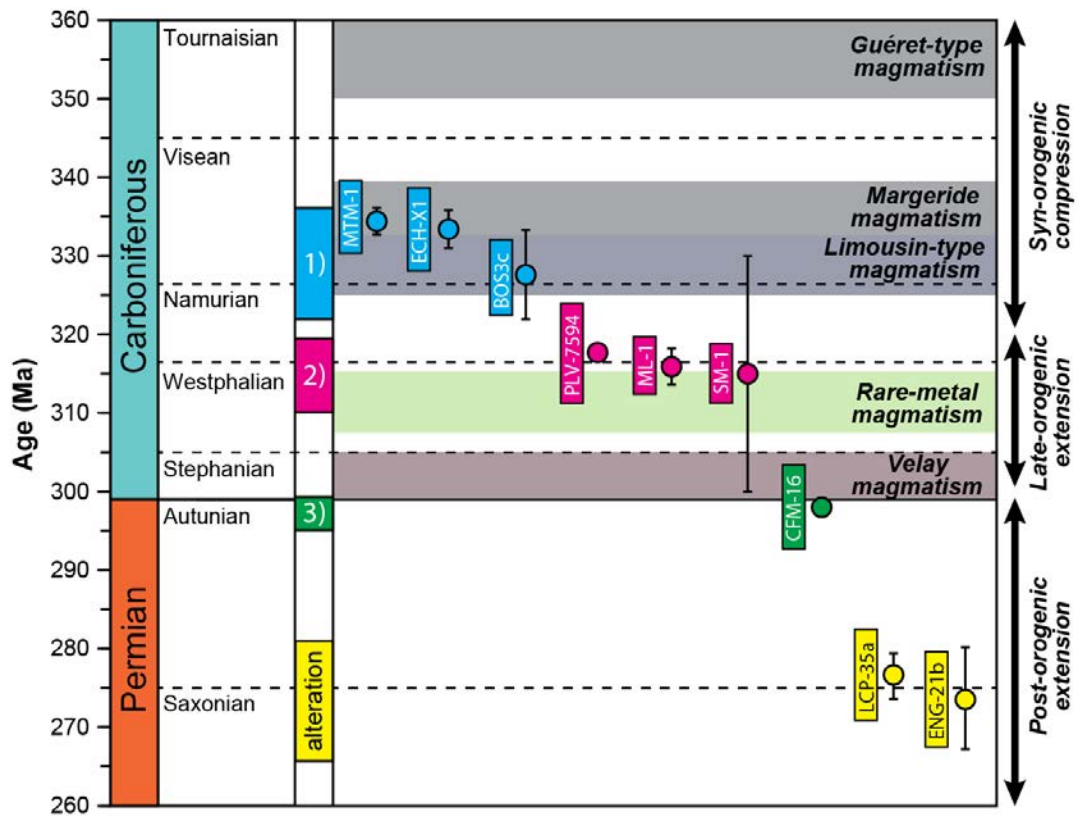


Figure 78. Wolframite U-Pb ages obtained in the FMC in comparison to the ages of the principal tectono-metamorphic and magmatic episodes (modified after Harlaux, et al., 2017 and references therein)

In conclusion, the FMC went through contrasting geodynamic settings during more than 40 Ma from syn-collisional regime to post-orogenic extension. These changing contexts were also observed in the Nanling Range during the Jurassic-Cretaceous period and seem to be adequate settings to produce W-Sn deposits, whether geodynamical models are significantly different in the FMC and the Nanling Range. Owing that, the main difference between the FMC and the Nanling Range that could explain the difference of W-Sn stocks in both regions, can reside in the melted material that could have been initially more enriched in the Nanling Range.

2.2 W-Sn deposits model

In the FMC, the magmatic source of W-Sn is believed to be derived from partial melting and metal mobilization from anomalously W-Sn enriched sedimentary rocks related to heat input from the mantle during continental collision, nappe stacking or post-orogenic crustal extension (Romer and Kroner 2015 and 2016). The following model is proposed in the FMC

(applied to the case of intermediate depth intrusions): a primary migration of the metals from the granite to the metasedimentary cover to create a metal-rich perigranitic stock through magmatic fluids and a second migration provoked by regional prograde metamorphism that remobilizes the perigranitic stock into drains as fractures until reaching a trap (Harlaux, 2017). Tungsten precipitation is believed to be due to the dilution of metamorphic metal-rich fluids by low-salinity cooler fluids (e.g. Vindel et al., 1995; Norohna et al., 1999).

It appears that the ore-processes involved in the FMC and the Nanling Range are similar: metal are believed to be of magmatic origin and dilution by a meteoric fluid would be the predominant precipitating process. The main difference in the models invoked resides in the implication of one or several migration processes. In the FMC as well as in the Variscan belt in general, a secondary migration by metamorphic fluids is enlighten by the lack of obvious magmatic fluids, tourmalinization, C-O-H-N-NaCl fluids, a rare-metal halo around the deposit and regional metamorphism widely observed in the surrounding rocks (Vallance et al., 2001; Harlaux, 2017). However, most fluid inclusion studies from the Variscan belt show the same microthermometric and isotopic values as in the Nanling Range: intermediate values between meteoric and metamorphic-magmatic values due to mixing. In this work, no such metamorphism as seen in the FMC could be observed nor does contact halo near the veins or the granite.

The question remains to know if such metamorphism is present in the Nanling Range but not observed due to the difficult visibility induced by lateritization or underground work or if it was absent and unnecessary in the context of the Nanling Range. The Variscan belt would appear as a special case of W-Sn deposit emplacement where the present metal stock contained in the melt was not sufficient to directly saturate the fluid and precipitation the veins and needed posterior remobilization to acquire a sufficient concentration of metals. The principal difference between the FMC and Nanling Range would therefore be the rate of pre-enrichment of the melt prior to the granite emplacement.

4. Conclusions

This thesis permitted to develop a petrological, mineralogical, fluid and timing view of hydrothermal W-Sn systems located in the southern Jiangxi province and give some new input in order to provide new elements of response in the field of W-Sn deposits.

(i) How do hydrothermal W-Sn deposits formed in the Nanling Range?

W-Sn vein-type deposits of the Nanling Range are all associated to granitic intrusions that are largely described as peraluminous. The veins, generally rooted in these granites, experience magmatic to hydrothermal conditions. During the hydrothermal settings, multiple fluids are identified and characterized as magmatic and meteoric. The main process involved is mixing (as often described). In this study, we defined that through time, multiple magmatic pulses of fluids with different P-T-x characteristics take turn and induce the crystallization of different mineral phases. These different magmatic pulses are believed to either be, from a magmatic source evolving through time, or exsolved from distinct intrusions (if the associated mineralogical assemblage varies). In our case, two magmatic fluids are defined as involved during the W-Sn mineralizing stage and discriminate the crystallization of gangue minerals from the crystallization of W-Sn minerals. These two magmatic fluids were identified as exsolved from the same peraluminous source that provided two pulses: a fluid exsolved from a low differentiated intrusion crystallizing the gangue minerals, and a fluid exsolved from a highly differentiated intrusion crystallizing W-Sn minerals.

(ii) Why is there that much deposits in the Nanling Range at this period?

The location and the timing of the W-Sn mineralizations in the Nanling Range are triggered by the geodynamical settings during the Jurassic-Cretaceous period. The W-Sn tungsten deposits are emplaced in a subduction context where the Paleo-Pacific plate is subducted under the Eurasia continent in the form of a flat-slab subduction. During the Jurassic, the slab broke-off and delamination under the Nanling range created a window

between the slab roll-back and slab foundering on each side and developed convective cells that enforced the rise of mixed magmas (crustal and asthenospheric components) within the crust.

(iii) Comparing to the FMC, why is the Nanling Range richer in W-Sn resources?

In term of hydrothermal processes involved in the formation of the W-Sn veins, only one process differs between the FMC and the Nanling Range which is the involvement of external fluids remobilizing tungsten widespread in the crust, within the veins. The absence of evidences concerning a possible remobilization within the crust in the Nanling Range may be explained by the timing of emplacement of both regions. Indeed, the FMC W-Sn deposits were emplaced in an immature crust where remobilization and concentration of rare metals was possible while the Nanling W-Sn deposits appear at a timing where the crust is already mature and underwent multiple metamorphism event that already remobilized these elements from the crust. This hypothesis could explain the lack the boron and therefore, tourmaline, in most deposits of the Nanling Range. However, these processes do not explain the wide difference of resources between the two regions.

Giving this study, the difference between the Nanling Range and the FMC resides in pre- granite emplacement processes. As granites associated to W-Sn deposits in the Nanling do not seem to drastically differ from the peraluminous granites emplaced in the FMC, the key of understanding the differences between these two provinces would reside in the characterization of the enriched melts that emplaced the granites, and their sources.

5. Perspectives

In the light of this work, further studies would be needed to enhance our knowledge of W-Sn deposits and particularly in the Nanling Range.

Firstly, further work in and around the mines is needed and particularly in term of structure. This work could not be done during this thesis due to a lack of time but could help constraint the conditions of emplacement of these deposit as exposed in the case of the Panasqueira mine by Jacques et al. (2017).

Secondly, very rare articles at this day provide substantial petrographic and mineralogical descriptive work. It would be interesting to compare detailed paragenesis at the scale of the province in order to provide some possible regional observations. Moreover, detailed paragenesis are a prerequisite to geochemical, fluid inclusion and dating studies. A lot of data are provided in the literature without any mineralogical chronology and may need to be context.

It would also be interesting to find and study melt inclusions to constraint the magmatic processes before the transition toward hydrothermal conditions. In term of fluid inclusions, it would be necessary to increment the number of fluid inclusion study in the Nanling Range with systematic study of opaque minerals such as wolframite and integrated studies gathering multiple approaches. Moreover, the need of experimental work seems crucial to our understanding of processes able to transport and precipitate W-Sn minerals.

Finally, as concluded in the discussion, further little scale work needs to be done on the granitic intrusions associated to the W-Sn veins in order to characterize the very first step of concentration of these metals in the melt and in particular in the Nanling Range.

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Résumé

Le tungstène est défini comme “ressource minérale critique” par la Commission Européenne. La province de Jiangxi, située au sud-est de la Chine, dans le bloc Cathaysia, représente 90 % des réserves en tungstène chinoises. Ce travail est basé sur l'étude des gisements hydrothermaux à W-Sn de Maoping et Piaotang, tous deux situés dans le district de Dayu (sud de la province de Jiangxi). Cette thèse a permis de (i) développer des traceurs pétrographiques et minéralogiques de processus minéralisateurs à travers des études paragénétiques détaillées et la géochimie des micas lithinifères et donc d'apporter un modèle impliquant des fluides multiples se chevauchant dans le temps et associés à plusieurs épisodes distincts de mise en place de minéralisations en métaux rares, (ii) définir par l'étude d'inclusions fluides deux processus fluides comme seul responsables de la précipitation de la minéralisation dans ces gisements « géants » : la différenciation magmatique de granites peralumineux et des processus de mélange, et (iii) de développer des approches de datation associés à ces systèmes montrant que la minéralisation en tungstène se met en place aux alentours de 160 Ma, antérieurement à la plupart des âges obtenus sur les minéraux de gangue datés dans cette zone et défini alors une remise à zéro majeure des systèmes isotopiques par de multiples circulations fluides entre 150 et 155 Ma. De plus, les stades post-minéralisations ont pu être définis pour la première fois et révèlent l'implication de magmatisme peralkalin impliqué dans la précipitation de minéralisations à Nb-Ta-Y-REE aux alentours de 130 Ma. A la lumière de cette observation, cette thèse s'est aussi tournée vers le développement de méthodes de datation *in situ* sur columbo-tantalite.

Abstract

Tungsten is defined as a “critical mineral resource” by the European Commission. The Jiangxi province, located in the southeastern part of China, in the Cathaysia block, represents 90% of the Chinese tungsten resources. This work is based on the study of the Maoping and Piaotang W-Sn hydrothermal deposits located in the Dayu district (southern Jiangxi). This thesis managed to (i) develop mineralogical and petrological tracers of ore-forming processes through detailed paragenetic sequences and geochemistry of Li-micas and shows that multiple overlapping fluids associated to several and distinct rare-metal mineralizing stages, (ii) distinguish by fluid inclusions studies that peraluminous magmatic differentiation and mixing processes are the only prerequisite for the formation of these giant deposits, and (iii) develop dating approaches associated to these systems to demonstrate that the W mineralization formed at ca. 160 Ma, prior to most ages obtained on gangue minerals in the area, defining a major resetting of isotopic systems due to multiple fluid circulations around 150-155 Ma. Moreover, post-“silicate-oxide” stages have been defined for the first time and reveal the implication of peralkaline new fluid sources involved in the precipitation of Nb-Ta-Y-REE minerals at ca. 130 Ma. In the light of these results, this thesis gives new developments for *in situ* direct dating of ore-bearing minerals such as columbo-tantalite.