

Coupling between plasticity and phase transformation in iron

Nicolas Bruzy

▶ To cite this version:

Nicolas Bruzy. Coupling between plasticity and phase transformation in iron. Solid mechanics [physics.class-ph]. École centrale de Nantes, 2018. English. NNT: 2018ECDN0056. tel-02355314

HAL Id: tel-02355314 https://theses.hal.science/tel-02355314

Submitted on 8 Nov 2019

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers. L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.





THESE DE DOCTORAT DE

L'ÉCOLE CENTRALE DE NANTES

COMUE UNIVERSITE BRETAGNE LOIRE

ECOLE DOCTORALE N° 602 Sciences pour l'Ingénieur Spécialité : Mécanique des Solides, des Matériaux, des Structures et des Surfaces

Par Nicolas Bruzy

Couplage entre plasticité et transformation de phase dans le Fer

Étude par corrélation d'images et modélisation

Thèse présentée et soutenue à Nantes, le 14 décembre 2018 Unité de recherche : Institut de Recherche en Génie Civil et Mécanique, UMR CNRS 6183

Rapporteurs avant soutenance :

Éric Charkaluk Bertrand Wattrisse Directeur de Recherche CNRS, LMS, École Polytechnique Professeur des Universités, LMGC, Université de Montpellier

Composition du Jury :

Brigitte Bacroix	Directrice de Recherche CNRS, LSPM, Université Paris 13
Présidente du Jury	
Guillaume Kermouche	Professeur des écoles des Mines, LGF,
Éric Charkaluk	Directeur de Recherche CNRS, LMS, École Polytechnique
Bertrand Wattrisse	Professeur des Universités, LMGC, Université de Montpellier
Michel Coret	Professeur des Universités, GeM, Centrale Nantes
Directeur de thèse	
Laurent Stainier	Professeur des Universités, GeM, Centrale Nantes
Co-directeur de thèse	
Bertrand Huneau	Maître de conférences, GeM, Centrale Nantes
Co-encadrant de thèse	

Résumé substantiel

Les alliages à base de fer peuvent présenter une grande variété de microstructures. Cela tient à la composition des alliages, à leur histoire thermomécanique mais aussi à l'une des particularités du fer qui est de présenter deux structures allotropiques stables en dessous de sa température de fusion: une structure cubique centrée (CC) stable à température ambiante et au-dessus de 1394°C, et une structure cubique faces centrées (CFC) stable entre 910°C et 1394°C. Le passage d'une structure à l'autre est un phénomène complexe mettant en jeu l'état mécanique du matériau, ce qui en fait un sujet d'étude riche. Afin de se concentrer sur le couplage entre l'état mécanique du matériau et les changements dans sa microstructure, un fer haute pureté a été choisi comme matériau de cette étude.

Comme la plupart des métaux, le fer se trouve généralement sous forme polycristalline. La régularité de l'arrangement atomique est rompue par la présence de défauts, notamment de dislocations et de joints de grains. Ces défauts sont le siège d'incompatibilités donnant lieu à des déformations locales que le matériau cherche à réduire. Les mécanismes de restauration sont un exemple de phénomène impliquant une évolution notable de la population de défauts au sein du matériau.

La transformation allotropique d'une phase ferritique de structure CC vers une structure austénitique de structure CFC au cours du chauffage implique elle-aussi des évolutions dans la répartition des défauts au sein de la microstructure. La complexité de son étude résulte dans le fait que la répartition des défauts dans le matériau influence l'initiation de la transformation qui en retour induit une accomodation élastoplastique de l'incompatibilité de volume entre ferrite et austénite. Dans le cas de la transformation austénite-ferrite, deux mécanismes de transformation peuvent mis être en avant. Si le refroidissement est rapide, le matériau se transforme par un mouvement coordonné des atomes. La transormation est dite displacive. A l'inverse, si le refroidissement est lent, un front entre la phrase parente et la phase fille progresse régulièrement dans le matériau et les déformations à l'interface entre les deux phases sont relaxées par auto-diffusion. On dit alors que la transformation est diffusive. Chacun de ces deux mécanismes nécessite des descriptions adéquates en termes de phénomènes microstructuraux et cinétiques.

A l'échelle macroscopique, il est aisé de constater les effets macroscopiques du changement de volume entre ferrite et austénite en réalisant un essai de dilatométrie et en observant une contraction de l'échantillon au moment de la transformation ferrite-austénite. A l'échelle d'un groupe d'atomes, des modèles portant sur la forme des sites de germination ont été développés et l'influence de la présence de défauts peut être expliquée qualitativement. A mi-chemin entre les deux, il serait intéressant de disposer d'un moyen de caractériser le couplage entre l'état mécanique du matériau et ses évolutions microstructurales à l'échelle de quelques grains.

Le principal objectif de ce travail est de mettre au point un moyen expérimental pour l'observation des changements de phase dans le fer à l'échelle des grains. A cette échelle, il n'est pas possible d'observer les mouvements individuels des atomes. En revanche, plusieurs marqueurs mésoscopiques peuvent être exploités pour mettre en évidence le changement de phase : la formation et le mouvement de nouveaux interfaces entre les phases, la modification des propriétés de diffraction locales du matériau ou les perturbations du champ de déformation autour de la phase en formation. C'est ce dernier marqueur qui a été retenu dans le cadre de ce travail car il apparaît comme un moyen naturel d'étudier le couplage entre transformation et état mécanique.

Un dispositif expérimental a été conçu pour chauffer et refroidir des échantillons autour de la température de transformation et extraire l'évolution des champs cinématiques au niveau de leur suface. Les échantillons sont chauffés par effet Joule tandis qu'une caméra optique haute résolution capture des images de la surface. Un logiciel de corrélation d'images numériques, UFreckles, est ensuite utilisé pour calculer des valeurs de déplacement en tout point de la surface et en extraire les déformations locales. Des aménagements sont faits pour que la qualité des images ne se dégrade pas tandis que les échantillons sont portés à des températures supérieures à 900°C.

Des essais préliminaires ont été réalisés sur des échantillons de fer industriel Armco pour évaluer la fiabilité du dispositif. L'apparition de la transformation ferrite-austénite est trahie par des déformations localisées au sein du matériau. La déformation de cisaillement maximale est proposée comme le meilleur outil pour identifier le début de la transformation et caractériser son intensité. En moyennant les composantes du tenseur de déformation sur tout l'échantillon, un comportement caractéristique en dilatation est retrouvé, ce qui fournit une validation du protocole expérimental. En revanche, le diamètre moyen des grains dans les échantillons de fer Armco n'est pas suffisant pour caractériser les hétérogénéités de déformation à l'intérieur des grains.

Plusieurs traitements thermomécaniques ont été explorés afin de rendre la microstructure initiale plus grossière. Trois traitements ont conduit à des résultats satisfaisants : un traitement de recristallisation primaire après laminage, un traitement de croissance de grain en phase austénitique et un traitement de strain annealing. Dans le cas des échantillons ayant été recristallisés, le matériau considéré est du fer haute pureté. Cela permet d'éliminer les effets relatifs à la diffusion d'éléments chimiques autres que le fer.

Dans les trois cas, la taille des grains est suffisante pour mettre en évidence que les zones de localisation de la déformation se situent à proximité des joints de grains et plus particulièrement des points triples. Cela confirme que du fait des incompatibilités d'orientations entre grains, les joints de grains constituent des sites thermodynamiquement favorables pour la germination de la nouvelle phase. Le recours à plusieurs méthodes de préparation d'échantillons permet d'étudier l'influence de la microstructure initiale sur le déroulement de la transformation ferrite-austénite. Dans le cas des échantillons ayant subi un traitement de strain annealing, les mêmes tendances sont observées quant à la localisation de la déformation au moment où la transformation a lieu. En revanche, les très petits grains de la microstructure initiale ont constitué des sites de germination préférentiels au début de la transformation, ce qui rend plus difficile l'interprétation des champs cinématiques obtenus.

Des cartographies par diffraction des électrons rétro-diffusés (EBSD) sont réalisées avant et après les tests afin de suivre les évolutions dans la microstructure des échantillons. La microstructure finale présente des désorientations intragranulaires caractéristiques de phénomènes plastiques. De plus, elle montre que la présence d'un gradient de température encourage plus la croissance que la germination de nouveaux grains, ce qui conduit à une microstructure allongée. Un inconvénient du protocole expérimental est que l'information sur la morphologie de la phase austénite en température est perdue. Pour palier cela, une méthode est mise au point pour déterminer son orientation. Elle consiste à supposer que tous les points matériels suivent le même chemin de transformation, ce qui présuppose une transformation diffusive de ferrite vers austénite et une transformation displacive d'austénite vers ferrite. Ce chemin de transformation conduit à un ensemble de désorientations possibles entre les grains de ferrite dans leurs états intiaux et finaux qui sont comparées aux mesures expérimentales.

Pour terminer, une comparaison est faite entre le comportement en transformation d'un échantillon de référence non déformé et d'un échantillon plastiquement déformé en traction avant le chauffage. Contrairement à ce qui pouvait être attendu, la déformation est moins localisée dans le cas des échantillons pré-déformés, ce qui met en évidence que les sites de germination se concentrent dans les zones comportant la plus grande densité de défauts. Par ailleurs, les zones de localisation de la déformation sont plus diffuses. Cela peut s'interpréter en considérant que les premiers moments de la croissance de la nouvelle phase sont pilotés par le gradient local de défauts, ce qui induit une croissance anisotrope.

Le principal obstacle à la poursuite de l'interprétation des résultats expérimentaux est que seul le champ de déformation est obtenu. Les densités locales de dislocations ne sont pas connues. Un travail de modélisation est mené à bien pour aborder ce problème et tenter de découvrir comment l'intervention de la transformation ferrite-austénite réorganise la population locale de défauts. Cela réclame de coupler au sein d'un même modèles des ingrédients relevant du comportement mécanique du matériau, de la thermodynamique des changements de phase, et de la thermique. Plusieurs philosophies de modélisation peuvent être envisagées selon que la transformation de phase est vue comme un problème de mise à jour d'une interface, comme la recherche de l'équilibre d'un mélange de phases, ou comme l'étude du comportement d'un système thermodynamique hors équilibre. C'est ce troisième axe qui est approfondi dans le cadre de cette thèse. Une méthodologie variationnelle est adoptée pour résoudre le couplage entre les différentes physiques impliquées dans l'étude des transformations ferrite-austénite et austénite-ferrite.

Dans un premier temps, le modèle est construit au niveau d'un point matériel. L'accent est mis sur le maintien de la consistance du modèle par rapport aux lois de la thermodynamique. Deux points sont traités avec une attention particulière. Le premier a trait au choix de modèles de plasticité cristalline représentant au mieux le comportement des phases ferritique et austénitique. Le second concerne les hypothèses relatives aux interactions entre phases. L'originalité de l'approche proposée est que moyennant un ajustement de la loi de mélange sur les énergies libres et les potentiels de dissipation, des mécanismes displacifs et diffusifs peuvent être modélisés au sein d'un même formalisme. Dans le premier cas, appelé modèle SLM, la formation de la phase fille intervient comme un moyen de réduire les déformations locales tandis que dans le second cas, appelé modèle ELM, un variant de la phase fille apparait dès lors qu'il est le variant le plus favorisé thermodynamiquement.

Ce modèle 0D est ensuite intégré à la biliothèque de calcul éléments finis Zorglib, développée au laboratoire GeM. Le problème étant résolu de manière itérative, le modèle est tout d'abord discrétisé en temps. Cette forme fait apparaître l'un des principaux avantages du formalisme variationnel : la détermination des incréments des variables internes revêt un sens physique en termes d'optimalité par rapport à l'énergie libre du matériau.

Des calculs sont d'abord conduits sur des domaines monocristallins pour illustrer le potentiel des modèles ELM et SLM. Aucun traitement des interfaces n'étant intégré dans le modèle, des hétérogénéitiés sont introduites au travers de solicitations mécaniques afin d'éviter que tous les points du domaine ne se transforment en même temps. Lorsque plusieurs variants de la phase fille sont considérés, le modèle ELM sélectionne en chaque point le variant le plus favorable énergétiquement tandis que le modèle SLM converge vers le mélange de ces variants qui limite au mieux les déformations locales.

D'importants coûts de calcul ont limité la taille des microstructures testées. Le comportement d'un échantillon polycrystallin soumis à un chauffage ou un refroidissement est étudié dans un premier temps sur une microstructure de référence contenant trois grains puis sur une microstructure issue d'une simplification des données EBSD. De manière générale, les étapes de la formation de la phase fille sont bien représentées. En particulier, en présence d'un gradient de température, l'échantillon se transforme à mesure que le front de transformation peut avancer dans l'échantillon. En revanche, les champs de déformation obtenus après la transformation sont décevants dans le sens où ils ne présentent aucune localisation comme cela peut être le cas expérimentalement. Cela limite grandement la pertinence de la comparaison entre les tendances expérimentales et numériques.

Contents

1	Intr	oductio	n: Microstructure evolutions in pure iron	1			
	1.1	Pure in	ron	2			
		1.1.1	General properties	2			
		1.1.2	Crystals	2			
		1.1.3	Defects in the crystalline structure	3			
	1.2	Ferrite	grains evolution	5			
		1.2.1	Recovery	5			
		1.2.2	Primary recrystallization	5			
		1.2.3	Grain growth	6			
	1.3	Allotro	opic transformation	7			
		1.3.1	Diffusive vs displacive mechanisms	7			
		1.3.2	Microstructures taxonomy	8			
		1.3.3	Orientation relationship	9			
		1.3.4	Mechanisms of the diffusive transformation	9			
		1.3.5	Kinetics	11			
		1.3.6	Summary	12			
	1.4	Coupli	ing between mechanics and transformation	12			
		1.4.1	Mechanical effect of the transformation	12			
		1.4.2	Influence of prior deformation on the allotropic transformation	13			
	1.5 Conclusion						
		1.5.1	Remaining challenges on the mechanics-transformation coupling	14			
		1.5.2	Motivation for the present work	15			
2	Desi	gn of a	n experimental set-up for a mesoscopic study of allotropic transforma-	10			
	tion	s in iror	1	19			
	2.1	Indica	tors of transformation occurrence	19			
		2.1.1	Boundary motion tracking	20			
		2.1.2	Phase contents tracking	20			
		2.1.3	Mechanical effect tracking: DIC	21			
	2.2	Digita	I Image Correlation	22			
		2.2.1	General idea behind DIC	22			
		2.2.2	High-resolution high-temperature DIC	22			
		2.2.3	Post-processing of the experimental data	24			
	2.3	Design	n of the experimental set-up	26			
		2.3.1	Samples design	26			
		2.3.2	Principle of the experiment	27			

CON	TENTS
-----	-------

		2.3.3	Heating system	28
		2.3.4	Optical system	30
		2.3.5	Conclusion: general view of the experimental equipment	31
	2.4	Valida	tion of the experimental set-up: tests on Armco iron sample	32
		2.4.1	Material	32
		2.4.2	Temperature solicitation	32
		2.1.2 2.4.3	DIC computations	33
		2.4.3 2.4.4	Macroscopic response	33
		2.7.7	Macroscopic response	34
	25	Conch		35
	2.3	conen		00
3	Mes	oscale e	experimental investigation of the interplay between mechanics and al	
	lotro	opic tra	nsformation	39
	3.1	Sampl	es preparation	40
		3.1.1	Need for coarse microstructures	40
		3.1.2	Recrystallized samples	40
		3.1.3	Austenized samples	40
		3.1.4	Strain annealed samples	41
		3.1.5	Conclusion: tested samples nomenclature	42
	3.2	Procee	lure for the mesoscopic study of allotropic transformations	43
		3.2.1	Pre and post mortem EBSD	43
		3.2.2	Temperature loading	44
	3.3	Result	s of heating and cooling tests on undeformed samples	44
		3.3.1	Recrystallized samples	44
		3.3.2	Reconstruction of the austenite phase	52
		3.3.3	Austenized samples	57
	3.4	Result	s of heating and cooling tests on deformed samples	59
		3.4.1	Introduction of plastic deformation	59
		3.4.2	Results obtained for strain annealed samples	60
		3.4.3	Results obtained for recrystallized samples	62
	3.5	Conclu	usion on the experimental work	63
		3.5.1	Viability of the method	63
		3.5.2	Results highlights	64
		3.5.3	Limitations of the experimental procedure	64
4	Соп	nled th	ermo-mechanical model for the mesoscopic transformational behavio	r
	of ir	on alloy	ys	67
	4.1	Motiva	ation for the development of a model	67
	4.2	Mesos	copic description of the thermo-mechanical behavior of iron	68
		4.2.1	Basics of crystal plasticity	68
		4.2.2	BCC phase behavior	70
		4.2.3	FCC phase behavior	73
	4.3	Dealin	g with phase changes in mesoscopic models	74
		4.3.1	Geometry-based approach	74
		4.3.2	Phase-field methods	75
		4.3.3	Variational models	75
			· · · · · · · · · · · · · · · · · · ·	

				ix	
	4.4 Efficient coupling between physics through variational methodolog			76	
		4.4.1	Laws of thermodynamics	76	
		4.4.2	Generalized Standard Material description	76	
		4.4.3	Standard material hypothesis	77	
		4.4.4	Heat conduction potential	77	
	4.5	Rephr	asing of crystal plasticity models	78	
		4.5.1	Thermomechanical problem	78	
		4.5.2	Variational expression for the dissipation	78	
		4.5.3	Helmholtz free energy	79	
		4.5.4	Intrinsic dissipation	80	
	4.6 Application to the case of metallic materials undergoing solid state phase				
		4.6.1	Main hypotheses	80	
		4.6.2	Latent heat of transformation	81	
		4.6.3	Dissipation	81	
		4.6.4	Law of mixtures on strains (SLM model)	81	
		4.6.5	Law of mixtures on free energies (ELM model)	82	
		4.6.6	Tangent modulus	83	
	4.7	Concl	usion on the model formulation	84	
_					
5	Nun	Numerical implementation of a variational model for the simulation of polycrys-			
		alline behavior with phase change			
	5.1	Discre	tete form of the material point model	88	
		5.1.1	Thermomechanical response of a polycrystal	88	
		5.1.2	Calculation over a time increment	88	
		5.1.3	Consistent update of dissipation	89	
		5.1.4	Mechanical internal variables update	89	
		5.1.5	Minimization strategy	90	
	5.2	Applic	cation of the ELM model to the ferrite-to-austenite transformation	92	
		5.2.1	Material properties	92	
		5.2.2	Transformation behaviour of a monocrystal	92	
		5.2.3	Transformation behaviour of a polycrystal	94	
	5.3	Respo	nse of a polycrystal under continuous heating	95	
		5.3.1	Main challenges brought by dealing with experiment-like samples	95	
		5.3.2	Construction of the numerical microstructure	97	
		5.3.3	Estimation of the macroscopic response of the sample	97	
		5.3.4	Mesoscopic behavior obtained with the ELM model	99	
		5.3.5	Comparison with the SLM model	101	
	5.4	Applic	cation of the SLM model to the austenite-to-ferrite transformation	101	
	5.5	5 Conclusion on numerical experiments		102	
6	Con	clusion		105	

List of Symbols and Abbreviations

a	First-order tensor
\underline{A}	Second-order tensor
<u>I</u>	Identity of second-order tensors
<u>A</u>	Forth-order tensor
\underline{A}^{T}	Transpose
<u>A.B</u>	Tensor dot product
<u>A</u> : <u>B</u>	Tensor double dot product
BCC	Body Centered Cubic
FCC	Face Centered Cubic
dof	degree of freedom
OR	Orientation Relationship
KS	Kurdjumov-Sachs orientation relationship
NW	Nishiyama-Wasserman orientation relationship
KJMA	Kolmogorov-Johnson-Mehl-Avrami
EBSD	Electron BackScattered Diffraction
DIC	Digital Image Correlation
MSS	Maximum Shear Stress
CRSS	Critical Resolved Shear Stress
SLM	Model based on a Law of Mixtures on the elastic Strain
ELM	Model based on a Law of Mixtures on the free Energy

General introduction

Ab initio calculations, Molecular Dynamics, Discrete Dislocation Dynamics, Crystal Plasticity: in the recent years, many numerical tools have been developed to gain understanding on the physical and mechanical behaviours of metallic materials at fine scales. It seems today that computational cost is the only barrier to a parameter-free model for the prediction of metallic parts response, where the parameters at one scale would be obtained from computations at a finer scale. In contrast to this, the spatial resolution of the experimental devices available to materials science researchers have improved and tend towards the exploration of local material properties. At the crossroads of these two trajectories is the need for simple test cases that would be complex enough to provide a means of validation of the numerical model yet accessible to modern experimental tools.

The objective of this thesis is to come up with an experimental observation that could be one of these validation cases. The focus has been on two main aspects to achieve this goal. First, the present developments will be conducted at an intermediate scale. Neither a group of packed atoms nor an entire part are considered but a small number of grains in a polycrystal. Secondly, the physics in the problem have been reduced to their minimum so that the observed behaviours could be simulated without plugging in too many numerical tools. In particular, the material that will be the subject of this study is pure iron. Working with a pure element removes from the scope of phenomena to be considered many chemistry-related events: partition into several chemical compounds, precipitates formation and so on. Henceforth the response of the material is mainly driven by a competition between the repartition of mechanical defects inside its microstructure and the changes in the microstructure itself upon heating or cooling.

The criteria to be respected in the testing being known, it remains to study a phenomenon that represents this competition between the population of local defects and the grain organization of iron. Emphasis is put on the allotropic ferrite-to-austenite and austenite-to-ferrite transformations. Indeed, iron has the particularity to exhibit two stable structural forms. Ferrite, stable at room temperature, transforms upon heating into austenite. Not only the mechanisms responsible for this transformation are influenced by the mechanical state of iron but there is a difference in volume between ferrite and austenite that induces an evolution of the population of defects. Consequently, the ferrite-to-austenite and the austenite-to-ferrite transformations are ideal candidates for a study on the coupling between the microstructure and the mechanical state of iron.

When it is proven that it is possible to submit an iron sample to a thermomechanical loading and to monitor its response at the scale of a few grains, an effort will be made to relate this response to elementary transformation mechanisms. To this end, the study of ferrite-toaustenite-to-ferrite transformation cycles is of particular interest because it can be checked that the proposed mechanisms are coherent with the measurements of the initial and final grain orientations. Besides, this may reflect industrial issues because it is frequent to cross the transformation temperature during the thermomechanical treatments of iron-based alloys.

In parallel, an insight on the path that could be followed to reproduce the experimental trends through simulation will be presented. The driving idea of the numerical work is to rephrase the determination of the updated material state in the form of the minimization of a power functional so that the evolutions in the microstructure and the density of defects recover meaning in terms of energy minimization. Moreover, in view of a direct comparison with the experimental results, the model should be flexible enough to adapt to the different conditions that can be found during a ferrite-to-austenite-to-ferrite transformation cycle.

The first chapter will be an introduction to the properties of iron. It will describe the elementary mechanisms that drive its response when it is submitted to temperature variations and it will introduce some basic tools that can be used to characterize the ferrite-to-austenite and the austenite-to-ferrite transformations.

The second chapter will enumerate the challenges that arise when dealing with the characterization of transformations in temperature. The design of the experimental equipment will be detailed and will be shown to tackle these issues. A proof of its viability will be brought by a preliminary test on an industrial Armco iron sample.

The third chapter will present further usage of this experimental equipment. Tested samples will have a higher purity and coarser microstructures. This will permit a finer characterization of the transformation process. A study on the influence of an initial plastic deformation on the ferrite-to-austenite transformation will also be presented.

In the fourth chapter, a variational model will be constructed at a material point level. Its goal is to compensate the flaws of the experimental procedure. The medium will be described as a Generalized Standard Material, which will permit an efficient coupling between thermal, mechanical and transformational contributions. Two distinct derivations will be proposed: a strain-based approach for the description of displacive transformations and a free energy-based approach for the description of diffusive transformations.

In the fifth chapter, this material point model will be embedded in an in-house finite element code to conduct computations on microstructures extracted from experimental measurements. The ability of the model to reproduce the formation of a new phase and to capture the strain field evolution during the ferrite-to-austenite transformation will be evaluated.

1

Introduction Microstructure evolutions in pure iron

Iron has the particularity to exhibit two stable allotropic structures below its melting temperature, which makes it an inexhaustible subject of study. A wide variety of microstructures and properties can be obtained for iron-based alloys as a result of the intricacy between the occurrence of the allotropic phase changes, the chemical composition of the alloy and the thermomechanical history of the material. This work deals with pure iron so that only the coupling between the mechanical state of the material and the changes in its microstructure can be studied.

Like most metals, iron is generally encountered in a polycrystalline form. It is formed by a cluster of crystalline arrangements that are called grains. Their main particularity is that they contain a lot of geometrical defects (mainly dislocations and grain boundaries) that are responsible for the mechanical response of the material and that condition the possible microstructure changes. Some examples on the importance of defects can be found in the restoration mechanisms that iron undergoes when heated. Moreover, the allotropic transformations are only another illustration of the microstructure-defects interaction. However, what makes their study more complex is that different transformation mechanisms may be at stake depending on the loading conditions. These mechanisms require adequate descriptions in terms of microstructural phenomena and kinetics.

Both these aspects are influenced by the mechanical state of the material, which can clearly be stated at the macroscopic scale. Some theoretical explanations were proposed in the literature for this influence. Halfway between the two, there is a lack of experimental evidence of the coupling between mechanics and transformation at the scale of the grains. The core motivation for the present work is to provide tools for the characterization of the allotropic transformations in iron at the mesoscale.

1.1 Pure iron

1.1.1 General properties

Iron is in mass the most abundant transition metal on Earth. No wonder that Humankind used it for tools production as early as 3000BC. One would generally refer to the iron-carbon phase diagram to get a first glance at iron alloys properties. It is displayed in Figure 1.1.



Figure 1.1: Iron-carbon phase diagram [1].

In the frame of this study, only the left-hand part of the diagram is of interest. Iron melts at 1534 °C. Before that point, it undergoes two allotropic transformations: α -iron is stable at room temperature, γ -iron at intermediate temperatures and δ -iron at high temperatures. One point of interest that does not show in the diagram is the Curie point, at 760°C. Some thermal and electrical properties of the material drop at that point, for instance heat capacity [2].

1.1.2 Crystals

 α -Iron, also called ferrite, has a BCC (Body-Centered Cubic) structure. At 910 °C, an FCC (Face-Centered Cubic) structure called austenite, becomes more stable. Both structures are shown in Figure 1.2. At the transformation temperature, the lattice parameters of these lattices were reported to be 0.291 nm for ferrite and 0.365 nm for austenite [3, 4]. As a matter of fact, δ -Iron also has a BCC structure.

Whether the atomic structure is BCC or FCC, the crystalline arrangement is formed by the periodic repetition in space of the lattices. However, a lattice is not enough to fully describe a crystal. It can have various orientations with respect to the lab frame. The orientation is classically defined by the rotation that sends the axes of the lab frame onto the axes of the crystal. Several ways are used to represent such a rotation: rotation matrices, axis/angle pairs, quaternions, Euler angles, Rodrigues vectors [5].

INTRODUCTION



Figure 1.2: Crystal lattices geometries.

In general, Iron is encountered in polycrystalline form, which means that several crystals coexist inside a sample. Each of them is called a grain. It may have the same composition as the surrounding grains but it must have a different orientation. The atomic arrangement in a polycrystal is far from being perfect. At the level of the grain, it is frequent that some atoms or groups of atoms are missing in the arrangement. Besides, since grains have different orientations, there cannot be a perfect continuity in the atoms disposition, which again creates gaps.

1.1.3 Defects in the crystalline structure

Dislocations

The most relevant defects to consider in the frame of this study are dislocations and grain boundaries. Dislocations are categorized as line defects because they correspond to a situation where a group of atoms is missing inside an atomic plane. To be more specific, dislocations can be of two types: edge dislocations result from an extra half atomic plane being present in the crystal whereas screw dislocations correspond to a global shearing of the crystal.

Figure 1.3 shows the deformation they induce in a crystal structure. Dislocations cause elastic straining of the material by distorting the atomic network. The distortion they induce is characterized by a vector called the Burgers vector. It is constructed by considering a loop that joins fully coordinated points (*i.e.* points pertaining to the ideal arrangement). The difference between the closed loop that would be obtained in a perfect crystal and the open loop that results from the presence of the dislocation is the Burgers vector. In the general case, what is called a dislocation is a combination of edge and screw segments.



Figure 1.3: The two types of dislocations [6].

Dislocations are abundant in the crystal. Plastic deformation is actually due to the irreversible movement of dislocations. They move on specific gliding planes that are the most packed planes of the crystal structure. They stay on a normal basis in the same gliding plane. This movement is called dislocation glide. Under certain circumstances, they may change their gliding plane:

- climb is the mechanism by which an edge dislocation bypasses a punctual obstacle by moving in a direction normal to its gliding plane. This movement is allowed by the displacement of vacancies;
- cross-slip consists for a screw dislocation in deviating to a plane that intersects its original gliding plane.

Grain boundaries

The interface between two grains is called a grain boundary. It is defined thanks to five degrees of freedom (dofs): three macroscopic dofs that describe the rotation that brings one grain orientation onto the other, and two dofs that relate the position of the grain boundary plane with respect to one of the two crystals. Depending on whether the angle associated with the rotation between the two grains is greater or less than 15°, the boundary is said to be a High Angle Grain Boundary (HAGB) or a Low Angle Grain Boundary (LAGB) respectively [7].

Grain boundaries can be classified into three categories depending on the relative orientations of the two grains:

- coherent interfaces relate to the situation where there is continuity between some gliding
 planes of the two grains. In other words, each atom of the first grain located at the
 boundary connects with one atom of the second grain. A dislocation arriving at the
 interface could then cross the interface staying in the same gliding plane;
- the interface is said to be semi-coherent if dislocation climb is necessary for a dislocation arriving at the interface to cross the interface. An atom of the first grain located at the boundary may connect with an atom of the second grain but it is not systematic. In between, edge dislocations relax the misfit strain;
- when none of the two other situations is encountered (no continuity and no possible climb), the interface is said to be incoherent. Dislocations would then tend to pile up at the interface.

The three types of interfaces are schematized in Figure 1.4. Again, it is rare that an interface is only coherent or only incoherent. Rather, when the boundary is long enough, it consists in a succession of coherent, semi-coherent and incoherent parts.



Figure 1.4: Classification of interfaces according to their level of coherency.

1.2 Ferrite grains evolution

An ideal equilibrium is conceptually attained in a material when there are no dislocations nor grain boundaries. Dislocations introduce local straining that the material wants to reduce or eliminate. Grain boundaries appear when it is not possible to maintain a continuity in the atomic structure and minimizing interfacial energy inevitably leads to the reduction of the number of grain boundaries. The purpose of this part is to explain how iron reduces the influence of these defects upon heating in the frame of a process that is called restoration. Restoration encompasses grain growth, recovery and recrystallization. It can be either dynamic if an external loading is applied on the material or static. Emphasis will be put on static processes.

1.2.1 Recovery

Heating increases the mobility of dislocations. They move in a way that reduces the total strain energy they are responsible for. Recovery proceeds by dislocation annihilation or dislocation rearrangement into configurations that are energetically more favourable [8].

Annihilation refers to the self-suppression of dislocations of opposite signs. The compression induced by one dislocation is balanced by the tension induced by the other, and vice versa. When annihilation is not possible, *i.e.* when dislocations are of the same sign, a rearrangement of the dislocations takes place. Through gliding and climbing, dislocations concentrate on the edges of array-like substructures, as shown in Figure 1.5. This reorganization into sub-cells lowers the overall strain energy induced by dislocations.



Figure 1.5: Dislocations reorganisation during recovery.

1.2.2 Primary recrystallization

Recrystallization occurs when the energy stored inside the grains is so high that a new dislocation-free phase is encouraged to nucleate and grow. Nucleation of the new phase requires the presence of HAGBs. In iron, since ferrite has a high-stacking fault energy, recovery mechanisms are predominant. As a consequence, this mechanism is most often limited to situations where the material has undergone high deformation [9]. In materials with high stacking fault energy, nucleation of dislocation-free grains occurs by two main mechanisms: either the progressive movement of dislocations forming the walls of a sub cell, which leads to the coalescence of two subgrains that constitue a nucleus, or the bulging of a grain boundary to form an area free of defects [10].

1.2.3 Grain growth

Either before or after recrystallization, the grains structure tends to coarsen. The driving force for grain growth is the reduction in the energy stored at grain boundaries: large grains have boundaries with a higher volume surface and are less energetic. Grains boundaries generally move towards their center of curvature to reduce surface tension [11].

Two specific kinds of grain growth deserve to be highlighted: secondary recrystallization and strain annealing. Both are reported as abnormal grain growth mechanisms since they lead to the growth of some grains at the expense of the others.

Secondary recrystallization

Secondary recrystallization shares common features with primary recrystallization in terms of microstructural mechanisms and kinetics. It occurs when normal grain growth is no longer possible. The impingement of grains, causing the end of a grain growth phase, may be caused by:

- the presence of impurities in the material;
- surface effects. This is called "sheet-thickness" effect: since the surface is flat, grain that intersect it have a larger radius of curvature than they would normally have [11].
- the texture of the material: grains with similar orientations cannot move with respect to each other.

The grains that grow are particular grains of the initial structure that encountered favourable conditions for growth with respect to those impingements. In other words, they do not result from a nucleation process because they were already present in the microstructure. One possible explanation for the abnormal growth of large grains lies in the fact that at triple points, grains maintain a curvature as close as possible to 120° so as to be at equilibrium. In the presence of large grains that intersect many others, this equilibrium is not attained everywhere. Henceforth, the boundary of the large grains is curved, which fosters their growth at the expense of the other grains in the microstructure. This mechanism stops once the coarsening of the grain is blocked by an obstacle [9].

Strain annealing

Strain annealing is a specific case of secondary recrystallisation. It is a mechanism of grain growth that occurs when the deformation of the material is not enough to permit primary recrystallization. In this case, grain growth is impeded by a polygonized dislocation structure [12].

The energy associated with cold working, even to small extents, is superior to what is stored at grain boundaries. A mechanism accounting for strain annealing is that each grain rearrange its dislocation structure at a different rate. Grains that reach a state with few dislocations first are allowed to grow inside the material. Strain annealing thus occurs only after a certain strain threshold is reached (when there is enough heterogeneity in the deformation to accommodate). After that threshold, the more the material is deformed, the more grains there are that act as initial sites for abnormal growth, which means that the obtained grains are smaller. Figure 1.6

displays a typical strain annealing recrystallization curve, that is to say the final grain size as a function of the strain applied to the sample and of the annealing temperature. As can be seen in Figure 1.6, there exists a strain at which the final grain size. It is called critical strain.



Figure 1.6: Typical curve for the grain size at the end of an annealing treatment as a function of annealing temperature and cold deformation [13].

1.3 Allotropic transformation

1.3.1 Diffusive vs displacive mechanisms

If one keeps heating an iron sample, whatever its state of deformation, it reaches a temperature at which the phase transformation from ferrite to austenite occurs. Two kinds of transformation are classically put to fore [14]:

- diffusive transformations take place when an uncoordinated movement of atoms is responsible for the change in crystal structure. Their main feature is the constitution of an interface that progresses in the material. They are also referred to as reconstructive mechanisms because atoms move across the interface to form a new microstructure;
- displacive transformations are characterized by the coordinated movement of several atoms. Atoms move together from an initial configuration to a final configuration. As a consequence, displacive transformations are frequently associated with a macroscopic strain. More precisely, the deformation caused by the growth phase of a displacive transformation can be classified as an invariant-plane strain deformation [15].

It is important to remark that although it may seem a bit odd, there is self-diffusion of atoms in the case of the diffusive transformation of a pure element [16]. Atoms have to move along the interface to permit a change in lattice structure without the macroscopic strain that the same lattice change would induce for a displacive transformation.

For the ferrite-to-austenite transformation, first results reported that raising the heating rate does not modify the temperature at which the transformation takes place [17]. However, Langner et al. [18] performed rapid heating (up to 300,000 $K.s^{-1}$) tests and observed that the

transformation temperature increased almost linearly with the heating rate. In any case the time-temperature response of the material exhibited a thermal arrest, which indicated that the transformation occurs by a diffusive mechanism. It can then be considered that the ferrite-to-austenite transformation is always diffusive. That said, the ferrite-to-austenite transformation is quite fast. Its speed is in the order of 0.016 $m.s^{-1}$ for a 200 μm grain size (*i.e.* a grain transforms in 1/80 s) [19].

On the other side, cooling austenite at different rates has a noticeable influence on the temperature at which the austenite-to-ferrite transformation occurs, as highlighted in Figure 1.7 for a hypoeutectoid steel. A similar behaviour is also reported in [20]. Depending on the cooling rate, the austenite-to-ferrite transformation may then start at a different temperature. Empirical formulae are usually proposed for the transformation temperature as a function of the cooling rate and of the iron-alloy composition. By deriving such a formula, Liu et al. [21] established that displacive mechanisms may occur from temperatures as high as 795 °C, which is in relatively good agreement with the experimental value of 750 °C.



Figure 1.7: Diversity of microstructures for a hypoeutectoid steel that can be obtained depending on cooling conditions [21]

1.3.2 Microstructures taxonomy

The variety of possible ferrite microstructures reflects the diversity of the transformation mechanisms. Microstructures were classified in order to facilitate their description. A notable work in this domain was made by Dubé et al. [22]. In the case of pure iron, this classification can be reduced to two possible microstructures [16]:

- an equiaxed structure with low-angle grain boundaries between grains. It stems either from allotriomorphic ferrite, which nucleated at austenite grain boundaries, or idiomorphic ferrite that nucleated intragranularly;
- a Widmanstätten-like ferrite which has the shape of thin wedges. When additional elements are present in the material, austenite may further decompose into the acicular structures like bainite or martensite that can be seen in Figure 1.7. However, as reminded for instance in [21], in the case of pure iron, no visual distinction can be made between microstructures obtained at high cooling rates.

Bain (B)	$\{001\}_a // \{001\}_f$	$<-110>_a//<-111>_f$
Kurdjumov-Sachs (KS)	$\{111\}_a // \{011\}_f$	<101> _a //<111> _f
Nishiyama-Wasserman (NW)	$\{111\}_a // \{011\}_f$	<112> _a //<011> _f
Pitsch (P)	$\{010\}_a // \{101\}_f$	<101> _a //<111> _f

Table 1.1: Orientation relationships for the austenite-to-ferrite transformation.

1.3.3 Orientation relationship

The transformation from austenite to ferrite can be described in a first approximation by the Bain mechanism, which is represented in Figure 1.8. It can be decomposed into a dilatation along two axes (the [110] and [-110] directions of the FCC structure) and a compression along the third axis (the [001] axis of the FCC structure).

It comes without saying that important shearing will result from such a mechanism in the case of a displacive transformation. As pointed out above, for a diffusive transformation, even if the lattices keep the relative orientations given by Bain mechanism, diffusion of atoms along the interface erases this shearing.



Figure 1.8: Bain mechanism.

The Bain mechanism is never observed in practice. One reason for that is notably that it does not constitute an invariant plane transformation. Rather, additional lattice vectors rotations intervene in the transformation process to permit a better correspondence between atomic directions of the lattices. When a ferrite grain emerges from a previous austenite grain, an Orientation Relationship (OR) between the parent and product phases usually remains. Several ORs, listed in Table 1.1, were suggested in the case of the austenite-to-ferrite transformation. Experimental ORs generally approximate the Kurdjumov-Sachs (KS) or Nishiyama-Wasserman (NW) relationships. Both ORs are within 11° of the Bain OR.

1.3.4 Mechanisms of the diffusive transformation

Chemical driving force - DSC

The transformation from ferrite to austenite is caused by the necessity to reach a more stable configuration. It is then associated with a release in energy. Tajima and Umeyama [23] measured the latent heat for the ferrite-to-austenite transformation to be $16kJ.kg^{-1}$. The latent heat for the austenite-to-ferrite transformation is $19kJ.kg^{-1}$.

Nucleation and growth

Solid-state nucleation for the reconstructive transformation According to the classical nucleation theory, a potential nucleus forms by a random movement of atoms inside the crystalline structure. It is necessary that the volume energy of the nucleus compensates the interfacial energy for this nucleus to be stable and grow. The energy barrier to be crossed by the nucleus reads:

$$\Delta G = -V * \left(\Delta G_{\alpha - \gamma} - \Delta G_{misfit} \right) + A * G_{interface} - G_{defects}. \tag{1.1}$$

V is the volume of the forming grain and A its area. The driving force $V * \Delta G_{\alpha-\gamma}$ and the additional driving force due to the presence of defects that reduce the coherency of the lattice structure $G_{defects}$ favour the growth of a new phase whereas the energy required for the accommodation of the misfit between the two phases $V * \Delta G_{misfit}$ and the energy for the constitution of an interface $A * G_{interface}$ tend to hamper it.

Militzer et al. [24] showed in the case of the austenite-to-ferrite transformation that the nucleation sites that are selected depend on the cooling rate. At low cooling rates, nucleation occurs mainly at triple junctions. At higher cooling rates, nucleation is also observed at other places such as grain faces. As stated above, interfaces are the junction of coherent, semi-coherent and incoherent parts. Several models have been proposed for the shape of a nucleus nucleating at a grain boundary. The most popular ones are:

- the original model of a spherical cap by Clemm and Fisher [25], which supposes that a grain appears at the interface with two incoherent boundaries with respect to the parent grains;
- Enomoto et al. [26] proposed a model where the boundaries parallel to the interface with the parent grains are coherent. This model was called the pillbox model;
- the intermediate configuration is that of a pillbox with a spherical cap [7]. The nucleus has a coherent interface with one of the parent grains and an incoherent interface with the other.

These three configurations are represented in Figure 1.9.





(b) Pillbox model



(c) Pillbox with a spherical cap

Figure 1.9: Three different models for the shape of the nucleus. In red: incoherent or semicoherent interfaces, in green: coherent interfaces.

The mechanism that accounts for most nucleation events inside an austenitic microstructure is the one of a pillbox nucleus with a spherical cap [7]. The basic idea is that an orientation relationship exists between the newly formed ferrite grain and one of the adjacent austenite grains, thus resulting in a coherent interface. The other interfaces are incoherent and more susceptible to permit grain growth. Indeed, the incoherent boundaries grow faster than those with an orientation relationship [27].

INTRODUCTION

Growth In the case of pure iron, growth is controlled by the uncorrelated motion of atoms near the austenite/ferrite interface. A simple expression of the velocity of the interface between ferrite and austenite can be taken from [16]:

$$v = \frac{\delta_b f^*}{R} \exp\left(\frac{-\Delta G^*}{RT}\right) \left(1 - \exp\left(\frac{-\Delta G_{\alpha\gamma}}{RT}\right)\right),\tag{1.2}$$

where ΔG^* is the activation energy for the transformation, $\Delta G_{\alpha\gamma}$ is the free energy associated with the transformation, δ_b is the thickness of the interface and f^* is related to the frequency of jumps across the interface.

Finally, the mobility of the interface is commonly defined as the quantity M such that:

$$v = M * \Delta g, \tag{1.3}$$

where v is the speed of the interface and Δg the driving force for the movement of the interface.

1.3.5 Kinetics

Diffusive transformation

We focus on non isothermal conditions. Upon continuous cooling or heating, the overall kinetics of the diffusive austenite-to-ferrite or ferrite-to-austenite transformations are fairly well described by formulations inspired by the Kolmogorov-Johnson-Mehl-Avrami (KJMA) model. All particles are first supposed to grow without being impinged by others. This allows calculating an extended transformed volume fraction x_e that is corrected in order to get the real volume fraction f:

$$\frac{df}{dx_e} = 1 - f. \tag{1.4}$$

Yet this formula is established under the assumption that there is a random nucleation in the sample. As mentioned before, grains edges and boundaries are preferential nucleation sites. A correction is then made to equation 1.4:

$$\frac{df}{dx_e} = 1 - f^{\zeta},\tag{1.5}$$

where ζ is an impingement parameter.

The expression of the transformed volume fraction is derived in terms of the number of particles nucleated at all times of the experiment and of the evolution of their volume during the growth phase [28]:

$$x_e = \int_{T_0}^{T(t)} \dot{N}(T(\tau)) Y(T(\tau), T(t)) \frac{1}{\phi} dT,$$
(1.6)

where ϕ is the heating or cooling rate. The volume of a particle nucleated at time τ is expressed at time t as:

$$Y = \frac{4}{3}\pi \left(\int_{T(\tau)}^{T(t)} \frac{v}{\phi} dT \right)^3.$$
(1.7)

v is the velocity of the austenite/ferrite or ferrite/austenite interface, given by equation 1.2. This equation underlines that the evolution of the volume of the formed grains is highly dependent

on temperature, which is illustrated for instance by ultrafast heating experiments in which the appearance of small austenite grains is favoured [29, 30].

Either classical nucleation theory or site saturation hypotheses are then made [31] to describe nucleation. In the latter case, the nucleation rate reads:

$$\dot{N}(T(\tau)) = N_0 \delta(\frac{T - T_0}{\phi}), \qquad (1.8)$$

where N_0 is a reference nucleation rate established at temperature T_0 . Note that the nucleation rate is rather hard to determine experimentally, which accounts for the attempts that were made to come up with a formulation where it does not appear. For instance, Rath and Pande [32] developed an approach based on the evolution of the interface area. A typical transformed fraction evolution profile obtained by solving equations (1.4) and (1.6) is presented in Table 1.2.

Displacive transformations

If the transformation from austenite to ferrite is diffusive, it follows the same kinetic laws as the one exposed in section 1.3.5. However, it is more common to use the Koistinen-Marburger model when the transformation is displacive. This model reads:

$$f = 1 - e^{-c(M_s - T)},\tag{1.9}$$

where c is a constant. M_s is the transformation at which the displacive austenite-to-ferrite transformation begins. It can be evaluated using the formulae discussed in section 1.3.1. Again, a typical transformed fraction evolution given by the Koistinen-Marburger model during cooling is shown in Table 1.2.

1.3.6 Summary

Distinction is not clear between diffusive and displacive. For instance, it is frequent to encounter microstructures where phases resulting from both diffusive and displacive mechanisms coexist. However, it may be convenient in certain cases to consider a binary demarcation between the two so as to isolate some elementary mechanisms that could be of interest. The main characteristics of each type of transformation are presented in Table 1.2.

1.4 Coupling between mechanics and transformation

1.4.1 Mechanical effect of the transformation

The occurrence of allotropic transformations at the macroscale can be detected by performing dilatation tests on pure iron or ultralow carbon steel samples [33]. The temperaturelongitudinal strain response of the material is quite linear everywhere except around 910°C where longitudinal strain tends to drop in the case of the ferrite-to-austenite transformation or to rise in the case of the austenite-to-ferrite transformation. This trend highlights the role of allotropic transformations on the mechanical state of the material.

What accounts for the deformation induced by the allotropic transformation is the difference in volume between ferrite and austenite. The volume change can be calculated by remarking in

In	TRODUCTION			
		Diffusive transformation	Displacive transformation	
	Self-diffusion during growth	Yes	No	
	Reconstructive	Yes	No	
	Large shearing	No	Yes	
	Microscopic marker	Interface between the phases	Invariant line	
	Thermal arrest	Yes	No	
	Relative speed	Slow	Fast	
	Kinetics model	KJMA	Koistinen-Marbuger	
	Typical kinetic response	Temperature (during heating)	Temperature (during cooling)	

Table 1.2: Distinction between diffusive and displacive transformation mechanisms.

Figure 1.8 that one lattice of austenite forms from two lattices of ferrite due to the difference in compacity between the BCC and the FCC structures. The volume variation then reads:

$$\delta V = \frac{2a_{\alpha}^3 - a_{\gamma}^3}{2a_{\alpha}^3},\tag{1.10}$$

where a_{α} and a_{γ} are the lattice parameters of ferrite and austenite respectively. As iron transforms from a BCC to an FCC structure, the volume misfit to accomodate is then of 1.3%.

A first approximation of the energy associated with the transformation is proposed in [34]:

$$W = 2 * (\mu_{\alpha} + \mu_{\gamma}) * \delta V^2 \tag{1.11}$$

where μ_i are the shear moduli of the two phases. This expression suggests an elastic accommodation of the transformation by the surrounding medium whose properties are an average of the properties of the two phases. This description is rather macroscopic in the sense that it disregards the local repartition of phases and the microplasticity that accommodates the volume misfit. One of the objectives of this work is to gain insight on these phenomena.

1.4.2 Influence of prior deformation on the allotropic transformation

Influence of prior deformation on the austenite-to-ferrite transformation

The transformation is fostered by loadings that are able to interact with the population of local defects. By contrast, it is for instance inhibited by a hydrostatic pressure. A prior austenite deformation mainly affects the nucleation phase of the process by increasing the nuclei density. This nucleation is ruled by the locally stored defect energy. In the framework of classical nucleation theory, this means that the presence of defects increases the value of the $G_{defects}$ term in equation (1.1).

Austenite tends to become less stable after deformation. This explains why it may be employed to reduce the size of the final ferrite grains. Deformation has three main effects on the grain boundaries according to Sietsma [35]:

- increased dislocation density and local creation of regions with high deformation;
- anisotropic deformation of the grain and augmentation of the total boundary area of the crystalline structure;
- accumulation of dislocations near boundaries. The local mismatch generates internal stresses and increases the probability to encounter defects in these zones.

Due to all these aspects, the formation of a nucleus is more likely to lower the total energy of the area. Consequently, nuclei form more easily. Growth might also be enhanced due to the increased driving force for transformation linked to the defect energy. Experimentally, the number of ferrite nuclei has been found to triple due to the effect of a slight plastic deformation [7]. Landheer et al. suggest that deformation generates more LAGBs that act as potential pillbox nucleation sites.

Concerning growth phenomena, ferrite grain size evolution is explained by both coalescence and normal grain growth. The final grain size is a result of the competition between the nucleation rate and the ferrite grain coarsening [36]. But normal growth mainly affects HAGBs while coalescence concerns LAGBs. Consequently, prior deformation of austenite favors coarsening phenomena taking place after the transformation. This is consistent with the fact that the effect of deformation on growth rate seems to be significant only at very low undercoolings [37]. As stated in [35], the equation for the mobility of the interface in the presence of defects reads:

$$v = M * (\Delta G + g_d) \tag{1.12}$$

where g_d is the dislocation defect energy, approximated by $g_d = \frac{1}{2}\mu b^2 \rho$ (with ρ the local dislocation density).

Influence of prior deformation on the ferrite-to-austenite transformation

There are less studies on the influence of a prior deformation on the ferrite-to-austenite transformation in the literature. Indeed, although iron microstructure is influenced by the whole transformation cycle it went through, it seems natural to study only the last step, that is the austenite-to-ferrite transformation.

When there is prior deformation of ferrite, a competition takes place during heating between recrystallization of ferrite and transformation into austenite [38]. As recalled in [39], a uniaxial compressive loading imposed on the sample increases the driving force needed for the ferrite-to-austenite transformation and delays the onset of transformation. The opposite trend is observed in the case of the austenite-to-ferrite transformation. This can be interpreted easily by remembering that in the Bain mechanism for the ferrite-to-austenite transformation, two axes work in compression and only one in traction.

1.5 Conclusion

1.5.1 Remaining challenges on the mechanics-transformation coupling

Iron transforms from a BCC ferrite structure to an FCC austenite structure when the temperature is high enough. The study of allotropic transformations has been shown to lie in the broader frame of the creation and attenuation of line or surface defects inside the microstructure. These defects induce straining that the material try to reduce. Henceforth, the mechanical state and the microstructural evolutions of iron have reciprocal influences on each other. They are not fully understood yet. The presence of defects such as dislocations is known to increase misfits in the material and to provide an additional driving force for the transformation but it is not known with exactitude how microstructure reconstructs around these areas. Conversely, allotropic transformations induce microplasticity in the local area around the transforming zone but it is hard to determine how it develops inside the material.

More generally, there seems to be a gap between two scales:

- at the macroscopic scale, *i.e.* at the scale of a polycrystalline sample, the interaction between allotropic transformations and the mechanical state of iron is well understood in terms of overall kinetics and can be characterized by dilatation tests. Nonetheless, it is obvious that these behaviours are just an averaged response of more complex and more local mechanisms taking place inside the grains or at grain boundaries.
- at the microscopic scale, *i.e.* at the scale of the atomic arrangement, several theoretical models have been proposed to describe the transformation mechanisms. They may even account for the influence of plastic straining on the nucleation and growth of a new phase. However, it is extremely difficult to conduct experiments at such a fine scale and very few experimental data is available on the interactions between the population of local defects in the material and microstructure evolution phenomena.

1.5.2 Motivation for the present work

In this work, we will be considering an intermediate scale that we will call mesoscopic scale. This term refers to representative volumes whose characteristic lengths are in the same order of magnitude as the size of the grains. This choice implies sacrificing the possibility to observe directly atomic movements. For instance, only dislocation densities can be studied at the mesoscopic scale and not single dislocations. That said, the strain fields that may be captured at the scale of the grains are the direct consequence of the activity of dislocations. Moreover, another averaging of the obtained experimental data should allow retrieving classical laws established at the macroscopic scale. The idea is then to fill the gap between the microscopic and macroscopic scales.

Explaining the local transformational behaviour of an iron sample could be extremely valuable for instance in the automotive industry. Indeed, the combination of high temperature loading and strong deformation leads to failure mechanisms that are not well understood. The purpose of the present work is to bring further insight with respect to these questions.

To the knowledge of the author, kinematic fields during allotropic transformations have never been observed at the scale of the grains. The main objective of this thesis work is to come up with an experimental device that will allow the monitoring of the formation of a new phase in the material through the study of the relevant kinematic fields evolution. The coupling between transformation and plastic activity will be studied by direct post-processing of the experimental data and polycrystalline simulations on realistic geometries.

Bibliography

[1] H. Bhadeshia and R. Honeycombe. *Steel: Microstructure and properties (4th edition)*. Elsevier, 2017. 2

- [2] J.B. Austin. Heat capacity of iron: a review. *Industrial and Engineering Chemistry*, 24:1225 1235, 1932. 2
- [3] Z.S. Basinski, W. Hume-Rothery, and A.L. Sutton. The lattice expansion of iron. *Proceedings of the Royal Society A*, 1955. 2
- [4] M. Onink, C.M. Brakman, F.D. Tichelaar, E.J. Mittemeijer, S. van der Zwaag, J.H. Root, and N.B. Konyer. The lattice parameters of austenite and ferrite in Fe-C alloys as functions of carbon concentration and temperature. *Scripta Metallurgica et Materialia*, 29, 1993. 2
- [5] V. Randle and O. Engler. *Texture Analysis: Macrotexture, microtexture & Orientation mapping.* Taylor and Francis, 2000. 2
- [6] J.P. Hirth and J. Lothe. Theory of dislocations. Krieger Publishing Company, 1982. 3
- [7] H. Landheer, S.E. Offerman, R.H. Petrov, and L.A.I. Kestens. The role of crystal misorientations during solid-state nucleation of ferrite in austenite. *Acta Materialia*, 57(5):1486–1496, March 2009. 4, 10, 14
- [8] F.J. Humphreys and M. Hatherly. *Recrystallization and Related Annealing Phenomena*. Elsevier, 2004. 5
- [9] A. Schmickl. *Warm deformation of low carbon steels*. University of Wollongong (thesis), 1997. 5, 6
- [10] H.J. McQueen and J.J. Jonas. Recovery and Recrystallization during High Temperature Deformation. In *Treatise on Materials Science & Technology*, volume 6, pages 393–493. Elsevier, 1975. 6
- [11] P Felthams. Grain growth in metals. page 9. 6
- [12] C. Antonione, G. Della Gatta, G. Riontino, and G. Venturello. Grain growth and secondary recrystallization in iron. *Journal of Materials Science*, 8(1):1–10, January 1973.
 6
- [13] R. Cahn and P. Haasen. *Physical metallurgy (4th edition)*, pages 2399 2500. Elsevier, 1996. 7
- [14] W.A. Soffa and D.E. Laughlin. *Physical Metallurgy*, chapter Diffusional phase transformations in the Solid State. Elsevier, 2014. 7
- [15] C.M. Wayman and H.K.D.H. Bhadeshia. Phase Transformations: Nondiffusive. In *Physical Metallurgy*, pages 1507–1554. Elsevier, 1996. 7
- [16] H.K.D.H. Bhadeshia. Diffusional formation of ferrite in iron and its alloys. Progress in Materials Science, 1985. 7, 8, 11
- [17] V. I. Savran, S. E. Offerman, and J. Sietsma. Austenite Nucleation and Growth Observed on the Level of Individual Grains by Three-Dimensional X-Ray Diffraction Microscopy. *Metallurgical and Materials Transactions A*, 41(3):583–591, March 2010. 7
- [18] Jeremy Langner and J.R. Cahoon. Increase in the Alpha to Gamma Transformation Temperature of Pure Iron upon Very Rapid Heating. *Metallurgical and Materials Transactions* A, 41(5):1276–1283, May 2010. 7
- [19] F G Caballero and C Capdevila. Kinetics and dilatometric behaviour of non-isothermal ferrite-to- austenite transformation. page 26. 8

- [20] E A Wilson. The alpha-gamma transformation in Iron. 12(11):8. 8
- [21] Cheng Liu, Zhenbo Zhao, Derek O. Northwood, and Yunxu Liu. A new empirical formula for the calculation of MS temperatures in pure iron and super-low carbon alloy steels. *Journal of Materials Processing Technology*, 113(1-3):556–562, June 2001. 8
- [22] C.A. Dubé, H.J. Aaronson, and R.F. Mehl. La formation de la ferrite proeutectoïde dans les aciers au carbone. *Revue de métallurgie*, 3:201 – 210, 1958. 8
- [23] Mamoru Tajima and Yoshio Umeyama. Latent heats of phase transformations in iron and steels. *High Temperatures-High Pressures*, 34(1):91–97, 2002. 9
- [24] M. Militzer, R. Pandi, and E. B. Hawbolt. Ferrite nucleation and growth during continuous cooling. *Metallurgical and Materials Transactions A*, 27(6):1547–1556, June 1996. 10
- [25] P.J. Clemm and J.C. Fisher. The influence of grain boundaries on the nucleation of secondary phases. Acta Metallurgica, 3:70 – 73, 1955. 10
- [26] M. Enomoto, W. F. Lange, and H. I. Aaronson. The kinetics of ferrite nucleation at austenite grain edges in Fe-C and Fe-C-X alloys. *Metallurgical Transactions A*, 17(8):1399– 1407, August 1986. 10
- [27] Khaled Al-Hajeri. The grain coarsening and subsequent transformation of iron. page 220. 10
- [28] Y.-H. Jiang, F. Liu, and S.-J. Song. An extended analytical model for solid-state phase transformation upon continuous heating and cooling processes: Application in γ/α transformation. *Acta Materialia*, 60(9):3815–3829, May 2012. 11
- [29] F.M. Castro Cerda, I. Sabirov, C. Goulas, J. Sietsma, A. Monsalve, and R.H. Petrov. Austenite formation in 0.2% C and 0.45% C steels under conventional and ultrafast heating. *Materials & Design*, 116:448–460, February 2017. 12
- [30] Dorien De Knijf, Maria J. Santofimia, Hui Shi, Vitaliy Bliznuk, Cecilia Föjer, Roumen Petrov, and Wei Xu. In situ austenite-martensite interface mobility study during annealing. Acta Materialia, 90:161–168, May 2015. 12
- [31] Y. Liu, P.R. China, F. Sommer, and E.J. Mittemeijer. Nature and kinetics of the massive austenite-ferrite phase transformations in steels. In *Phase Transformations in Steels*, pages 311–381. Elsevier, 2012. 12
- [32] B.B. Rath and C.S. Pande. An analytical description of overall phase transformation kinetics applied to the recrystallization of pure iron. *Acta Materialia*, 59(20):7538–7545, December 2011. 12
- [33] Yongchang Liu, Dongjiang Wang, Ferdinand Sommer, and Eric Jan Mittemeijer. Isothermal austenite–ferrite transformation of Fe–0.04at.% C alloy: Dilatometric measurement and kinetic analysis. Acta Materialia, 56(15):3833–3842, September 2008. 12
- [34] E.A. Wilson. Chemical driving force, shear moduli of ferrite and metastable austenite, and strain energy for $\gamma \alpha$ transformation in pure iron. *Materials Science and Technology*, 11:1110 1115, 1995. 13
- [35] J. Sietsma. Nucleation and growth during the austenite-to-ferrite phase transformation in steels after plastic deformation. In *Phase Transformations in Steels*, pages 505–526. Elsevier, 2012. 13, 14

- [36] R. Bengochea, B. López, I. Gutierrez, B. López, and I. Gutierrez. Microstructural evolution during the austenite-to-ferrite transformation from deformed austenite. *Metallurgical* and Materials Transactions A, 29(2):417–426, February 1998. 14
- [37] David N. Hanlon, Jilt Sietsma, and Sybrand van der Zwaag. The Effect of Plastic Deformation of Austenite on the Kinetics of Subsequent Ferrite Formation. *ISIJ International*, 41(9):1028–1036, 2001. 14
- [38] Pei Li, Jun Li, Qingge Meng, Wenbin Hu, and Dechao Xu. Effect of heating rate on ferrite recrystallization and austenite formation of cold-roll dual phase steel. *Journal of Alloys* and Compounds, 578:320–327, November 2013. 14
- [39] Y.C. Liu, F. Sommer, and E.J. Mittemeijer. Kinetics of austenitization under uniaxial compressive stress in Fe–2.96at.% Ni alloy. *Acta Materialia*, 58(3):753–763, February 2010. 14

2

Design of an experimental set-up for a mesoscopic study of allotropic transformations in iron

The experiments conducted in this work lie at the scale of a few grains. Consequently, one does not observe the movements of individual atoms but the mesoscopic consequences of these movements: formation and motion of new grain boundaries, modification of the local diffraction properties in the material, elasto plastic straining around the newly formed phase. This latter aspect is further studied because it appears as a good way to study the coupling between mechanics and transformation.

The observation of the ferrite-to-austenite and austenite-to-ferrite transformations requires to raise the temperature of a sample up to over 900 °C while capturing images of constant quality. A set-up is developed in-house to heat a sample by Joule effect and to take images of its surface. Precautions are taken to avoid any kind of image degradation. Digital Image Correlation (DIC) is employed to obtain the straining induced by the transformation from the images of the sample. The idea behind DIC is to track the motion of unambiguously identifiable patterns on the surface of the sample in order to reconstruct the displacement field.

Tests are performed on industrial Armco iron to assess the reliability of the experimental device. Although the grain size is not sufficient to qualify the results as mesoscopic, characteristic localizations of shear strain are detected when the ferrite-to-austenite transformation takes place.

2.1 Indicators of transformation occurrence

Since it is based on the local reorganization of atoms, the observation of allotropic phase changes at the mesoscale has to rely on indirect markers of its occurrence. Three kinds of markers can be proposed in light of the introductory section: the interface between the phases, the fraction of a given phase or the strain fields resulting from the transformation.

2.1.1 Boundary motion tracking

Confocal Laser Microscopy

The most straightforward approach is certainly to track the interface that is formed between parent and product phases. One possibility to achieve this is Laser Scanning Confocal Microscopy (LSCM). A set-up to perform LSCM is shown in Figure 2.1. A Laser Beam is sent on the surface of a sample on which it is reflected. The closer from the focal point, the more intense the signal is. LSCM allows capturing with precision the surface of the sample. Henceforth, surface irregularities such as the one formed by a grain boundary can be observed.



Figure 2.1: Set-up for LSCM observations [1].

Zhang and Komizo [2] used a Laser Scanning Confocal Microscope (LSCM) apparatus to observe the appearance of new grain boundaries between ferrite and austenite through modifications on the surface morphology of the samples. Lee et al. [3] studied the cooling of ultralow carbon samples. They noticed that the fraction of Widmanstätten ferrite formed increased with the cooling rate. Similarly, Niknafs and Dippenaar [4] reported that the morphology of austenite at the end of the $\delta - \gamma$ transformation process is dependent on the cooling rate.

High-temperature SEM

This kind of interface identification can be performed at even finer scales. Zijlstra et al. [5] resorted to a heating system inside a Scanning Electron Microscope (SEM), taking special care not to damage detectors. Electron Back Scattered Diffraction (EBSD) maps allow to distinguish between austenite and ferrite. The formation of austenite grains in a ferrite microstructure is highlighted in Figure 2.2. The observation of the interface between ferrite and austenite at different time steps leads to an estimation of boundary motion velocity.

2.1.2 Phase contents tracking

The idea behind these methods is to measure physical quantities that are influenced by the nature of the phases, which leads to a quantification of the phase fractions evolution. Laser Ultrasonics is based on the analysis of ultrasonic waves after they cross a sample. Attenuation



Figure 2.2: Apparition of austenite grains (in green) in a ferrite structure (in red) [5].

of the ultrasonic wave can be related with microscopic features, in particular with grain size. Maalekian et al. [6] captured the growth of austenite grains in a Ti-Nb steel using this technique.

X-Ray diffraction puts to good use the intensity of the diffraction patterns induced by the grains in the microstructure. This kind of measurement is performed at Synchrotron sources since the X-Ray beam has to be very energetic. The sample holder is rotated so that several grains come into conditions for diffraction. Diffraction spots are put in relation with the phase and particle volumes are calculated. This provides a fine quantification probe for nucleation and growth kinematics [7]. Savran et al. [8] measured the evolution of austenite fraction during the heating of hypoeutectoid steels.

Likewise it is possible to conduct neutron diffraction tests. Shi et al. [9] used a diffractometry equipment to highlight the influence of cold-rolling on the microstructural evolutions of a 22SiMn2TiB steel. An advantage of this method is that the lattice changes can be easily identified from the diffraction profiles [10].

2.1.3 Mechanical effect tracking: DIC

A number of experimental methods were developed so as to measure deformation in a given material. Traditional contacting methods, such as strain gauges and extensioneters, only allow doing measurements that could be seen as global in the sense of integrated and by opposition to local field measurements. This is not interesting in the case of this work where emphasis is put on observing strain fields during transformation. Besides, contacting techniques may damage and deform the sample at high temperatures.

Non contacting techniques include digital laser speckle correlation, laser speckle interferometry, Moiré interferometry, microextensometry or digital image correlation. This latter method was retained in the frame of this work due to its rather simple experimental set-up and the high resolution that can be attained. Moreover, although other techniques like laser speckle correlation were adapted to high temperatures [11], DIC seems to be the most suitable for these conditions, which was underlined in a wide variety of applications:

- Characterization of creep in Zirconium alloys [12];
- Investigation of fatigue crack growth at temperatures up to 650°C [13];
- Identification of asymmetric laws for aluminium titanate ceramics based on four-point bending tests in a furnace [14];
- Characterization of dynamic strain aging in Hastelloy X by observing the propagation of Portevin-Le Chatelier bands [15];
- Determination of the brittle to ductile transition temperature of carbon fiber reinforced
silicon carbides [16].

2.2 Digital Image Correlation

2.2.1 General idea behind DIC

The idea is to compute the displacement of a region of interest by tracking a pattern between two images. This principle is illustrated in Figure 2.3.



Figure 2.3: Principle behind the Digital Image Correlation algorithms.

The precision of the kinematic fields is related to the resolution of the images. It follows from this that the challenge for the study of allotropic phase changes is to combine highresolution and high-temperature requirements. The fact that DIC has never been applied to the study of allotropic phase changes may be attributed to the difficulty of taking accurate images of the surface of a sample at temperatures from room temperature to over 800°C continuously.

2.2.2 High-resolution high-temperature DIC

High-resolution applications

In the frame of this study, high-resolution refers to the possibility to capture in-grain phenomena. In other words, the microstructure has to be way coarser than the pixels. Either microstructures with large grains are used or equipments that have a very high resolution (for optical cameras).

Concerning the first point, a good example can be found in studies on ice recrystallisation, where grain sizes reach several millimetres. Chauve et al. [17] define an equivalent strain as:

$$\varepsilon_{eq} = \sqrt{\frac{2}{3}(\varepsilon_{xx}^2 + \varepsilon_{yy}^2 + 2\varepsilon_{xy}^2)},\tag{2.1}$$

where ε_{ij} are components of the Green-Lagrange tensor. They were able to follow the evolution of this quantity around a triple point junction and showed that its values could be related to the zones where recrystallization initiates.

Concerning the second point, DIC has been successfully applied to the characterization of plastic activity inside grains of polycrystalline materials. Di Gioacchino and Quinta da Fonseca [18] used gold remodelling to produce a speckle pattern on 304 stainless steel samples. They captured images using a SEM and were able to capture slip bands in grains with a typical

CHAPTER 2

size of 50 μm . Guery et al. [19] also used SEM images to study slip activity in polycrystals of 316L steel. The originality of their approach is that they performed DIC computations on a mesh that conformed with the underlying microstructure, which provided a direct comparison with crystal plasticity calculations.

High-temperature conditions

Conducting experiments at high temperature raises a number of challenges which will be listed below.

Oxidation The main challenge that arises when it comes to high-temperature conditions is oxidation. According to Chen and Yuen [20], the weight gain per unit area W_p is commonly obtained with:

$$\frac{dW_p}{dt} = \frac{k_p W_p}{2} \tag{2.2}$$

The evolution of the constant k_p is plotted in Figure 2.4. As can be seen from the figure, oxidation of iron is a very fast process. Besides, given the high susceptibility of iron to oxidation, it would require vacuum levels as low as 10^{-6} mbar to prevent oxidation of iron samples.



Figure 2.4: Evolution of the parabolic constant for oxidation rate with temperature [20].

Heat haze As pointed out in previous studies dealing with DIC at high temperatures [21], a heat haze effect, *i.e.* a distortion of the images due to local variations of the refractive index of the surrounding gaseous atmosphere, may occur. Lyons et al. [22] were among the first ones to carry out high-temperature DIC tests. They identified two sources of image distortion: the window of their furnace and air distortion near this window. They managed to reduce the effect of convective currents by using a fan near the furnace window. Novak et al. [23] used an air knife to minimize thermal disturbance due to heat haze. Besides, it can also be noted that algorithmic corrections to take into account thermal disturbance were suggested. Su et al. [24] calculated a coefficient to model this effect from the deviation of the average grayscale value of the speckle images.

Lightning It is also necessary to avoid the influence of gray body radiation from the sample. As the sample is heated, it radiates a light whose energy increases with the wavelength. More precisely, the intensity of the radiation is governed by Planck's law:

$$I(\lambda,T) = \frac{2hc^2}{\lambda^5} \frac{1}{e^{\frac{hc}{\lambda kT}} - 1}$$
(2.3)

where λ is the wavelength, c the speed of light, h is Planck's constant and k is Boltzmann's constant. Figure 2.5 shows the evolution of the energy radiated from the sample as a function of the wavelength. The higher the wavelength, the more detrimental radiation is to image quality.



Figure 2.5: Energy radiated at different temperatures as a function of the wavelength.

Speckle pattern Traditional correlation paintings cannot be used for high temperature experiments since they burn out around 250°C. Among other phenomena that may deteriorate the speckle pattern, reaction with the substrate material, cracking or plastic flow have to be carefully avoided. All these lead to either a loss of contrast or the impossibility to carry out image correlation. It is thus necessary to find a refractory paint so as to deposit a pattern that would not get altered by a raise in temperature. Usually, a mixture of ceramics compounds and adhesive is employed to create a high temperature painting. For instance, Cholewa et al. [25] used TiO paint to combine DIC and infrared thermography measurements, and Mao et al. [16] used ZrO2 paint.

2.2.3 Post-processing of the experimental data

FE-DIC

Once the conditions for the acquisition of high-resolution and high-temperature images are met, a proper algorithmic treatment of the obtained data is necessary to get the evolution of kinematic fields on the surface of a sample. FE-DIC computations are carried out using UFreckles software [26] in the frame of this thesis. The following paragraph describes the main steps to obtain the displacement field in the region of interest. Let us consider two images, denoted by f and g, f being the reference image. Both are described by their gray values at each pixel. It is supposed that g is the consequence of transforming points in f according to a displacement field u. Conservation of the optical flow is assumed:

$$f(\boldsymbol{x}) = g(\boldsymbol{x} + \boldsymbol{u}(\boldsymbol{x})). \tag{2.4}$$

At this point, two main locks prevent the determination of u: this scalar equation is not enough to compute the vector quantity u, and there may not be unique correspondence between points in f and g. To solve these issues, the optical flow equation is reformulated in weak form. Making additional use of a Taylor expansion, the correlation problem sums up to the minimization of the difference between these two quantities on the whole domain:

$$\min_{\boldsymbol{u}} \iint_{\Omega} ||\delta \boldsymbol{u}(\boldsymbol{x}) \cdot \boldsymbol{\nabla} g(\boldsymbol{x}) + g(\boldsymbol{x} + \boldsymbol{u}(\boldsymbol{x})) - f(\boldsymbol{x})||^2 d\boldsymbol{x}.$$
(2.5)

Non-pixel values are determined using a bi-cubic interpolation.

The displacement field is further discretized by constructing a mesh and using a finite element approximation of displacement. Q4 finite element shape functions are chosen in the same fashion as in [27]:

$$\boldsymbol{u}(\boldsymbol{x}) = \sum_{\alpha,n} a_{\alpha n} \Psi_n(\boldsymbol{x}) \boldsymbol{e}_{\alpha}, \qquad (2.6)$$

where a_{ij} are the discretisation parameters, Ψ_i the shape functions, and e_{α} the considered direction. The minimization problem (2.5) is non linear. Therefore it is solved iteratively. It can be shown that after discretization, the minimization problem sums up to a series of linear problems with the displacement increment as unknown.

DIC procedure then takes the following form:

- an initial displacement increment guess is made. However, a Taylor approximation is resorted to. Hence, displacement has to remain small. A coarse graining strategy is adopted. Calculations are performed on supergrids of 2x2, 4x4, 8x8 cells, etc, and the obtained displacements are used as starting points for the iterations on the finer grids.
- quantities of interest are computed for the current iteration. In particular, image gradients are obtained thanks to a finite difference scheme.
- if the increment of displacement is small enough, computations are stopped. Otherwise, the process is iterated until convergence.

Regularization

Since strain localizations are expected to appear, regularization is performed to avoid that strain becomes excessively large for an element, thus impeding convergence of the DIC algorithm. As explained in [28], an additional term is added to the functional to be minimized in the fashion of a Tikhonov regularization:

$$\min_{\boldsymbol{u}} \left(\iint_{\Omega} ||\delta \boldsymbol{u}(\boldsymbol{x}) \cdot \boldsymbol{\nabla} g(\boldsymbol{x}) + g(\boldsymbol{x} + \boldsymbol{u}(\boldsymbol{x})) - f(\boldsymbol{x})||^2 d\boldsymbol{x} + \omega(l_c) \iint_{\Omega} ||\nabla \boldsymbol{u}(\boldsymbol{x})||^2 d\boldsymbol{x} \right).$$
(2.7)

This contribution is weighted by a parameter $\omega(l_c)$. The regularization is shown to act as a low-pass filter whose cut-off wavelength l_c can be adjusted.

The linear problems to be solved can be expressed as finding the parameters $a_{\alpha n}$ such that

$$\sum_{n}\sum_{\alpha}a_{\alpha n}\iint_{elem}\left(\Psi_{n}(x)\Psi_{m}(x)\partial_{\alpha}g(x)\partial_{\beta}g(x)+\omega\partial_{\beta}\Psi_{m}(x)\partial_{\alpha}\Psi_{n}(x)\right)dx=$$
(2.8)

$$\iint_{elem} \left(f(x) - g(x) \right) \Psi_m(x) \partial_\beta g(x) dx \ \forall m = 1, \dots M,$$
 (2.9)

where the notation $\nabla h(x) \cdot e_{\alpha} = \partial_{\alpha} h(x)$ is adopted.

2.3 Design of the experimental set-up

The methodology developed in this chapter aims at testing in conditions as close as possible to industrial processes. For instance, to circumvent the delay involved by scanning the surface with a SEM, optical cameras are used. The precision of obtained results is bound to improve continuously given the progresses of optical systems [29].

2.3.1 Samples design

Geometry

The geometry of the tested sample is shown in Figure 2.6. Electrical resistance is inversely proportional to the section. The fillets aim at ensuring that temperature is maximal in the center of the sample. Besides, the central part is long enough for the temperature to establish a zone where it is homogeneous. During heating, the ferrite-to-austenite transformation should initiate in this zone. A "stationary" transformation behaviour, where heterogeneities in the mechanical state drive the response of the material, can be characterized in this zone. In the rest of the sample, a gradient of temperature is likely to develop. At a given time, the transition temperature for transformation is only crossed in a narrow slice of the sample. It can be expected that a transformation front will propagate towards the extremities of the sample, thus leading to the characterization of a "transient" transformation behaviour.



Figure 2.6: Drawing of the sample (dimensions in mm). The sample thickness is 1mm.

Spatial evolution of temperature

Steady state simulations were conducted to get an idea of the temperature repartition inside the samples during the tests. They were performed using Zorglib software, which is developed at GeM Institute. The following assumptions are made:

- the simulation uses a volumic source term obtained by assuming a uniform current density through the cross-section. The dissipated power per unit volume is expressed as $\rho \frac{I^2}{S^2}$ where I is the current that crosses the sample, ρ is the electrical resistivity of iron and S is the section of the sample;
- there is thermal conduction inside the sample;
- power is dissipated through gray-body radiation at all free surfaces of the sample. The energy flux associated with radiation is $\varepsilon \sigma \Theta^4$ where σ is Boltzmann's constant, ε is the emissivity of the material and Θ the temperature.

The material constants used in the computation are displayed in Table 2.1.

As a matter of example, let us consider heating an iron sample under a constant current of 100A. When the central part of the sample reaches 900°C, the temperature field has the profile

Dilatation coeff.	Specific heat capacity	Conductivity coeff.	Emissivity coeff.	Electrical resistivity
$10^{-5}K^{-1}$	$450 \ J.K^{-1}.kg^{-1}$	$80 W.m^{-1}.K^{-1}$	1.0	9.87 $10^{-8} kg.m^3.s^{-3}.A^{-2}$

Table 2.1: Material constants used in thermal computations.



Figure 2.7: Simulation of the temperature evolution in the sample during testing.

shown in Figure 2.7a. The evolution of temperature along the middle horizontal line (x axis) is plotted in Figure 2.7b. This plot confirms that there exists a zone in the center of the sample where the temperature is homogeneous and that a temperature gradient develops towards the outer extremities of the sample.

2.3.2 Principle of the experiment

A schematic view of the experimental set-up is presented in Figure 2.8. Its three main subsystems are:

- a heating system that raises the temperature of the sample by Joule effect;
- an optical system that takes images of the surface of the sample;
- a temperature measurement system that takes point measurements of the temperature during the tests.

These subsystems are detailed below.



Figure 2.8: Schematic view of the experimental set-up.

2.3.3 Heating system

Joule heating apparatus

Joule heating is preferred over other heating methods owing to its volumetric character and the easy access to the sample it provides. A TDK-Lambda Genesys 5kW power supply with 500A maximum current provides electrical current. The samples gripping area is inserted into a notch in the clamps. A screw is tightened in the clamps to maintain good electrical contact during the whole experiment. Copper clamps are massive enough to ensure that almost only the sample undergoes notable temperature variations, which prevents degradation of the equipment. As a matter of fact, the volume of one copper clamp is approximately 75000 mm^3 while the volume of the sample is around 750 mm^3 .

Thermal PID regulation synthesis

A PID regulation has been synthesized to pilot the tests with the values of temperature. It is necessary to know the transfer function of the heating system before determining the coefficients of the regulator. The point of interest during the tests is the transformation temperature. An effort is thus made to restrict the interval of temperatures around this point during the synthesis phase in order to increase the performances of the regulation. The procedure for the PID regulation synthesis is the following:

- 1. An initial guess for the PID coefficients is made.
- 2. A PseudoRandom Binary Sequence (PRBS) is generated. Its minimum and maximum values are set to be 800°C and 1000°C respectively.
- 3. The response of the sample to this PRBS in closed loop is recorded.
- 4. From this response, the transfer function of the system is determined using the Output Error (OE) method.
- 5. The optimal PID coefficients are deduced from this transfer function thanks to the ASTA toolbox developed at LS2N laboratory (Centrale Nantes/Université de Nantes).

Pyrometer measurements calibration

Temperature is measured by means of a 2-color pyrometer (SensorTherm METIS M322). 2-color measurements make the measured quantities independent from the intrinsic value of the emissivity of the sample [2]. However, the emissivity slope remains to be calibrated. Pure iron samples are heated in the experimental equipment for the purpose of achieving this calibration. The difference between the measured temperature for the beginning of transformation and its reference value is used to fit the emissivity slope of the pyrometer.

Let us remark that during testing, temperature measurements are only performed at the center of the sample, where the temperature is maximal. The diameter of the pyrometer spot is $0.9 \ mm$, that is much smaller than the sample width.

Atmosphere quality

The box is cleaned from impurities before testing by pumping it down to a vacuum of 3 *mbar* using a rotary vane pump. However, this operation could lead to the degassing of some materials. As a matter of example, Figure 2.9 compares the outgassing performances of various elastomer materials used for sealing applications. It clearly shows that Viton seals are more suitable for vacuum applications. The particles removed from the materials by outgassing could react with the material then deteriorate the quality of images. Consequently, materials with a tendency for outgassing were excluded from the design of the box.



Figure 2.9: Outgassing rates of several elastomer materials [30].

As pointed above, the vacuum levels necessary to protect iron samples could not be reached with the equipment available in the frame of this study. Therefore, the samples are further immersed in an inert argon (Argon Alphagaz 2 from Air Liquide) atmosphere to prevent oxidation.

Heat-haze effect Figure 2.10 shows two images of an iron sample taken by the camera used in the present work at room temperature and at 850°C. A constant flow of argon is maintained

during the tests to keep control over a possible heat haze effect. In the experimental tests, the images exhibit no distortion. No correction is thus applied to the obtained data.



Figure 2.10: Images of an iron sample at room temperature and at 850°C captured by the optical system.

2.3.4 Optical system

Optical equipment

Image acquisition A high-resolution optical camera (Prosilica GT6600 from Allied Vision, 6576x4384 px resolution) is used. It is equipped with a x4 telecentric lens (Opto-Engineering TC 16M 009-F). The magnification it provides allows focusing on a region of interest of 9 mm per 6 mm. With this optical system, a spatial resolution for imaging of 1.4 μ m is attained. At full resolution, the maximum framerate is four images per second. Image acquisition is monitored by means of Vic-Snap software. It is synchronized with the temperature signal coming from the pyrometer so that the values of temperature are recorded each time a photo is taken.

Lighting It is possible to maintain good image contrast up to 1400°C with UV illumination [31]. However, working in the blue wavelengths domain is sufficient for the range of temperatures involved in the study of the ferrite-to-austenite transformation [32]. A blue LEDs ring provides light. A blue filter is put in front of the camera to cut-off higher wavelengths and restrict information to the blue light reflected by the sample. As a proof of the viability of this lighting system, one may consider the gray levels histograms in Figure 2.11. An acceptable contrast is preserved during testing, even at temperatures as high as 950°C.

Effect of the observation window Lyons et al. [22] reduced by 50% the deviation of the correlation results with a higher quality glass. In the frame of this work, high-quality sapphire windows (0-deg orientation, 0.2λ flat) are used as observation windows.

Speckle pattern deposition

Dong et al. [33] tested and compared several patterning techniques, namely:



Figure 2.11: Gray levels histograms of two images taken at different temperatures.

- Spin coating deposition of NiO powder;
- Application of Al2O3 and SiC paints with a fine airbrush;
- Grinding of a polished surface with 3 μm diamond grinding medium in order to obtain random scratches.

They conducted high-temperature experiments and evaluated the quality of the correlation through the mean intensity gradient and the speckle size distribution. Results showed that grinding and Al_2O_3 allowed obtaining the best correlation. Besides, results for a paint sprayed with a 0.18 mm nozzle were more accurate than those with a 0.8 mm nozzle because it helped to reduce the changes of pattern morphology with temperature. Following their recommendations, in the case of this work, an alumina-based paint is deposited on the surface of the sample with a fine nozzle airbrush.

2.3.5 Conclusion: general view of the experimental equipment

Figure 2.12 shows a photo of the experimental set-up, with a sideview on the interior of the testing box. Samples are maintained in this box by two copper clamps, that are visible in Figure 2.12b. In order to prevent buckling during heating, an operative clearance is preserved in such a way that one of the clamps can move freely and follow the sample dilatation movement.



(a) General view of the equipment

(b) View on the interior of the box



2.4 Validation of the experimental set-up: tests on Armco iron sample

2.4.1 Material

For reasons of cost, industrial purity iron was used in calibration tests. The material is Armco iron provided by AKSteel International. Its composition is given in Table 2.2.

The initial Armco iron microstructure is shown in Figure 2.13. In the as-received state, Armco iron has an equiaxed microstructure with a 15 μm average grain size. This grain size is inferior to the resolution of the correlation results. Consequently, the kinematic fields that will be computed will represent the average response of several grains. This obviously hinders any in-depth characterization of the mesoscale mechanical behaviour of Armco iron during the ferrite-to-austenite transformation. However, testing may provide a first proof of viability of the experimental system.



Figure 2.13: Armco iron microstructure in the as-received state.

2.4.2 Temperature solicitation

The temperature profile during the test is shown in Figure 2.14. A plateau is made at 650° C to check that parameters associated with image acquisition, namely exposition time and focusing, are adapted to the capture. The temperature of the sample is raised from 650 to 1000 $^{\circ}$ C in 40s. At the end of this ramp, the power supply is stopped.

The sample being relatively thin, important temperature variations can be expected. With the set-up shown in Figure 2.12 and the sample geometry in Figure 2.6, the maximum heating rate that can be attained is actually 500°C/s. However, the variation rates to be imposed are limited by the framerate of the image acquisition system.

С	Mn	Ni	S	Cr	other elements
0.001	0.05	0.022	0.0028	0.019	< 0.04

Table 2.2: Armco iron composition.



Figure 2.14: Temperature solicitation.

2.4.3 DIC computations

DIC computations are carried out on a rectangular grid of Q4 finite elements. The grid size is 43 pixels per 43 pixels. Regularization is performed on strain. The cut-off wavelength is set at the grid size. Three coarse graining steps are performed. Once displacement is known, strain field is computed with a finite difference scheme.

Elasto plastic strains accommodating the compacity difference between the phases have principal directions that are not necessarily aligned with the lab frame, depending on the orientation of the forming product grain and of the surrounding parent grains. Consequently, shearing can be expected when transformation occurs. We thus choose to represent the Maximum Shear Strain (MSS) quantity, defined as:

$$s_{max} = \sqrt{\left(\frac{E_{xx} - E_{yy}}{2}\right)^2 + E_{xy}^2},$$
 (2.10)

where E is the 2D Green-Lagrange strain tensor. One advantage of this definition is that the $E_{xx} - E_{yy}$ term eliminates thermal expansion.

2.4.4 Macroscopic response

Longitudinal and shear strains averaged over the whole domain of study are displayed in Figure 2.15. The periods of time during which the ferrite-to-austenite and austenite-to-ferrite transformations take place are highlighted by a green overlay. A dashed line separate the heating and the cooling periods.

Let us note that the first image used for DIC computations is supposed to be the reference state, which accounts for the zero value of longitudinal strain at the beginning. During the heating phase, the longitudinal strain exhibits a regular increase due to dilatation followed by a sudden drop caused by transformation. During cooling, the inverse trend is obtained. Longitudinal strain decreases linearly but for a jump towards positive values when the austenite-to-ferrite transformation occurs. The macroscopic longitudinal strain matches the classical dilatation behaviour described in section 1.4.1, which provides a validation for the experimental procedure.

Shear only takes non zero values after the onset of the ferrite-to-austenite transformation. There is indeed local shearing induced by the accommodation of the volume misfit between



Figure 2.15: Evolution of longitudinal and shear strains averaged on the whole domain.

ferrite and austenite. However, it should self-equilibriates at the level of the polycrystal. It may be that the size of the domain of study is too small for the shear strain values to stay at zero. Nonetheless, the shear strain is way less important in magnitude than the longitudinal strain.

2.4.5 Max shear strain

Max shear strain field evolution during testing is displayed in Figure 2.16. In the first image, MSS equals zero everywhere. There is no shearing inside the material. The sample is only submitted to thermal expansion that is isotropic.

MSS takes non zero values starting from Figure 2.16b. This evolution proves that the ferriteto-austenite transformation induces shearing and that it is possible to use this information to track the progress of the allotropic transformation. In the legend of the figure, temperature at which strain localizations first appear is called $T_{\alpha-\gamma}$. This definition is of course only valid at the center of the sample and it ignores the temperature gradient. The transformation begins in the zone where the temperature is maximal. It then reaches the zone of the sample where a thermal gradient is present. Between Figure 2.16c and Figure 2.16f, a transformation front forms and progresses towards the extremities of the sample. Figure 2.16f corresponds to the end of the heating phase.

As the power supply is stopped after heating, the austenite-to-ferrite transformation occurs very quickly. Figure 2.16g and Figure 2.16h show that during cooling, the MSS goes down to zero in a large part of the sample, although MSS peaks associated with microplasticity remain. This indicated that the microplasticity induced by the ferrite-to-austenite transformation contributed to drive the response of the material during the austenite-to-ferrite transformation, that tried to reduce global straining.

The localizations of the MSS quantity seem to be promising information for the study of allotropic transformations in pure iron. Nonetheless, the zones concerned by these localisations have approximately the same size as the individual grains of the microstructure. Therefore, the obtained results cannot be used for an in-depth study of mesoscopic phenomena.

CHAPTER 2



(i) t=80 s, T = $T_{\alpha-\gamma} + 97K$

Figure 2.16: Maximum shear strain evolution with temperaure variations (in percent). The size of the domain of study is 7.6 mm per 3.0 mm.

2.5 Conclusion

An experimental device has been developed for the *in situ* observation of phase changes in metals. The device is suitable for any phase change that involves a change in volume between the two phases. Such a change induces microplasticity that is taken advantage of to track the progress of the transformation.

In the design of the experimental set-up, care has be taken to avoid image degradation and to develop a flexible heating system. It is hoped that this will be a gateway for other usages of the apparatus with temperature loadings and resolutions tailored for the applications. The validity of the equipment has been assessed on an Armco iron sample. At the macroscopic scale, the response of the material is similar to that obtained in classical dilatometry tests. At the scale of a few grains, the observation of the Max Shear Strain (MSS) quantity is shown to be a good indicator of the progress of the transformation. Although the tested microstructure is a bit small with respect to image resolution, strain localizations can be observed when transformation occurs. Tests on coarser microstructures would help to understand better the subgrain phenomena that are at stake in this process.

The proof of feasibility brought by the experiment on Armco iron is an encouraging step towards more elaborate testing. In order of priority, the following studies will be carried on to address the coupling between plasticity and allotropic transformations:

- performing tests on grains with coarser microstructures in order to understand how microplasticity develops inside the grains;
- recording a full cycle of heating and cooling of the samples to relate the final microstructure with the transformation history of the sample;
- introducing initial plastic straining inside the material to visualize how it influences the onset of the transformation.

These developments will be the topic of the next section.

Bibliography

- [1] M.M. Attallah, H. Terasaki, R.J. Moat, S.E. Bray, Y. Komizo, and M. Preuss. In-Situ observation of primary γ' melting in Ni-base superalloy using confocal laser scanning microscopy. *Materials Characterization*, 62(8):760–767, August 2011. 20
- [2] Xin-Fang Zhang and Yu-ichi Komizo. In Situ Investigation of the Allotropic Transformation in Iron. *Steel Research International*, 84(8):751–760, August 2013. 20, 29
- [3] Jin-mo LEE, Koji SHIBATA, Kentaro ASAKURA, and Yasuyuki MASUMOTO. Observation of $g \rightarrow a$ Transformation in Ultralow-carbon Steel under a High Temperature Optical Microscope. *ISIJ International*, 42(10):9, 2002. 20
- [4] S Niknafs and R Dippenaar. In-situ Observation of the Delta-ferrite to Austenite Phase Transition in Fe-C Alloys. page 11. 20
- [5] G. Zijlstra, M.S.B. van Daalen, D.I. Vainchtein, V. Ocelík, and J.Th.M. De Hosson. Interphase boundary motion elucidated through in-situ high temperature electron back-scatter diffraction. *Materials & Design*, 132:138–147, October 2017. 20, 21
- [6] M. Maalekian, R. Radis, M. Militzer, A. Moreau, and W.J. Poole. In situ measurement and modelling of austenite grain growth in a Ti/Nb microalloyed steel. *Acta Materialia*, 60(3):1015–1026, February 2012. 21
- [7] S. Eric Offerman and Hemant Sharma. Following individual grains during solid-state phase transformations with 3dxrd microscopy. *Comptes Rendus Physique*, 13(3):268– 279, April 2012. 21
- [8] V. I. Savran, S. E. Offerman, and J. Sietsma. Austenite Nucleation and Growth Observed on the Level of Individual Grains by Three-Dimensional X-Ray Diffraction Microscopy. *Metallurgical and Materials Transactions A*, 41(3):583–591, March 2010. 21

- [9] Zengmin Shi, Yo Tomota, Stefanus Harjo, Yuhua Su, Bo Chi, Jian Pu, and Li Jian. Effect of non-isothermal deformation of austenite on ferrite transformation behavior studied by in-situ neutron diffraction. *Materials Science and Engineering: A*, 631:153–159, April 2015. 21
- [10] P.G. Xu, Y. Tomota, P. Lukáš, O. Muránsky, and Y. Adachi. Austenite-to-ferrite transformation in low alloy steels during thermomechanically controlled process studied by in situ neutron diffraction. *Materials Science and Engineering: A*, 435-436:46–53, November 2006. 21
- [11] M. Anwander, B. G. Zagar, B. Weiss, and H. Weiss. Noncontacting strain measurements at high temperatures by the digital laser speckle technique. *Experimental Mechanics*, 40(1):98–105, March 2000. 21
- [12] D. Campello, N. Tardif, J. Desquines, M.-C. Baietto, M. Coret, A. Maynadier, and P. Chaudet. Validation of a multimodal set-up for the study of zirconium alloys claddings' behaviour under simulated loca conditions. *Strain*, 2018. 21
- [13] Mallory C. Casperson, Jay D. Carroll, John Lambros, Huseyin Sehitoglu, and Robert H. Dodds. Investigation of thermal effects on fatigue crack closure using multiscale digital image correlation experiments. *International Journal of Fatigue*, 61:10–20, April 2014. 21
- [14] Paul Leplay, Julien Réthoré, Sylvain Meille, and Marie-Christine Baietto. Identification of asymmetric constitutive laws at high temperature based on Digital Image Correlation. *Journal of the European Ceramic Society*, 32(15):3949–3958, November 2012. 21
- [15] Bharath Swaminathan, John Lambros, and Huseyin Sehitoglu. Digital image correlation study of mechanical response of nickel superalloy Hastelloy X under thermal and mechanical cycling: Uniaxial and biaxial stress states. *The Journal of Strain Analysis for Engineering Design*, 49(4):233–243, May 2014. 21
- [16] W.G. Mao, J. Chen, M.S. Si, R.F. Zhang, Q.S. Ma, D.N. Fang, and X. Chen. High temperature digital image correlation evaluation of in-situ failure mechanism: An experimental framework with application to C/SiC composites. *Materials Science and Engineering: A*, 665:26–34, May 2016. 22, 24
- [17] T. Chauve, M. Montagnat, and P. Vacher. Strain field evolution during dynamic recrystallization nucleation; A case study on ice. *Acta Materialia*, 101:116–124, December 2015. 22
- [18] Fabio Di Gioacchino and João Quinta da Fonseca. An experimental study of the polycrystalline plasticity of austenitic stainless steel. *International Journal of Plasticity*, 74:92– 109, November 2015. 22
- [19] Adrien Guery, François Hild, Félix Latourte, and Stéphane Roux. Slip activities in polycrystals determined by coupling DIC measurements with crystal plasticity calculations. *International Journal of Plasticity*, 81:249–266, June 2016. 23
- [20] R.Y. Chen and W.Y.D. Yuen. Review of the high-temperature oxidation of iron and carbon steels in air or Oxygen. *Oxidation of metals*, 59:433 467, 2002. 23
- [21] B M B Grant, H J Stone, P J Withers, and M Preuss. High-temperature strain field mea-

surement using digital image correlation. *The Journal of Strain Analysis for Engineering Design*, 44(4):263–271, May 2009. 23

- [22] J. S. Lyons, J. Liu, and M. A. Sutton. High-temperature deformation measurements using digital-image correlation. *Experimental Mechanics*, 36(1):64–70, March 1996. 23, 30
- [23] Mark D. Novak and Frank W. Zok. High-temperature materials testing with full-field strain measurement: Experimental design and practice. *Review of Scientific Instruments*, 82(11):115101, November 2011. 23
- [24] Yun Quan Su, Xue Feng Yao, Shen Wang, and Yin Ji Ma. Improvement on measurement accuracy of high-temperature DIC by grayscale-average technique. *Optics and Lasers in Engineering*, 75:10–16, December 2015. 23
- [25] N. Cholewa, P. T. Summers, S. Feih, A. P. Mouritz, B. Y. Lattimer, and S. W. Case. A Technique for Coupled Thermomechanical Response Measurement Using Infrared Thermography and Digital Image Correlation (TDIC). *Experimental Mechanics*, 56(2):145– 164, February 2016. 24
- [26] J. Réthoré. Automatic crack tip detection and stress intensity factors estimation of curved cracks from digital images. *International Journal for Numerical Methods in Engineering*, 103(7):516–534, August 2015. 24
- [27] G. Besnard, F. Hild, and S. Roux. "Finite-Element" Displacement Fields Analysis from Digital Images: Application to Portevin–Le Châtelier Bands. *Experimental Mechanics*, 46(6):789–803, December 2006. 25
- [28] Jérémy Marty, Julien Réthoré, Alain Combescure, and Philippe Chaudet. Finite Strain Kinematics of Multi-scale Material by Digital Image Correlation. *Experimental Mechanics*, 55(9):1641–1656, November 2015. 25
- [29] J.T. Bosiers, I.M. Peters, C. Draijer, and A. Theuwissen. Technical challenges and recent progress in CCD imagers. *Nuclear instruments and Methods in Physics Research A*, 565:148 – 156, 2006. 26
- [30] R.N. Peacock. Practical selection of elastomer materials for vacuum seals. *Journal of Vacuum Science & Technology*, 1980. 29
- [31] Yali Dong, Hideki Kakisawa, and Yutaka Kagawa. Optical system for microscopic observation and strain measurement at high temperature. *Measurement Science and Technology*, 25(2):025002, February 2014. 30
- [32] Bing Pan, Dafang Wu, Zhaoyang Wang, and Yong Xia. High-temperature digital image correlation method for full-field deformation measurement at 1200 °C. *Measurement Science and Technology*, 22(1):015701, January 2011. 30
- [33] Yali Dong, Hideki Kakisawa, and Yutaka Kagawa. Development of microscale pattern for digital image correlation up to 1400°C. *Optics and Lasers in Engineering*, 68:7–15, May 2015. 30

3

Mesoscale experimental investigation of the interplay between mechanics and allotropic transformation

The previous part has highlighted the need for coarse microstructures in order to study qualitatively the coupling between plasticity and allotropic phase transformations. Several restoration mechanisms can be taken advantage of to attain grain coarsening, namely normal and abnormal grain growth as well as primary recrystallization. Exploring several treatments allows assessing the influence of the initial microstructure on the ferrite-to-austenite transformation onset.

On samples that were submitted to a recrystallization thermomechanical treatment to coarsen the microstructure, there are less shear strain localizations than on the Armco iron samples. Moreover, the locations of these localization points can be put in relation with the initial microstructure. The same conclusion can be drawn for samples that underwent a strain annealing treatment but the presence of very small grains makes the interpretation of the results more difficult.

Electron BackScattered Diffraction (EBSD) mappings are performed before and after the tests so as to track microstructural evolutions of the samples. However, the information on the morphology of the austenite phase is lost in the process of testing the samples. A methodology, based on the assumption of a transformation path at each point, is proposed to infer which variant was formed when ferrite transformed into austenite.

A comparison is finally made between the behaviour of a reference sample and the behaviour of samples plastically deformed in tension. Contrarily to what could be expected, the localization areas are even less numerous during the ferrite-to-austenite transformation when samples are pre deformed, which indicates that the nucleation sites are concentrated in some specific areas. Besides, these areas are more diffuse because the first steps of growth are piloted by the gradient of defect energy.

3.1 Samples preparation

3.1.1 Need for coarse microstructures

As stated in the previous chapter, getting coarse microstructures is extremely important with respect to the interpretation of the obtained strain fields. Consequently, efforts have been made to come up with a thermomechanical treatment that would provide coarse initial microstructures.

Three possibilities can be considered to coarsen the samples microstructure based on Section 1.2:

- the first one is to perform strain annealing, as described in section 1.2.3. For an amount of cold rolling of 5% and an annealing time of 150 h, Pippan [1] obtained 3 mm grains.
- the second one is to anneal the samples in the austenite domain and to take advantage of the high mobility of grain boundaries in temperature. This method is used in [2] to obtain grains with a size of $300 \ \mu m$. It is also used in [3] and in [1] with a less positive result.
- the third one is to introduce an important amount of deformation in the material to initiate primary recrystallization and then to fit the deformation and annealing conditions to obtain coarse grains.

The facilities to perform these treatments were not all available at Ecole Centrale de Nantes. Only the strain annealing method was done at Ecole Centrale de Nantes in a furnace that did not, however, have a vacuum atmosphere. The austenization method was explored in Institut des Matériaux Jean Rouxel (Université de Nantes/CNRS) thanks to the precious help of Dr Emmanuel Bertrand. Recrystallised samples were prepared at Laboratoire Georges Friedel (Ecole des Mines de Saint-Etienne/Université de Lyon/CNRS). The assistance of Prof Guillaume Kermouche and Ms Marilyne Mondon is greatly appreciated.

3.1.2 Recrystallized samples

High purity (99.9997% purity) iron samples were made in house by cold crucible melting. Cold-rolling was used to deform the samples. They were placed in a sealed silica ampoule under a 200 *mbar* partial pressure of argon. They were heated up to 800 °C and maintained in temperature for 2 hours. They were then cooled in a furnace. As a result of this process, the microstructure is equiaxed and the grains are rather coarse. The maximum diameter reached by the ferrite grains is 800 μm .

3.1.3 Austenized samples

Armco iron was the base material for this treatment. Samples were austenized and maintained in temperature according to the profile in Figure 3.1.

Samples were cooled at a slow rate in order to let time for the ferrite nuclei to grow and to reach the boundaries of the former austenite grains. This method should be suitable to attain coarse-grained microstructure. However, nucleation of small ferrite grains cannot be avoided, which causes the grains sizes distribution to be very large.



Figure 3.1: Temperature profile imposed to the sample during the austenization treatment.

3.1.4 Strain annealed samples

Determination of critical strain for strain annealing

As explained in section 1.2.3, it is necessary to first know the critical strain for grain growth. It has been determined using trapezoidal samples. The geometry of these samples is shown in Figure 3.2. The section of the sample increases linearly so that a gradient of longitudinal strain will be present in the material. Another advantage of this geometry is that Armco iron is subject to plastic instabilities in the form of Lüders bands. The geometric irregularity of the sample tends to attenuate this effect.



Figure 3.2: Drawing of the trapezoidal samples. Dimensions are in *mm*. The thickness of the sample is 3 *mm*.

A speckle pattern is deposited on the sample. In order to get an acceptable resolution on kinematic fields, the following procedure is followed:

- 1. Deposition of a black paint background;
- 2. Projection of white paint drops using an air spray;
- 3. Deposition of India ink droplets using an airbrush;
- 4. Deposition of small white paint droplets using an airbrush.

Samples are tested in traction on an MTS 100kN machine. Images of the sample are captured using a Prosilica GT6600 camera (see section 2.3.4) and the displacement field is obtained with the DIC software UFreckles.

After testing, the sample is polished and a micrography is made. The zone with the coarsest grains is put in relation with the longitudinal strain field so as to infer the value of the critical strain. The final value for the critical strain is 5.2%.

Thermomechanical treatment

Once the critical strain for strain annealing is known, samples are deformed to the critical strain using the same machine. The loading is driven by the displacement and the testing is performed incrementally. At each step, a small displacement increment is applied to the sample. The test is paused and a correlation calculation is performed using VIC-2D software. UFreckles software was not used here because it was not accessible on the testing computer. However, this should not be too detrimental to the correlation results, all the more so that no particular precision is required for that application. The longitudinal strain value is obtained as a result of the correlation computations. The test is stopped when this value matches the critical strain.

Samples are then annealed in a furnace. They are heated from room temperature to 850° C in one hour and maintained in temperature during one hour. The furnace used for this treatment do not operate under vacuum. Consequently, the sample is grinded with 500 to 4000 SiC papers and polished with 1 μm diamond suspension after the thermomechanical treatment. A Nital solution (2% nitric acid, 98% ethanol) is used to etch the metal and reveal the ferrite microstructure.

It is well-known that mechanical polishing induces cold-working at the surface of the sample. The microstructure beneath the surface exhibit in some cases a polygonized form [4]. Consequently, the kinematic fields that will be observed will not represent the behaviour of a sample in an annealed state. Rather, the mechanical state at the surface will influence the behaviour in transformation. That said, it could also be beneficial since it will concentrate most transformation events at the surface of the sample, thus making the observations easier.

3.1.5 Conclusion: tested samples nomenclature

A summary of the tested samples and their denomination is presented in Table 3.1. They are divided into three categories: A for austenized samples, R for recrystallized samples and SA for strain annealed samples. A4 is the sample presented in the previous chapter.

Several tests are carried out on samples produced with the same thermomechanical treatment. The observed phenomena are so microstructure-dependent that it would be excessive to claim that this is enough to assess the repeatability of the tests. However, the additional tests were used to check that the observed trends were qualitatively similar. The following points could be verified:

- samples obtained with a similar treatment have comparable mean grain sizes and textures;
- the density of nucleation events captured by the experimental equipment is approximately the same;
- the range of variations of the Maximum Shear Strain (MSS) during the test is similar.

In the rest of this chapter, only a selection of the results obtained with these samples will then be presented. The next section will describe the general procedure adopted to characterize the behaviour of the iron samples during heating and cooling at the mesoscale while the rest of the chapter will be devoted to the presentation of the results obtained with the different microstructures and under different loading conditions.

3.2 Procedure for the mesoscopic study of allotropic transformations

3.2.1 Pre and post mortem EBSD

The testing methodology is the following:

- 1. A first EBSD analysis is made;
- 2. The speckle pattern evoked in section 2.3.4 is applied on the surface of the sample.
- 3. The sample is heated up to the transformation temperature then cooled to room temperature in the experimental equipment;
- 4. after testing, the sample is cleaned in an ultrasonic oscillated bath of ethanol and another EBSD analysis is carried out. Grains morphologies and orientations in the final state are thus captured.

EBSD mappings acquisition

In the case of R samples (cf Table 3.1), Electron Back Scattered Diffraction is performed before testing in order to obtain information on initial grain sizes and texture. To map the largest region possible, more than 150 acquisitions with a resolution of $5\mu m$ and a magnification of x250 are performed and the corresponding "submaps" are stitched together. EBSD raw data are then processed with the MTEX toolbox [5]. Grains boundaries are computed with a tolerance of 5 degrees. Non indexed regions are left blank.

Landmarks

Two different observation tools are used in the process of testing. It is thus necessary to put marks on the samples in order to ensure to study the same zone every time. Three landmarks are made on the sample by micro-indentation to register experimental images and EBSD maps. They are deliberately placed far from the zone of interest not to introduce residual strain that

Sample	Thermal	Cooling	Migrographies	Initial	Final	Pre
name	treatment	recorded	wherographies	EBSD	EBSD	deformed
A1	Austenization		·	\checkmark	\checkmark	
A2	Austenization	\checkmark	\checkmark			
A3	Austenization	\checkmark	\checkmark			
A4	None	\checkmark	\checkmark			
R1	Recrystallization	\checkmark		\checkmark	\checkmark	
R2	Recrystallization	\checkmark		\checkmark		
R3	Recrystallization	\checkmark				\checkmark
SA1	Strain annealing		\checkmark			
SA2	Strain annealing	\checkmark	\checkmark			\checkmark
SA3	Strain annealing	\checkmark	\checkmark			\checkmark

Table 3.1: Samples denomination	1.
---------------------------------	----

may have an influence on the transformation onset. Registration is made by identifying the affine transformation that best warps the EBSD map to the photos taken by the camera. It makes use of the Procrustes algorithm.

3.2.2 Temperature loading

The temperature loading when cooling is not controlled is presented in section 2.4.2. As far as the tests for which cooling is studied are concerned, temperature follows the profile in Figure 3.3. Around the transformation region, the target solicitation consists of a rising temperature ramp followed by a plateau and a falling temperature ramp. As earlier, another plateau is made at 650°C to check that parameters associated with image acquisition are adapted to the capture. Photos are taken between t = 50s and t = 150s, which means that around 400 images are available for post-processing at the end of the experiment. The advantage of implementing coarsening treatments is that samples grain sizes become sufficient to justify not measuring temperature and kinematic fields on the same side of the sample.



Figure 3.3: Temperature solicitation.

3.3 Results of heating and cooling tests on undeformed samples

Two kinds of microstructures have been tested in the experimental device without applying a mechanical solicitation before testing. Recrystallized samples have an equiaxed microstructure with grains of similar size whereas austenized samples present some large grains but exhibits a spread distribution of grain sizes.

3.3.1 Recrystallized samples

Initial microstructure

Obtained orientations are displayed in Figure 3.4 for the sample R1. They are colorized according to the given Inverse Pole Figure.

The microstructure is equiaxed and the mean initial grain size is around 250 μm , as confirmed by the grain sizes histogram shown in Figure 3.5. This microstructure is adequate for this work since it has to be much coarser than the image resolution of optical cameras to capture sub grain phenomena. A typical recrystallisation γ -fiber texture [6] can be expected given



Figure 3.4: Grains orientations as obtained from EBSD measurements. Grains are colorized according to the adjacent key Inverse Pole Figure.

that the samples are annealed. Indeed, 48.1% of the measured orientations in Figure 3.4 are reported to be along that fiber with a tolerance of 10 degrees.



Figure 3.5: Histogram of equivalent grain diameters in the studied area.

Macroscopic response

Longitudinal and shear strains averaged over the whole domain of study are displayed in Figure 3.6. The color code is the same as in part 2.4.4: the periods of time during which the ferrite-to-austenite and austenite-to-ferrite transformations take place are highlighted by a green overlay and dashed lines separate the heating phase, the temperature plateau and the cooling phase.

The macroscopic longitudinal strain matches the classical dilatation behaviour with ruptures in a linear thermal expansion behaviour induced by the ferrite-to-austenite and austenite-toferrite transformations. The shear is equal to zero before the ferrite-to-austenite transformation then drops to negative values. As explained in section 2.4.4, the microstructure is too coarse for the shear strain to stay equal to zero at the macroscopic level.

Maximum shear strain field evolution during the ferrite-to-austenite transformation

The finite element mesh used for DIC computations has a grid size of 25 pixels, which is also the cut-off wavelength for regularization. Three levels of coarsening are considered. Convergence is attained once the norm of the increment of displacement is inferior to 0.01 pixel. Figure 3.7 shows the evolution of the Maximum Shear Strain (MSS) quantity during the test. The initial microstructure extracted from the EBSD measurements of section 3.3.1 is superimposed on the correlation results.

During heating and before transformation, the sample exhibits a dilatation deformation that is purely isotropic: MSS equals zero (Figure 3.7a). When transformation begins, strain local-



MESOSCALE EXPERIMENTS

Figure 3.6: Evolution of longitudinal and shear strains averaged on the whole domain. The letters correspond to the images labels in Figure 3.7.

izations start appearing (Figure 3.7b). The "stationary" transformation behaviour is at stake between Figure 3.7c and Figure 3.7f. Then the transformation front develops and progresses in the sample (Figures 3.7g to 3.7l). After some time, the temperature plateau is reached. Figure 3.7m depicts the maximum advancement of the transformation front. During cooling, once temperature allows the occurence of the austenite-to-ferrite transformation, a front propagates from the extremities of the sample to its center (Figure 3.7n to 3.7r). Eventually, the two opposing fronts coming from both extremities join (Figure 3.7s). Due to orientation incompatibilities, high plastic strains are recorded at this moment (Figure 3.7t).

There is globally a relation between the location of strain localization points and triple points of the initial microstructure, which indicates that nucleation events occurred preferably at grain boundaries. However, in certain cases the zones at which strain localize does not seem to correlate with the underlying microstructure. This may be due to the fact that the grains are smaller than the thickness of the sample. Therefore it is possible that some grains nucleated in the bulk of the material, thus inducing strains that are recorded when studying the surface of the sample.

Error quantification

The error committed during DIC computations should be checked if one wants to use quantitative values of the strain field. The local error of DIC computations can be defined as the gap between the gray values of the reference image and of the other image warped with the calculated displacement. Using the notations of part 2.2.3, it reads:

$$err = ||g(\boldsymbol{x} + \boldsymbol{u}(\boldsymbol{x})) - f(\boldsymbol{x})||$$
(3.1)

Figure 3.8 displays the mapping of this local error at the beginning of the temperature plateau, *i.e.* at t = 130s.

As can be seen from Figure 3.8, the local error stays under 5% in a very large part of the sample, which underlines the reliability of the method when it comes to the computation of

CHAPTER 3



Figure 3.7: Maximum shear strain evolution with temperaure variations (in percent). The size of the domain of study is 5.8 mm per 1.4 mm.

kinematic fields during the ferrite-to-austenite transformation. Moreover, the zones in which the error exceeds this 5% value can manifestly be related with the grain boundaries of the newly formed austenite. This behaviour could be expected given grain boundaries act as discontinuities that interfere with the correlation routine. It should not be detrimental to the characterization of allotropic phase change since the relevant evidence of the transformation process is to be searched in the strain localizations around the newly formed grains.



Figure 3.8: Pixelwise error in gray levels of correlation calculation at the end of heating phase (in per cent).

Propagation of the α/γ and γ/α transformation fronts

MSS values are used to study the kinetics of front propagation during the ferrite-to-austenite transformation. In order to distinguish between transformed and untransformed regions, a criterion on the MSS values is required. A value too high would not render the transformational behaviour properly whereas a value too low would make the front position dependent on the noise on the correlation results. It is chosen arbitrarily that any area where MSS is superior to 0.003 is considered to be transformed. This threshold introduces a clear demarcation between the zones upstream and downstream of the front. The result of this binarization process is presented in Figure 3.9.

The contours of the transformed region are shown in red. The area of the transformed zone is calculated in terms of number of pixels then converted to square millimetres. The green rectangles in Figure 3.9 are equivalent regions whose surface is equal to that of the transformed region. The evolution of the area of these rectangles is displayed in Figure 3.10 as a function of time.

It can be seen that the evolution of the transformed area follows regular profiles except at the end of cooling. At that moment, Figure 3.9 highlights that the front cannot be tracked, which may have several explanations:

- the MSS field at the end of the experiment is the combination of the fields resulting from both the ferrite-to-austenite and the austenite-to-ferrite transformations. The microplasticity induced by the former transformation influence the reverse transformation, which occurs in such a way that overall straining is reduced. Nonetheless, the MSS values do not go back to zero everywhere and the resulting kinematic fields are complex;
- it is also possible that the transformation from austenite to ferrite is too fast to be captured in the conditions of the experiment so that an interface between ferrite and austenite does not really exist at the end of the experiment. This would be the case is displacive mechanisms are involved, which could be only confirmed by studying the final microstructure of the samples.

Between points A and B, the response of the material in transformation is linear. By supposing that the transformed fraction grows only in one direction, a speed of $2 mm.s^{-1}$ can be calculated, altough this value is far from being precise. It is lower than the value given in



Figure 3.9: Representation of the transformed area after thresholding on MSS values.

section 1.3.1 but both fall in the same order of magnitude. Such a value for the speed of transformation combined with the possibility to identify a linear increase in transformed area is a clue that the ferrite-to-austenite transformation operates via diffusive mechanisms. Between points B and D, the evolution of the transformed area follows the temperature profile imposed to the sample. It can be understood that the temperature gradient limits the transformation rate and that the transformation progresses as the transition temperature for the ferrite-to-austenite temperature is crossed in given portions of the sample.



Figure 3.10: Evolution of the transformed area as a function of time for the R1 sample.

Obtained ferrite microstructure

Figure 3.11 shows a mapping of grain orientations after testing. Only 17.4% of the orientations are within the γ -fiber texture (considering the same tolerance of 10 degrees as above), which indicates an evolution in the texture of the material. The EBSD mapping in Figure 3.11 also exhibits more intragranular misorientations compared to the initial analysis. This is a consequence of the accumulation of dislocations that cause local rotations [7], which confirms that the allotropic transformation has been accommodated elasto plastically.

Besides, Figure 3.11 shows the formation of acicular grains reminding of Widmanstätten ferrite [8]. This structure indicates that displacive mechanisms took place during the austenite-to-ferrite transformation. The alternation of grains with two orientations is characteristic of a mechanism in which two variants formed while mutually accommodating the deformation induced by each other.



Figure 3.11: *Post mortem* EBSD analysis. Grains are colorized according to the adjacent key Inverse Pole Figure.

Minimal rotation angles can be computed point by point between the initial and final states to better highlight the differences between the two. They are defined as the angle associated with the rotation:

$$\underline{\Delta O} = \underline{O_i} \times \overline{O_f}^{-1} \tag{3.2}$$

where $\underline{O_i}$ and $\underline{O_f}$ are the orientation tensors in the initial and final states respectively. These angles are represented in Figure 3.12.

Three zones, delimited by red dotted lines, can be roughly identified in Figure 3.12:

- the zone to the right (1) corresponds to grains that were not reached by the transformation front. Consequently, the minimal rotation angle between the two states equals zero;
- in the central part (2), that is the zone of propagation of the front, areas with similar angular differences are quite large. Growth phenomena seem to have been dominant over



Figure 3.12: Minimal rotation angles in degrees between initial and final states.

nucleation events during the ferrite-to-austenite transformation. This results in a microstructure with elongated grains. Bayraktar et al. [9] reported in a study on the welding of interstitial free steels that a sharp temperature gradient may prevent the incubation period for the nucleation of the new phase from finishing. Consequently, no new grain is nucleated and the already formed grains keep growing. Such a competition between nucleation and growth rates seems to be also at stake here.

 in the zone to the left (3), where fronts join, the presence of small grains relates to the accommodation of orientation incompatibilities.

Local behavior around a triple point

Figure 3.13 provides a zoom on a region of the sample located around a triple point. This zone is indicated with a green square in Figure 3.7u. Again, the initial microstructure is superimposed on the correlation results. MSS can be viewed as a norm that quantifies mechanical perturbations induced by the transformation. The shear strain E_{xy} field is represented here in order to distinguish between shearing directions.

Two shear strain extrema, highlighted by the dotted circles, develop inside the material between the two snapshots. It can be inferred from this observation that an austenite grain nucleated near the zone marked by the black arrow, that is to say close to the triple point area. The ferrite grains at the top and the bottom of the image had to accommodate the volume misfit. The opposite signs of elasto plastic shear strains are a consequence of different initial orientations of the ferrite grains.

Partial conclusion

The microstructure of R samples is coarse enough so that even with an optical camera equipped with a telecentric lens, the resolution is sufficient to study the evolution of the strain fields at a subgrain scale. Iron samples are submitted to temperature ramps crossing the equilibrium ferrite-to-austenite and austenite-to-ferrite transition temperatures, with the heating rate being adjusted to take enough photos.

MESOSCALE EXPERIMENTS



Figure 3.13: Shear strain field near a triple junction before and after the onset of transformation (in percent).

During heating, the conditions of the present study dissociate two behaviours depending on whether the temperature is homogeneous in the zone of the sample reaching the transformation temperature. During the first phase, the adjacency of shear strain peaks is shown to be a marker of the nucleation of a new austenite grain. The second phase corresponds to the propagation and stabilization of a transformation front. *Post mortem* analysis of grain orientations reveal that front propagation gives priority to growth events over nucleation events. During cooling, transformation fronts propagate backwards, erasing most of shearing, except at the center of the sample where residual plastic straining remains. The fronts junction induces a lot of nucleation phenomena in an attempt to accommodate orientations misfit.

Finally, a study on the propagation of the transformation front shows that the temperature gradient limits the rate of the ferrite-to-austenite and austenite-to-ferrite transformations. Considering the behaviour of the material before the constitution of the front, it may be thought that the ferrite-to-austenite transformation is mainly diffusive whereas the austenite-to-ferrite transformation is mainly displacive.

3.3.2 Reconstruction of the austenite phase

An information that is missing in the experiments of the previous paragraph is the morphology of the austenite phase. In this section, a methodology relying on simple assumptions is proposed to reconstruct the phase in temperature. The method lies on the following hypotheses:

- the transformation from ferrite to austenite is diffusive and a coherent interface is always formed between the parent and product phases. Consequently, an orientation relationship can be assumed between a forming austenite grain and one of the ferrite grains of the initial microstructure.
- the transformation from austenite to ferrite is displacive and an orientation relationship exists between the forming grain and the austenite grain it formed in.

Motivation based on experimental observations

Figure 3.14 displays an histogram of angular differences in the central zone (2) of Figure 3.12. It can be seen in this Figure that angle differences in the central zone of the domain of study are comprised within a discrete set of angles. This specific distribution might be a clue for the identification of the austenite orientations that are selected during the transformation

process.



Figure 3.14: Histograms of angle differences in the central zone of Figure 3.12.

Moreover, lots of grain boundaries possess a twinning Orientation Relationship (OR) as shown in Figure 3.15. These two elements show that the orientations in the final microstructure are highly constrained, which makes it conceivable to propose a transformation path for the ferrite-to-austenite-to-ferrite cycle.



Figure 3.15: Final microstructure with the twinning boundaries highlighted in red.

Algorithm overview

The main steps of the algorithm are described in Figure 3.16. The generation of the transformation path relies on the comparison between the experimental angular difference between the initial and final states and the possible theoretical angular differences. The comparison is made using the Euclidean norm of the quaternion representations of misorientations.

Generation of the possible final ferrite orientations

Let $\underline{O_i}$ be the reference ferrite orientation. The goal of this section is to build the set of possible orientations that arise from a transformation to austenite and back to ferrite again. A polar decomposition of the transformation gradient can be performed: $\underline{F_t} = \underline{P} \cdot \underline{T}$ where \underline{P} is a rotation and \underline{T} is a stretch tensor. The combination of an orientation and a distortion will be called variant in what follows. In the case of a diffusive transformation, the distortion is of course not a relevant descriptor for the variant. However, the idea behind the present algorithm is to build the set of possible variants using the Bain distortion and keeping only the information on variant orientations.

One way to obtain the possible transformation gradients $\underline{F_t}$ would be to consider that the transformation minimizes straining in the material. It is the method adopted in [10]. In this



Figure 3.16: Algorithm for obtaining the austenite phase orientation.

work, the set of variants is generated using crystal symmetries starting from one reference transformation path. This procedure has first been introduced by Lew et al. [11] and is detailed in [12].

We introduce the following notations: \underline{T}^i is the distortion to obtain the ith variant expressed in the lab frame, \underline{T}^i_j is the distortion to obtain the ith variant expressed in the frame of the jth variant's frame. Cubic crystal symmetry imposes that each variant *i* possesses a set of point group rotations $\{\underline{R}_i^{\ 1}, ..., \underline{R}_i^n\}$ that leave the lattice unchanged. The idea behind the algorithm is to find the point group rotations that are not shared between the parent and the product variants. Let us consider a ferrite variant f1 obtained with the distortion \underline{T}^{f1} . If one variant of the austenite phase, obtained with the distortion \underline{T}^{a1} , is known, the distortions associated with other possible austenite variants aj are obtained with:

$$\underline{\boldsymbol{T}^{aj}} = \underline{\boldsymbol{R}_i}^T \cdot \underline{\boldsymbol{T}^{a1}} \cdot \underline{\boldsymbol{T}^{f1}}^{-1} \cdot \underline{\boldsymbol{R}_i} \cdot \underline{\boldsymbol{T}^{f1}}^{-1} \cdot (3.3)$$

Likewise, the rotations in the point group of symmetry of the variant aj are updated with:

$$\underline{\underline{R}_{aj}} = \underline{\underline{R}_i}^T \cdot \underline{\underline{R}_{a1}} \cdot \underline{\underline{R}_i}$$
(3.4)

Let us investigate the Kurdjumov-Sachs (KS) OR. The deformation gradient for the austenite-to-ferrite transformation comprises a Bain distortion and a rotation. According to Wayman and Bhadeshia [13], the Bain distortion reads:

$$\underline{B} = \begin{bmatrix} \sqrt{2} \frac{a}{a_0} & 0 & 0\\ 0 & \sqrt{2} \frac{a}{a_0} & 0\\ 0 & 0 & \frac{c}{a_0} \end{bmatrix}.$$
(3.5)

 a_0 is the lattice parameter of the BCC lattice and a = c the lattice parameter of the FCC lattice. The rotation associated with the transformation is taken from [14]. The transformation from ferrite to austenite is studied first. At the end of the generation of the possible variants, we compute the misorientations between ferrite and austenite. They are plotted in the (100) pole figure of Figure 3.17. They are in good agreement with the pole figure presented in [14].



Figure 3.17: Comparison of the obtained (100) pole figure for the misorientations between ferrite and austenite with the results of Koumatos et al. [14].

The misorientations after a full ferrite-to-austenite-to-ferrite cycle are then computed and represented in the (100) pole figure of Figure 3.18. As can be seen from the Figure, an important number of orientations can stem from one single initial orientation.





Comparison of the obtained misorientations

Let be a point M in the initial EBSD dataset. It has an orientation \underline{O}_M and it is part of a grain g. The construction of the transformation pathway involves the following steps:

- the corresponding point in the final configuration is retrieved. Let O_f be its orientation;
- a set Φ containing the mean orientations of the grains neighbouring \overline{g} is constructed;
- for each element in Φ , the set of all the possible misorientations Ω_{Φ} is generated using the method exposed in the preceding section;
- the misorientations between experimental measurement in the initial and final states are computed as described in section 3.3.1. They are converted to a quaternion representation. The norm of these quaternions are compared with those of the elements in Ω_{Φ} ;
- only the element that yields the best match is kept.

This methodology is applied to a reduced zone of the studied area. This zone is represented in Figure 3.19 together with the associated initial and final EBSD mappings.



Figure 3.19: Zone of interest with a representation of the initial orientations in which orientations are averaged inside the grains and a representation of the final orientations.

Figure 3.20 depicts the error between the theoretical and measured quaternion norms. The overall agreement between the two is good, which makes the chosen transformation paths plausible. However, in some zones the error exceeds 10% which indicates that the material followed other transformation paths. Either no OR was preserved during the ferrite-to-austenite transformation or the transformation from austenite-to-ferrite was partly diffusive instead of being only displacive. In any case, only the points where the error is inferior to 5% are considered in what follows.



Figure 3.20: Error between theoretical and measured angular differences.

First, the associated KS variants for the austenite phase are shown in Figure 3.21a. In some regions, the prediction is noisy which indicates a situation in which several orientations are plausible for the austenite phase. This may be due to the fact that no proximity criterion is imposed when considering the orientations of the neighbours of a grain. The microstructure prediction is smoother when considering the Bain group the KS variants belong to instead of the KS variants themselves, as shown in Figure 3.21b. The Bain group A corresponds to variants that formed via a compression of the parent lattice along the [100] axis. For the Bain groups B and C, this compression was along the [010] and [001] axes respectively.



(a) KS variants in the austenite phase deduced from the initial and final EBSD maps



(b) Bain group the KS variants are associated with



Main effects of the transformation on the microstructure

The prediction of the austenite microstructure confirms the formation of large grains that possess an elongation in the direction of the temperature gradient. Grains grew from the right to the left of the image and got impinged at certain grain boundaries which accounts for the fact that the austenite microstructure conforms to the initial ferrite microstructure at certain grain boundaries. Upon cooling, the austenite microstructure either kept the same morphology or decomposed into sets of self-accomodating variants, as it is the case in the bottom right-hand side of Figure 3.21b.

3.3.3 Austenized samples

Initial microstructure

Initial grain orientations for the A1 sample are shown in Figure 3.22. They are colorized according to the given Inverse Pole Figure.



Figure 3.22: Grains orientations as obtained from EBSD measurements (A1 sample). Grains are colorized according to the adjacent key Inverse Pole Figure.

The microstructure is different from the one in section 3.3.1. It mixes very large and elongated grains with a number of small circular grains. As a result, the grain sizes histogram shown in Figure 3.23 is less regular than before.


Figure 3.23: Histogram of equivalent grains diameters in the studied area.

Max shear strain field evolution during the ferrite-to-austenite transformation

Correlation computations are made on a grid with cells of 51 pixels per 51 pixels. The evolution of the MSS is computed and displayed in Figure 3.24. The initial microstructure is superimposed on the correlation results. The different steps of the transformation process are retrieved: initiation, consitution of a front, propagation, reverse transformation.

Obtained microstructure

The obtained microstructure after testing is presented in Figure 3.25. The conclusions that can be drawn from it are the same as before:

- the testing procedure induced a change in the texture of the sample;
- there is a lot of intragranular misorientations that are a marker of plastic activity;
- grains are elongated in the zone where the front propagates and much smaller in the zone where fronts join.

In comparison with the final microstructure of recrystallized samples, what can also be seen in Figure 3.25 is that the final grains are coarser. This means that there have been less nucleation events. One possible explanation is that the small grains in the initial microstructure were much more thermodynamically favourable than other possible nucleation sites such as large grain edges and faces.

Partial conclusion

Conducting tests on both R and A samples brings the possibility to study the influence of the initial microstructure on the material response during a full transformation cycle. Compared to the samples tested in section 3.3.1, austenized samples had the largest grains but a higher disparity in grain sizes. The small grains in the microstructures acted as preferential sites for nucleation. A proof that they have been often favored is that the final microstructure is also coarser, which indicates that there have been few nucleation events in the bulk of the large grains.

The interpretation of the resulting strain field is difficult because the size of the small grains is of the same order of magnitude as image resolution. However, the MSS field exhibits the same behaviour as in section 3.3.1, which underlines that the experimental bench is suited for any initial microstructure.



Figure 3.24: Maximum shear strain evolution with temperature variations (in percent). The size of the domain of study is 7.0 mm per 3.8 mm.

3.4 Results of heating and cooling tests on deformed samples

3.4.1 Introduction of plastic deformation

The tensile solicitation is applied using an MTS 100kN testing machine. A speckle pattern is deposited on the samples in the same way as in 3.1.4. The load is applied step-by-step by considering small increments of displacement and stopping the tests once the desired strain is attained.

Two types of samples are tested: R and SA samples. SA samples are mechanically polished and etched before testing. Micrographies are performed and grain boundaries are extracted from these images. They are superimposed in red on the correlation results in Figure 3.26. The registration of the images is of course not as precise as when EBSD mappings are used. Besides, it is hard to tell whether small grain are present in the microstructure.



Figure 3.25: Grains orientations as obtained from final EBSD measurements (A1 sample). Grains are colorized according to the adjacent key Inverse Pole Figure.

The longitudinal strain values in the sample after tensile loading and elastic springback are displayed in Figure 3.26 for the R3 and SA2 samples. The heterogeneity of these strain fields reflects the polycrystalline character of the samples. The R3 and SA2 were selected because they exhibit a strain maximum near their center. The ferrite-to-austenite transformation should then initiate here.

3.4.2 Results obtained for strain annealed samples

Max shear strain field evolution

The post-processing is the same for pre deformed samples and non deformed samples. Tests on non deformed samples are also reported here in order to offer a direct comparison between corresponding samples. In the case of strain annealed samples, the reference is the SA1 sample. The evolution of the MSS around the ferrite-to-austenite transformation temperature for this sample is shown in Figure 3.27. In parallel, the evolution of the MSS for the SA2 sample is represented in Figure 3.28. The initial microstructures, extracted from the micrographies, are superimposed on the results in red.

Just like for the non deformed samples, Figure 3.28 underlines a link between the location of localization points and the underlying microstructure. Besides, the Figure shows that, as expected, the transformation started in the zone of the sample that was the most deformed.

Influence of a prior deformation on the transformation behaviour

In the case of the pre deformed sample, the localizations are fewer and further between than in the non deformed sample. This is surprising given that as recalled in section 1.4.2, the number of potential nucleation sites increases with the level of deformation of the material. This assumption cannot be verified but it is certain that all nucleation sites are concentrated near certain triple points. The pre deformation has increased the mechanical energy locally stored from defects in these areas and has made them much more favourable nucleation sites. Moreover, the gradient of strain is smoother and the MSS decreases slowly along grain boundaries. A possible explanation for this observation is that pre deformation accentuates misfit at grain boundaries. Consequently, it induces an anisotropic grain growth where newly formed grains grow first along grain boundaries then in the bulk of the grains.



R3 sample

Figure 3.26: Longitudinal strain fields after tensile loading.



Figure 3.27: MSS evolution for the **non deformed** SA1 sample around the ferrite-to-austenite transformation temperature (in percent).



Figure 3.28: MSS evolution for the **pre deformed** SA2 sample around the ferrite-to-austenite transformation temperature (in percent).

For the non deformed samples, the MSS field was almost homogeneous during the front propagation phase. This was due to the growth rate being superior to the nucleation rate. The MSS was then homogeneous as a result of a uniform growth of the austenite grains as the transformation front advanced. Conversely, for the pre deformed samples, the MSS field in the front propagation zone exhibits several localizations. The introduction of plastic deformation modifies the balance between nucleation and growth so that more austenite grains can nucleate when the front advances.

3.4.3 Results obtained for recrystallized samples

A similar analysis can be carried out for recrystallized samples. In that case, the reference sample is the R2 sample. The evolution of the MSS around the transformation temperature is displayed in Figure 3.29. The evolution of the MSS for the R3 sample is shown in Figure 3.30. The same differences can be spot between non deformed and pre deformed samples as in section 3.4.2:

- there are less strain localizations for the pre-deformed samples;
- strain localizations are more spread;
- the MSS field during propagation is less homogeneous and localization points are clearly perceptible.

Since the same conclusions can be drawn for both types of samples, it can be said that the phenomena presented in section 3.4.2 are caused by the pre deformation and not by the microstructure.

CHAPTER 3



Figure 3.29: MSS evolution for the reference **non deformed** R2 sample around the ferrite-toaustenite transformation temperature (in percent).



Figure 3.30: MSS evolution for the **pre deformed** R3 sample around the ferrite-to-austenite transformation temperature (in percent).

3.5 Conclusion on the experimental work

3.5.1 Viability of the method

The results exposed in this chapter put to fore that the experimental equipment is able to capture variations in the kinematic fields during allotropic transformations. One can take advantage of these variations to characterize the onset of the transformation provided the right strain-related quantity is chosen. In this work, MSS is shown to be a good indicator of occurring transformations. MSS evolution is not dependent on the shape of the initial microstructure: small grains, large flat grains or large equiaxed grains all lead to similar kinematic fields. Yet, the larger the microstructure, the easier it is to propose an interpretation for the observed phenomena.

3.5.2 Results highlights

The principal conclusions of the experimental work are the following:

- the evolution of the shear strain field can be used to track the ferrite-to-austenite and austenite-to-ferrite transformations in high purity iron. In the case of the ferrite-toaustenite transformation, the appearance of a germ of the new phase is betrayed by strain localizations;
- 2. the presence of a temperature gradient modifies the transformational behaviour of the material. In particular, in conditions where an incubation time is not preserved for austenite to nucleate, growth events are favoured over nucleation events;
- 3. a simple transformation path can be inferred from the comparison between the theoretical and numerical orientation evolutions of a material point. The model lies on the hypothesis that the dominant nature of the ferrite-to-austenite transformation is diffusive (with an Orientation Relationship being preserved between a product austenite grain and a parent grain) whereas that of the austenite-to-ferrite transformation is displacive. This method is valuable to characterize some transformation mechanisms, although it is unlikely that is describes the behaviour of all the grains;
- 4. deforming the sample in tension exacerbates the differences between potential nucleation sites in terms of their inclination to transformation. It encourages an anisotropic growth of the newly formed phase along grain boundaries and rebalances the competition between nucleation and growth rates.

3.5.3 Limitations of the experimental procedure

As stated in section 2.1, choice has been made to focus on the mechanical consequences of transformations, to the detriment of direct microstructural observations and phase fractions quantification. It is then only logical that the developed methodology suffers from the following flaws:

- during the ferrite-to-austenite transformation, the relative position of the two phases is known only through the appearance of strain localizations. Grains boundaries cannot be tracked with precision;
- it is not known at a given instant what the fraction of transformed material is.

These questions could be answered using some of the equipments described in section 2.1 but that would deviate from the initial goal of developing a simple and exhaustive tool for the study of allotropic transformations involving volume changes. A variational numerical tool will be developed in the following chapters to understand how phase fractions evolve during the ferrite-to-austenite and austenite-to-ferrite transformations.

Bibliography

- [1] Reinhard Pippan. Threshold and effective threshold of fatigue crack propagation in ARMCO iron I: The influence of grain size and cold working. page 13. 40
- [2] Vahid Afshari and Changiz Dehghanian. The influence of grain size of pure iron metal on corrosion inhibition in presence of Sodium nitrite. *International Journal of Modern Physics: Conference Series*, 05:793–800, January 2012. 40
- [3] Camillus Sunday Obayi, Ranna Tolouei, Afghany Mostavan, Carlo Paternoster, Stephane Turgeon, Boniface Adeleh Okorie, Daniel Oray Obikwelu, and Diego Mantovani. Effect of grain sizes on mechanical properties and biodegradation behavior of pure iron for cardiovascular stent application. *Biomatter*, 6(1):e959874, January 2016. 40
- [4] P.A. Jacquet. Electrolytic and chemical polishing. *Metallurgical reviews*, 1:157 238, 1956. 42
- [5] Florian Bachmann, Ralf Hielscher, and Helmut Schaeben. Grain detection from 2D and 3D EBSD data—Specification of the MTEX algorithm. *Ultramicroscopy*, 111(12):1720– 1733, December 2011. 43
- [6] W.B. Hutchinson. Recrystallisation textures in iron resulting from nucleation at grain boundaries. *Acta metallurgica*, 37:1047–1056, 1989. 44
- [7] A.J. Wilkinson. *Electron Backscatterd Diffraction in Materials Science*, chapter Measuring strains using electron backscattered diffraction. Springer, 2000. 50
- [8] J. Lee, K. Shibata, K. Asakura, and Y. Masumoto. Observation of the $\alpha \gamma$ transformation in ultralow-carbon steel under a high temperature optical microscope. *ISIJ International*, 42:1135–1143, 2002. 50
- [9] E. Bayraktar, D. Kaplan, L. Devillers, and J.P. Chevalier. Grain growth mechanism during the welding of interstitial free (IF) steels. *Journal of Materials Processing Technology*, 189(1-3):114–125, July 2007. 51
- [10] X. Chen, Y. Song, N. Tamura, and R.D. James. Determination of the stretch tensor for structural transformations. *Journal of the Mechanics and Physics of Solids*, 93:34 – 43, 2016. 53
- [11] A. Lew, K. Caspersen, E.A. Carter, and M. Ortiz. Quantum mechanics based multiscale modeling of stress-induced phase transformations in iron. *Journal of the Mechanics and Physics of Solids*, pages 1276–1303. 54
- [12] C. Denoual and A. Vattré. A phase field approach with a reaction pathways-based potential to model reconstructive martensitic transformations with a large number of variants. *Journal of the Mechanics and Physics of Solids*, 90:91–107, May 2016. 54
- [13] C.M. Wayman and H.K.D.H. Bhadeshia. Phase Transformations: Nondiffusive. In *Physical Metallurgy*, pages 1507–1554. Elsevier, 1996. 54
- [14] K. Koumatos and A. Muehlemann. A theoretical investigation of orientation relationships and transformation strains in steels. Acta Crystallographica Section A Foundations and Advances, 73(2):115–123, March 2017. 54, 55

-4

Coupled thermo-mechanical model for the mesoscopic transformational behavior of iron alloys

The main obstacle to further interpretation of the experimental results is the lack of knowledge on local dislocation densities. A modelling work is carried out to address this problem in an attempt to uncover the elementary mechanisms underlying the behaviours observed in the previous sections.

Several modelling philosophies can be considered depending on whether phase transformation is viewed as the problem of updating an interface, seeking equilibrium of a mixture of phases, or studying the behaviour of an out-of-equilibrium thermodynamic system. A variational methodology seems appropriate in view of the diversity of loadings and transformation mechanisms highlighted in the previous sections.

In this chapter, the emphasis is on the construction of a thermodynamically consistent continuous material point model. As these are critical elements for the representation of experimental phenomena, particular attention is paid to the choice of appropriate crystal plasticity laws and to the hypotheses relating to interactions between phases. In this respect, two forms of the model are constructed. The first one treats the formation of variants of the new phase as a way to reduce straining locally and is suited for displacive transformations. The second model forces the formation of the most energetically favourable phase, which resembles the response of the material during diffusive transformations.

4.1 Motivation for the development of a model

The goal of this section is to develop a numerical tool to complement the experimental observations made in the previous chapters. It should be able to meet the following requirements:

- to couple mechanical, thermal and transformational responses;
- to be simple enough to allow computations on experiment-sized numerical samples;

- to be suitable for the study of either the ferrite-to-austenite or the austenite-to-ferrite transformations in the conditions described in the previous chapters. In particular, it should provide a way to investigate both displacive and diffusive mechanisms;
- to offer a means to distinguish between transformed and untransformed areas.

In the frame of this work, an attempt will be made to satisfy these criteria by building an adaptative variational model. For reasons of simplicity, the model will be written under the small strain format. An extension to large deformations, notably taking into account material rotations, would only lead to a more detailed description of mechanical heterogeneities within the material.

4.2 Mesoscopic description of the thermo-mechanical behavior of iron

4.2.1 Basics of crystal plasticity

Deformation decomposition

It is admitted that the mechanical transformation consists of two fully distinct phenomena: a purely elastic part linked to the distortion of the atomic network, and a plastic part associated with lattice gliding. This theory is called continuum slip theory [1]. The linear strain tensor $\underline{\epsilon}$ is additively decomposed into elastic and plastic parts:

$$\underline{\boldsymbol{\epsilon}} = \underline{\boldsymbol{\epsilon}}_{\underline{\boldsymbol{e}}} + \boldsymbol{\epsilon}_{p}. \tag{4.1}$$

Discretization of plastic behavior

Crystal plasticity theory is based on the idea that plastic deformation can be assimilated to the discrete summation of the activity of dislocations on all single slip systems [2]. Let us identify slip systems through the normal to the gliding plane n_{α} and the slip direction inside the plane, m_{α} (where α subscript refers to a slip system). The Schmid tensor for the system α is $\underline{P_{\alpha}} = sym(n_{\alpha} \otimes m_{\alpha})$. Dislocations move on the slip system α under the influence of a resolved shear stress, which reads:

$$\tau_{\alpha} = \underline{\boldsymbol{\sigma}} : \boldsymbol{P}_{\alpha}. \tag{4.2}$$

The rate of plastic strain is then expressed as:

$$\underline{\dot{\boldsymbol{\epsilon}_p}} = \sum_{\alpha} \dot{\gamma_{\alpha}} \underline{\boldsymbol{P_{\alpha}}}.$$
(4.3)

Link between slips and dislocations densities

Let us consider the example of a single edge dislocation, with Burgers vector of norm b, that travels a distance x on a slip plane. Its contribution to total displacement can be evaluated as:

$$u_{dislo} = \frac{bx}{l_s},\tag{4.4}$$

where l_s is the length available on the gliding plane for the displacement of the dislocation. For n dislocations, the associated displacement is:

$$u_{tot} = n \frac{b\bar{x}}{l_s}.$$
(4.5)

Noting d_s the distance between two slip planes, one gets:

$$\gamma = \frac{u_{tot}}{d_s} = \frac{nb\bar{x}}{l_s d_s}.$$
(4.6)

 $\frac{n}{l_s d_s}$ physically represents a density of mobile dislocations ρ_m . In rate form, the Orowan equation, that relates the plastic slip to dislocation densities, is then obtained:

$$\dot{\gamma} = \rho_m b \bar{v},\tag{4.7}$$

where \bar{v} is the mean velocity of moving dislocations.

Hardening

A hardening law is required to describe to what extent an increase in the resolved shear stress induces plastic slip. It relates the plastic slip rate and the resolved shear stress rate. The first expressions for the hardening were of the type:

$$\dot{\tau_{\alpha}} = \sum_{\beta} H_{\alpha\beta} \dot{\gamma_{\beta}}, \tag{4.8}$$

where \underline{H} is the hardening matrix. Taylor suggested an expression in which the hardening on all systems is the same and has a constant value:

$$H_{\alpha\beta} = h \quad \forall \alpha, \, \forall \beta. \tag{4.9}$$

This relation was then put into a more common form that takes into account latent hardening [1]:

$$h_{\alpha\beta} = h \left(q + (1 - q)\delta_{\alpha\beta} \right), \tag{4.10}$$

with δ the Kronecker symbol. q is a constant that adjusts the respective influence of dislocations on the same system and on different slip systems.

However, this type of expression does not give an explicit view on the activity of dislocations inside each slip system. This is why formulations based on dislocation densities are often preferred. Dislocation densities on each slip system are introduced as new internal variables. The models are based on the hypothesis that the contributions of the mechanisms impeding the movement of dislocations can be added together. The critical shear stress is then decomposed into two parts:

$$\tau_{c_{\alpha}} = \tau_{P_{\alpha}} + \tau_{f_{\alpha}}.\tag{4.11}$$

 $\tau_{P_{\alpha}}$ is related to the Peierls friction on the lattice network. The dislocation interacts with the bed of atoms it lies in when it glides on a plane, even in the absence of other dislocations. The evolution of the potential energy of the dislocation is schematized in Figure 4.1. The dislocation tends to maintain itself in a low energy position. It needs a shear stress $\tau_{P_{\alpha}}$ to move towards another local minimum of the potential energy. The value of the Peierls stress may depend on



Figure 4.1: Schematic illustration of the Peierls energy barrier.

the magnitude of slip on the different systems [3] but this dependence is not taken into account. Peierls mechanim is purely dissipative.

The second term in equation (4.11) is related to the so-called forest mechanism, *i.e.* dislocations interactions inside the slip systems. A moving dislocation can be pinned by other dislocations. An additional shear stress is needed for the movement of the dislocation to continue. The forest mechanism can be considered athermal, unlike the Peierls mechanism.

The yield surface for crystal plasticity is obtained from equation (4.11) by considering the set of equations:

$$\tau_{c_{\alpha}} - \tau_{P_{\alpha}} - \tau_{f_{\alpha}} \le 0 \quad \forall \alpha.$$
(4.12)

As dislocations densities were introduced as variables, a law for their evolution is added in order to close the problem. The hardening laws for the BCC and FCC phases used in the frame of this work will be described in the rest of this section.

4.2.2 BCC phase behavior

The model for the behaviour of the BCC phase is based on the work by Stainier et al. [4].

Peierls barrier

The values of the Peierls stress are high in BCC crystal. It is rare that an entire dislocation line manages to move at a given time. Rather, only some segments of this line jump towards another local minimum of the potential energy. This mechanism is called double kink and is illustrated in Figure 4.2.



Figure 4.2: Double kink mechanism.

The rate limiting mechanism with respect to the Peierls barrier in BCC crystals is the formation of kinks along screw segments, for which the driving force is:

$$\Delta G_k = E_k - b\tau L_k l_p, \tag{4.13}$$

where τ is the resolved shear stress applied on the slip system, E_k is the energy required for the formation of a kink, l_p is the spatial period of the lattice and L_k is the length of the kink segment. The latter can be determined in the frame of the transition state theory by considering that the kink forms by random jumps of dislocation segments. Making use of the Orowan equation, the shear stress τ_P required for the dislocation to overcome the lattice friction is:

$$\tau_P = \frac{\tau_0}{\beta E_k} asinh\left(\frac{\dot{\gamma}}{\dot{\gamma}_{k_0}} e^{\beta E_k}\right),\tag{4.14}$$

where $\beta = \frac{1}{k_B T}$ and k_B is the Boltzmann constant.

Forest mechanism

Dislocations impinge other dislocations motion. The critical shear stress associated with this mechanism is determined using statistical considerations because the interactions between dislocations are random in nature. The probability density to encounter an obstacle is [5]:

$$\tilde{f}_{0}^{\alpha}(r,t) = 2\pi n^{\alpha} r e^{-\Pi n^{\alpha} r^{2}},$$
(4.15)

where n^{α} is a surface density of obstacles. It is expressed as $n^{\alpha} = \sum_{\beta \neq \alpha} n^{\alpha\beta}(t)$ where $n^{\alpha\beta}$ is the number of obstacles on the system α that belong to the system β . Using geometrical considerations, it is computed using the expression:

$$n^{\alpha\beta}(t) = a_0 \frac{2}{\pi} \sqrt{1 - (\boldsymbol{m}_{\alpha} \cdot \boldsymbol{m}_{\beta})^2} \rho^{\beta}, \qquad (4.16)$$

where a_0 is a constant efficiency factor.

Equation (4.15) can be transformed into a probability of finding an obstacle of strength s by resorting to micromechanical arguments:

$$\tilde{f}_0^{\alpha}(s,t) = \frac{4U_{edge}\sqrt{n^{\alpha}}}{v(s-\tau_{\alpha})^2} exp\left(-\frac{\Pi n^{\alpha}}{b^2}\left(\frac{2U_{edge}}{s-\tau_{\alpha}}\right)^2\right).$$
(4.17)

This expression is only valid when the obstacles in the forest are infinitely rigid. A correction to account for penetration between the dislocations is:

$$\tilde{f}^{\alpha}(s,t) = \sum_{\beta \neq \alpha} p^{\alpha\beta}(t) \tilde{f}^{\alpha}(s|s^{\alpha\beta},t), \qquad (4.18)$$

where $p^{\alpha\beta}$ is the probability that the weakest obstacle on the system α comes from the system β . Finally, by assessing the dislocation density that is released when τ is increased and supposing that all obstacles have the same strength s_0 , Stainier et al. [4] derive an hardening law in the form:

$$\dot{\gamma_{\alpha}} = \frac{\tau_{f_{\alpha}}}{H_{\alpha\alpha}}.\tag{4.19}$$

where

$$H_{\alpha\alpha} = \frac{1}{\gamma_{c_{\alpha}}} \frac{\left[1 - \tilde{P}_{\alpha}(\tau_{\alpha}(t), t)\right]^{2}}{\tilde{f}_{\alpha}(\tau_{\alpha}(t), t)}.$$
(4.20)

 \tilde{P}_{α} is the probability distribution associated with \tilde{f}_{α} and γ_{α_c} is a characteristic plastic strain. The advantage of this expression is that the matrix \underline{H} is diagonal, even though it accounts for the interactions between slip systems. This improves computational efficiency.

Evolution law for the densities of dislocations

Concerning dislocation evolution, several aspects are considered: production of dislocations by fixed sources like Frank-Read sources, breeding by cross-slip and annihilation. The rate of the formation of new dislocations is expressed as:

$$b\dot{\rho_{\alpha}} = \lambda \sqrt{\rho_{\alpha}} \dot{\gamma_{\alpha}}, \tag{4.21}$$

where λ accounts for both cross slip and multiplication of dislocations by Frank-Read sources. The annihilation law is:

$$b\dot{\rho_{\alpha}} = -\kappa \rho_{\alpha} \dot{\gamma_{\alpha}},\tag{4.22}$$

where κ represents the characteristic distance associated with the annihilation of dislocations.

Other hardening laws for BCC crystals

Models dealing with BCC crystals differ mainly by the way micro mechanisms are coupled. For instance, in [6], the athermal part of hardening (forest mechanism) is dealt with in a fashion similar to what will be been presented for FCC crytals (see section 4.2.3). Concerning the temperature dependent part, two regimes are considered:

- A first regime when kinks are fully formed and the driving physics is elastic interactions between those kinks.
- A second regime when kinks cannot form totally, i.e. the next Peierls valley is not reached by the segment of dislocation involved in the kink. Line tensions models are then adopted.

Finally, shear stress is calculated as:

$$\tau_{\alpha} = \min\left(\tau_{elastic\ interactions}, \tau_{line\ tension}\right) + \tau_{obstacles}.$$
(4.23)

Another approach is that of Monnet et al. [7]. Two regimes are also distinguished:

- at low temperature, the Peierls friction mechanism is dominant;
- at higher temperatures comes to play the jog dragging mechanisms. Part of the jogs that are not aligned with the dislocation line lag behind the dislocation as it advances. This phenomenon tends to create punctual defects that then interact with the other dislocations.

A slip rate law is build for each mechanism. The mixture law that is adopted to combine both effects is:

$$\frac{1}{\dot{\gamma_{\alpha}}} = \frac{1}{\gamma_{drag_{\alpha}}} + \frac{1}{\gamma_{P_{\alpha}}}.$$
(4.24)

4.2.3 FCC phase behavior

Hardening law

Another modelling approach is developed for the FCC phase owing to the difficulty of finding micromechanical coefficients for FCC crystals. The Peierls stress is smaller in FCC crystals than in BCC crystals. As a matter of consequence, $\tau_{P_{\alpha}}$ is supposed to be constant on all systems. A classical law is chosen for the evolution of the shear stress with dislocation densities under the influence of the forest mechanism:

$$\tau_{f_{\alpha}} = \tau_{f_0} + \mu b \sqrt{\sum_{\beta} a_{\alpha\beta} \rho_{\beta}}.$$
(4.25)

 \underline{a} is an interaction matrix that accounts for the cooperation and competition between dislocations. Its coefficient may be determined experimentally [8] or through Discrete Dislocation Dynamics simulations [9].

Concerning the evolution of dislocations, the equation proposed by Kocks [10] is here used:

$$\dot{\rho_j} = \frac{1}{b} \left(\frac{1}{L_j} - 2y_c \rho_j \right) |\dot{\gamma_j}|, \qquad (4.26)$$

where y_c is related to the annihilation of dislocations and L is the mean free path of dislocations. It is expressed as:

$$L_j = \frac{K}{\sqrt{\sum_i \rho_{ij}}}.$$
(4.27)

Kocks law embodies a competition between the storage and the annihilation of dislocations. Examples of its application to crystal plasticity simulations are found in [11] or [12]. Some improvements were also suggested for the model. Lu et al. [13] introduce the mean grain diameter D_{grain} to account for a grain size effect:

$$\dot{\rho_j} = \frac{1}{b} \left(\frac{1}{D_{grain}} + \frac{1}{L_j} - 2y_c \rho_j \right) |\dot{\gamma_j}|.$$
(4.28)

Interaction coefficients for CP simulations

Running simulations with the right interaction coefficients is of crucial importance for the reliability of the results on the mechanical response of the material. Following Franciosi and Zaoui [14], the interactions between dislocations can be decomposed into four categories: interactions between dislocations on the same slip system (also called self-hardening), intersection between coplanar dislocations, intersection between collinear dislocations and dislocations between other dislocations. The latter type leads to the formation of three types of junctions: glissile, Hirth or Lomer-Cottrell junctions. Six coefficients are required in total to characterize the crystal plasticity of FCC crystals.

We adopt the Schmid and Boas denomination of the slip systems. Following Madec [15], the interaction matrix has the form shown in Table 4.1.

The values of the corresponding interaction coefficients are also taken from [15] and are listed in Table 4.2.

THERMOMECHANICAL MODEL

	A2	A3	A6	B2	B4	B5	C1	C3	C5	D1	D4	D6
A2	a_{SH}	a_{copl}	a_{copl}	a_{colli}	a_{gliss}	a_{gliss}	a_{Hirth}	a_{gliss}	a_{Lomer}	a_{Hirth}	a_{Lomer}	a_{gliss}
A3	a_{copl}	a_{SH}	a_{copl}	a_{gliss}	a_{Hirth}	a_{Lomer}	a_{gliss}	a_{colli}	a_{gliss}	a_{Lomer}	a_{Hirth}	a_{gliss}
A6	a_{copl}	a_{copl}	a_{SH}	a_{gliss}	a_{Lomer}	a_{Hirth}	a_{Lomer}	a_{gliss}	a_{Hirth}	a_{gliss}	a_{gliss}	a_{colli}
B2	a_{colli}	a_{gliss}	a_{gliss}	a_{SH}	a_{copl}	a_{copl}	a_{Hirth}	a_{Lomer}	a_{gliss}	a_{Hirth}	a_{gliss}	a_{Lomer}
B4	a_{gliss}	a_{Hirth}	a_{Lomer}	a_{copl}	a_{SH}	a_{copl}	a_{Lomer}	a_{Hirth}	a_{gliss}	a_{gliss}	a_{colli}	a_{gliss}
B5	a_{gliss}	a_{Lomer}	a_{Hirth}	a_{copl}	a_{copl}	a_{SH}	a_{gliss}	a_{gliss}	a_{colli}	a_{Lomer}	a_{gliss}	a_{Hirth}
C1	a_{Hirth}	a_{gliss}	a_{Lomer}	a_{Hirth}	a_{Lomer}	a_{gliss}	a_{SH}	a_{copl}	a_{copl}	a_{colli}	a_{gliss}	a_{gliss}
C3	a_{gliss}	a_{colli}	a_{gliss}	a_{Lomer}	a_{Hirth}	a_{gliss}	a_{copl}	a_{SH}	a_{copl}	a_{gliss}	a_{Hirth}	a_{Lomer}
C5	a_{Lomer}	a_{gliss}	a_{Hirth}	a_{gliss}	a_{gliss}	a_{colli}	a_{copl}	a_{copl}	a_{SH}	a_{gliss}	a_{Lomer}	a_{Hirth}
D1	a_{Hirth}	a_{Lomer}	a_{gliss}	a_{Hirth}	a_{gliss}	a_{Lomer}	a_{colli}	a_{gliss}	a_{gliss}	a_{SH}	a_{copl}	a_{copl}
D4	a_{Lomer}	a_{Hirth}	a_{gliss}	a_{gliss}	a_{colli}	a_{gliss}	a_{gliss}	a_{Hirth}	a_{Lomer}	a_{copl}	a_{SH}	a_{copl}
D6	a_{gliss}	a_{gliss}	a_{colli}	a_{Lomer}	a_{gliss}	a_{Hirth}	a_{gliss}	a_{Lomer}	a_{Hirth}	a_{copl}	a_{copl}	a_{SH}

Table 4.1: Interaction matrix for the FCC phase.

a_{Hirth}	a_{gliss}	a_{Lomer}	a_{SH}	a_{copla}	a_{colli}
0.05	0.095	0.069	0.023	0.009	0.55

Table 4.2: Interaction coefficients for the FCC phase.

4.3 Dealing with phase changes in mesoscopic models

The most natural method to compute mechanical behaviour of the sample at the mesoscale is crystal plasticity. It is often used in modelling works dealing with phase changes, which then differ by the description of the phases and of the transformation mechanisms. Several strategies can be adopted when taking the transformation into account in a coupled model, just like several option were available for its experimental observation:

- one may focus on the geometry of the transforming phase by either defining an initial configuration beforehand and setting rules for its evolution or by updating the current phases repartition according to a known final microstructure;
- it is also possible to define functions to set the phase that exist at a material point at a given point and to deduce the interface that they implicitly define;
- finally, one may not consider any interface and build the material behaviour from a mixture of a response of the different phases.

4.3.1 Geometry-based approach

The transformation can be modelled by stochastic approaches after discretization of the domain of study. Xiao et al. [16] studied the influence of austenite deformation on the austenite-to-ferrite transformation. They coupled the Monte Carlo method and crystal plasticity. The phase transformation was simulated by means of probabilistic rules. Each cell of the model was assigned several parameters, namely orientation, order parameter and stored energy, that evolved depending on these rules.

Lan et al. [17] were also interested in austenite decomposition into ferrite. Evolution rules were implemented into a cellular automaton model. The transformed fraction in each cell was defined as the calculated transformed length divided by the geometric length of the cell. An et al. [18] used a cellular automaton to study the heating of an iron-carbon alloy. They were able to predict the partition of Carbon during the ferrite-to-austenite transformation and the

evolution of the fraction of austenite.

An original approach was proposed by Barbe et al. [19]. They studied the consequences of diffusive transformations on the local strain field. The morphology of the product phase was fixed and grown from random sites in a representative volume. The transformation evolves at a constant rate. The interface between the parent and product phases is updated at each step and the strain fields are updated in a region around that interface. A hydrostatic transformation strain is imposed at the interface to account for the change in compacity.

4.3.2 Phase-field methods

Phase field models are very often employed to model the evolution of micostructures. They basically consist in assigning an order parameter ϕ to each grain. These variables vary smoothly from 0 to 1 across interfaces and their evolution laws are derived from thermodynamic considerations. The free energy Ψ of the material includes an interface energy based on the gradient of ϕ to control the thickness of the interface. The evolution of ϕ generally follows the Ginzburg-Landau equation:

$$\dot{\phi} = -L\frac{\partial\Psi}{\partial\phi}.\tag{4.29}$$

Phase field models are not only able to capture the changes in microstructure but also the temporal evolution of the interface. Yamanaka et al. [20] coupled a crystal plasticity finite element method (CPFEM) with a multi-phase field method in which nucleation follows classical nucleation theory. Coupling between both methods was realized by introducing dislocation densities in the mechanical model. A stored internal energy was calculated from them and injected as a driving force for the transformation. Similarly, Mecozzi et al. [21] showed that it is possible to match experimental kinetic behaviours provided the right coefficients are chosen for the evolution of the order parameter.

4.3.3 Variational models

Another way to deal with a coupled problem is to consider that a material point represent the mean response of its surroundings. Several phases, characterized by their volume fraction, may coexist at a given point. This approach is more often used for displacive transformations. Sadjadpour et al. [22] proposed a model for the $\alpha - \epsilon$ transformation in iron under shear conditions in which total strain is expressed as:

$$\epsilon = \epsilon_e + \lambda \epsilon_m + \epsilon_p. \tag{4.30}$$

where ϵ_m is a hydrostatic contribution associated with the transformation. They expressed the total free energy of the thermodynamic system and evolution laws for the internal variables. Their 1D simulations gave accurate predictions for the dynamic response of pure iron.

The advantage of this framework is that it ensured that the obtained behaviours are consistent with the laws of thermodynamics. Besides, the internal variables must satisfy optimality conditions with respect to the total energy of the system. This gives the possibility to determine the increments of internal variables by using minimization algorithms. The aim of the next parts of this chapter will be to develop a coupled model in the variational framework for the behaviour of metallic materials undergoing phase changes. The previous chapter underlined that in the conditions of the experiments, both diffusive and displacive transformation mechanisms may occur during a ferrite-to-austenite-to-ferrite transformation cycle. Consequently, two adaptations of the model will be proposed for either interface-controlled transformations or coordinated transformations. Emphasis will first be put on the description of the mechanical behaviour of the system, then thermics and phase transformations will be incorporated in the thermodynamic framework.

4.4 Efficient coupling between physics through variational methodology

The driving idea is to rephrase the thermomechanical problem in the shape of a functional whose stationarity conditions are equivalent to the initial problem. The model is written under the small strain format.

4.4.1 Laws of thermodynamics

Thermodynamic framework is viewed as the most natural way to select state variables and to compute their evolution. In local form, the first principle of thermodynamics states that:

$$\rho \dot{e} = \underline{\boldsymbol{\sigma}} : \dot{\underline{\boldsymbol{\epsilon}}} + r - \nabla \boldsymbol{q}, \tag{4.31}$$

with e the internal energy of the material, r is a heat source and q the heat flux. The second principle of thermodynamics reads:

$$D_i = \rho T \dot{\eta} - r + T \nabla \left(\frac{q}{T}\right) \ge 0, \qquad (4.32)$$

where η is the entropy. Let us now introduce Helmholtz free energy $\Psi = e - T\eta$. Equation 4.32 may be rewritten to obtain the so-called Clausius-Duhem inequality:

$$D_i = \underline{\boldsymbol{\sigma}} : \underline{\dot{\boldsymbol{\epsilon}}} - \rho \dot{\Psi} - \rho \eta \dot{T} - \frac{\boldsymbol{q} \cdot \boldsymbol{\nabla} T}{T} \ge 0.$$
(4.33)

4.4.2 Generalized Standard Material description

The local state assumption is adopted, that is to say the state of the material can be fully described by strain, temperature, and a set of internal variables $\{\alpha_i\}$:

$$\Psi = \Psi(\underline{\boldsymbol{\epsilon}}, T, \{\alpha_i\}). \tag{4.34}$$

This leads to another form of equation 4.32:

$$\left(\underline{\boldsymbol{\sigma}} - \rho \frac{\tilde{\Psi}}{\underline{\boldsymbol{\epsilon}}}\right) : \underline{\dot{\boldsymbol{\epsilon}}} - \rho \sum_{i} \frac{\partial \tilde{\Psi}}{\partial \alpha_{i}} \dot{\alpha}_{i} - \rho \left(\frac{\partial \tilde{\Psi}}{\partial T} + \eta\right) \dot{T} - \frac{\boldsymbol{q} \cdot \boldsymbol{\nabla} T}{T} \ge 0.$$
(4.35)

This equation holds for any \dot{T} and for any $\dot{\underline{\epsilon}}$, which brings the conjugacy relation:

$$\eta = -\frac{\partial \dot{\Psi}}{\partial T}.\tag{4.36}$$

as well as:

$$\underline{\boldsymbol{\sigma}} = \rho \frac{\partial \Psi}{\partial \underline{\boldsymbol{\epsilon}}}.$$
(4.37)

Combining equations (4.31) and (4.35) it is possible to write the heat equation in entropy form:

$$\rho T \dot{\eta} = \rho \sum_{i} \frac{\partial \dot{\Psi}}{\partial \alpha_{i}} \dot{\alpha}_{i} + r - \nabla q.$$
(4.38)

Equation (4.35) puts to fore two kinds of dissipations, one that is the consequence of internal processes, $-\rho \sum_i \frac{\partial \Psi}{\partial \alpha_i} \cdot \dot{\alpha}_i$, and the other one related to thermal effects: $-\frac{q \cdot \nabla(T)}{T}$. Both have to be positive to comply with second principle.

Let us consider as a matter of example an athermal elastic behaviour. The only variable that describes the system is the elastic strain $\underline{\epsilon_e}$ which yields the equation:

$$\underline{\boldsymbol{\sigma}} = \rho \frac{\partial \tilde{\Psi}}{\partial \boldsymbol{\epsilon}_{\boldsymbol{e}}}.\tag{4.39}$$

4.4.3 Standard material hypothesis

More generally, a conjugate quantity (force) is defined for any internal variable in the same fashion as in equation (4.36): $A_i = -\rho \frac{\partial \tilde{\Psi}}{\partial \alpha_i}$. At this point, it is worth noting that the Helmholtz free energy only describes the relations between state variables and their conjugates. Laws for the evolution of conjugated variables are required when dissipative phenomena such as plasticity occur. A model is said to be standard when it is possible to define a potential Ω such that:

$$\dot{\boldsymbol{\alpha}} = \frac{\partial \Omega}{\partial \boldsymbol{A}}.\tag{4.40}$$

 Ω has to be convex, positive and zero at the origin to enforce the condition $A.\dot{\alpha} \ge 0$. Let us now define the Legendre transform of this potential:

$$\Omega^{\star} = \sup_{\dot{\boldsymbol{\alpha}}} (\boldsymbol{A} \dot{\boldsymbol{\alpha}} - \Omega(\boldsymbol{A})), \qquad (4.41)$$

so that we get the relation:

$$\boldsymbol{A} = \frac{\partial \Omega^{\star}}{\partial \dot{\boldsymbol{\alpha}}}.$$
(4.42)

4.4.4 Heat conduction potential

Let us define a quantity G such that:

$$G = -\frac{\nabla T}{T}.$$
(4.43)

The existence of a potential Ξ such that:

$$q = \frac{\partial \Xi}{\partial G} \tag{4.44}$$

can be supposed. A Biot dissipation potential is thus retrieved [23]. This construction allows the positivity of the product q.G to be verified.

4.5 Rephrasing of crystal plasticity models

4.5.1 Thermomechanical problem

The set of variables that is chosen to study the thermomechanical behaviour of a crystal is $\{\underline{\epsilon_e}, T, G, \gamma\}$, where γ is the vector of the slips on the different systems $\{\gamma_{\alpha}\}$. The evolution of plastic slips is ruled by a potential Φ such that:

$$\dot{\boldsymbol{\gamma}} = \frac{\partial \Phi}{\partial \boldsymbol{\tau}},\tag{4.45}$$

with $\tau = -\rho \frac{\partial \tilde{\Psi}}{\partial \gamma}$. τ derives from the pseudo-dissipation potential: $\tau = \frac{\partial \Phi^{\star}}{\partial \dot{\gamma}}$. Equation (4.35) can be reformulated as:

$$\underline{\boldsymbol{\sigma}}: \underline{\boldsymbol{\epsilon}_{p}} + \boldsymbol{\tau}. \dot{\boldsymbol{\gamma}} - \frac{\boldsymbol{q}. \boldsymbol{\nabla} T}{T} \ge 0.$$
(4.46)

An extension of the principle of maximum dissipation states that any admissible (*i.e.* satisfying equation (4.12)) stress state ($\underline{\sigma}, \tau$) compares with the equilibrium stress state ($\underline{\sigma}^{\diamond}, \tau^{\diamond}$) in accordance with:

$$(\underline{\boldsymbol{\sigma}}^{\diamond} - \underline{\boldsymbol{\sigma}}) : \underline{\boldsymbol{\epsilon}_{p}} + (\boldsymbol{\tau}^{\diamond} - \boldsymbol{\tau}) \cdot \dot{\boldsymbol{\gamma}} \ge 0.$$
(4.47)

It is supposed that heat conduction and internal dissipation are dissociated. A unified dissipation potential is then built:

$$D = \Phi^* - \Xi. \tag{4.48}$$

Two temperatures are distinguished: an external temperature T and an internal temperature Θ , defined by the relation $\Theta = \frac{\partial e}{\partial \eta}$. This distinction will come in handy in the following section. Let B be the domain of study. The equations of the coupled thermomechanical problem are:

1. $\nabla \cdot \underline{\sigma} + \rho \mathbf{b} = \mathbf{0}$ in B2. $\tau \cdot \dot{\gamma} - \rho T \dot{\eta} + r - \nabla q = 0$ in B3. $T = \Theta$ in B4. $\tau = \frac{\partial D}{\partial \dot{\gamma}}$ in B5. $u = u_0$ on ∂B_1 6. $\underline{\sigma} \cdot \mathbf{n} = \mathbf{F}$ on $\partial B \setminus \partial B_1$ 7. $T = T_0$ on ∂B_2 8. $\frac{\partial D}{\partial \mathbf{G}} \cdot \mathbf{n} = q_0$ on $\partial B \setminus \partial B_2$

4.5.2 Variational expression for the dissipation

Multiplying the previous equations by small admissible variations of the variables lead to equations that cannot be integrated into an energy potential. A solution exposed in [24] is to perform a time rescaling of kinetic equations:

$$dt \to \frac{dt}{\zeta(T,\Theta)},$$
 (4.49)

so that $D = D(\zeta(T, \Theta)\dot{\gamma}, G, T, \gamma)$. After computations, Yang et al. [24] show that:

$$\zeta(T,\Theta) = \frac{T}{\Theta}.\tag{4.50}$$

4.5.3 Helmholtz free energy

Physics are coupled additively in the Helmholtz free energy. The total Helmholtz free energy reads:

$$\Psi(\underline{\boldsymbol{\epsilon}_{e}}, T, \boldsymbol{\gamma}) = \Psi_{e}(\underline{\boldsymbol{\epsilon}_{e}}, T) + \Psi_{p}(T, \boldsymbol{\gamma}) + \Psi_{th}(T) + \Psi_{coup}(\underline{\boldsymbol{\epsilon}_{e}}, T).$$
(4.51)

Elastic and plastic potentials

Equation (4.36) justifies writing elastic potential in the form:

$$\Psi_e = \frac{1}{2} \underline{\epsilon_e} : \underline{\underline{C_e}} : \underline{\underline{C_e}} : \underline{\epsilon_e}.$$
(4.52)

Besides, an expression for plastic potential has to be postulated. A quadratic form is employed in this work, as suggested for instance in [25]:

$$\Psi_p = \int_0^\gamma d\gamma_\alpha H_{\alpha\beta}\gamma_\beta,\tag{4.53}$$

where \underline{H} is the hardening matrix introduced in section 4.2.1. This matrix was explicitly constructed in the case of the BCC phase. For the FCC phase, combining the rate form of equation (4.25) with equation (4.26) yields:

$$H_{\alpha\beta} = \frac{a_{\alpha\beta}}{\sqrt{\underline{a}.\rho}} \left(\frac{\mu}{2L_{\beta}} - \mu b y_c \rho_{\beta}\right).$$
(4.54)

This construction lies on the hypothesis that only the friction mechanisms are dissipative in the mechanical behaviour of the crystals. The ratio between stored and dissipated energies could of course be adjusted to better reproduce experimental trends.

Thermal behaviour

A quadratic potential is introduced to account for the thermal behaviour:

$$\Psi_{th} = -C_v \frac{(T - T_0)^2}{2T_0},\tag{4.55}$$

where the thermal capacity of iron C_v and a reference temperature T_0 appear. No thermal deformation is added in the model, consistently with the use of a volumic thermal capacity. Rather, the term that encompasses the coupling between mechanics and thermics is introduced as:

$$\Psi_{coup} = -(T - T_0)\boldsymbol{\epsilon}_{\boldsymbol{e}} : \underline{\boldsymbol{\kappa}}(T), \tag{4.56}$$

where $\underline{\kappa}$ is the dilatation matrix. In the scope of this work, it does not exhibit any kind of anisotropy so it may be reduced to the form $\underline{\kappa} = \kappa \underline{C}(T) : \underline{I}$.

Finally, it should be noted that a temperature dependence of the tensor $\underline{\underline{C}}$ is introduced in the model to account for variations of material elastic properties with temperature.

4.5.4 Intrinsic dissipation

BCC phase The dissipation potential for the BCC phase is obtained from equation (4.14):

$$\Phi^{\star} = \tau_0 \dot{\gamma_{k_0}} \sum_{\alpha} \left(\frac{\dot{\gamma_{\alpha}}}{\dot{\gamma_{k_0}}} asinh\left(\frac{\dot{\gamma_{\alpha}}}{\dot{\gamma_{k_0}}}\right) + 1 - \sqrt{1 + \frac{\dot{\gamma_{\alpha}}}{\dot{\gamma_{k_0}}}} \right).$$
(4.57)

FCC phase A classical choice for the dissipation potential is made for the FCC phase:

$$\Phi^{\star} = \sum_{\alpha} \frac{m}{m+1} \tau_0 \dot{\gamma_0} \left(\frac{\dot{\gamma_\alpha}}{\dot{\gamma_0}}\right)^{\frac{m+1}{m}}, \qquad (4.58)$$

with m the parameter that rules rate dependency, τ_0 and $\dot{\gamma_0}$ are reference shear stress and slip rate respectively. It is equivalent to taking a power law for slip kinetics. When m tends towards infinity, the rate-independent potential is retrieved:

$$\Phi^{\star} = \sum_{\alpha} \tau_0 \dot{\gamma_{\alpha}}.$$
(4.59)

Taking into account dissipation, one is able to write a global power functional in the form:

$$\Pi(\underline{\boldsymbol{\epsilon}_{\boldsymbol{e}}},\boldsymbol{\gamma},T;\dot{\boldsymbol{\gamma}},\boldsymbol{G}) = \frac{d}{dt}\Psi(\underline{\boldsymbol{\epsilon}_{\boldsymbol{e}}},T,\boldsymbol{\gamma}) + \eta\dot{T} + \sup_{\underline{\boldsymbol{\sigma}},\boldsymbol{\tau}} D(T,\zeta\dot{\boldsymbol{\gamma}},\boldsymbol{G}),$$
(4.60)

where the supremum enforces the condition in equation (4.47) and hence the admissibility of the stress state [26].

4.6 Application to the case of metallic materials undergoing solid state phase changes

4.6.1 Main hypotheses

Let us consider the case where one reference parent phase transforms into N variants of a product phase. It is supposed that the state of transformation may be well represented by the volume fraction of the different phases $\mathbf{f} = (f_0, f_1, ..., f_N)$. f_0 is the volume fraction of the parent phase. $(f_1, ..., f_N)$ are the volume fractions of the product variants. It follows that $f_0 = 1 - \sum_{i=1,N} f_i$.

In what follows, we will use superscripts to designate the variant that is studied. For instance, Φ_e^j will be the Helmholtz elastic energy of the variant j and γ_{α}^k will be the slip on the system α of the variant k.

The set of internal variables is extended to be $\{f, \{\gamma^i\}_{i=1,N}\}$. The conjugate to the vector of transformed fractions is $d_f = -\rho \frac{\partial \tilde{\Psi}}{\partial f}$. The hypothesis is made that the kinetic evolution of f derive from a potential Ω such that $\dot{f} = \frac{\partial \Omega}{\partial d_f}$ and $d_f = \frac{\partial \Omega^*}{\partial \dot{f}}$.

As earlier, we suppose that we can dissociate the dissipation associated with the transformation and other sources of dissipation to write a unified kinematic pseudo-potential in the form:

$$D = \Omega^* + \Phi^* - \Xi. \tag{4.61}$$

Besides, a rescaling factor is applied to that dissipation to maintain the variational structure of the problem: $\Omega^*(\mathbf{\dot{f}}, T, f) \rightarrow \Omega^*(\zeta \mathbf{\dot{f}}, T, f)$.

4.6.2 Latent heat of transformation

The allotropic transformation of iron is a first order transformation: the partial derivative of the free energy with respect to temperature is discontinuous at the transition temperature. There is thus a latent heat of transformation involved in the process. A linearised expression is used in the frame of this work:

$$\Psi_{transfo} = L_T \frac{T_{trans} - T}{T_0},\tag{4.62}$$

where L_T is the latent heat and T_{trans} the temperature at which the transformation initiates.

4.6.3 Dissipation

A first approach to express the dissipation associated with the transformation is to consider a kinetic law as in [22]:

$$\dot{f}_i = \dot{f}_0 \left(\frac{d_{f_i} - d_{f_0}}{1 + d_{f_i} - d_{f_0}} \right)^{\frac{1}{2}},$$
(4.63)

where f_0 is a reference transformation rate, d_{f_0} is a reference value for the driving force conjugated to the transformed volume fraction. It represents a delay that would occur at the beginning of the transformation. The associated dissipation potential is:

$$\Omega^{\star} = \sum_{i} (d_{f_0} - 1)\dot{f}_i + \dot{f}_0 \, atanh\left(\frac{\dot{f}_i}{\dot{f}_0}\right). \tag{4.64}$$

A simple power-like dissipation is also investigated:

$$\Omega^{\star} = \sum_{i} \frac{p}{p+1} d_{f_0} \dot{f}_0 \left(\frac{\dot{f}_i}{\dot{f}_0}\right)^{\frac{p+1}{p}}, \qquad (4.65)$$

where p stands for the rate sensitivity of the transformation.

The difficulty that remains to be tackled is to chose a way to assess the thermomechanical contribution of each variant. Two approaches are presented in the frame of this work.

4.6.4 Law of mixtures on strains (SLM model)

Power functional expression

This formulation stems from a Reuss homogenization of the thermomechanical behaviour of the phases mixture. The volume fraction of each phase weights its inelastic strain:

$$\underline{\boldsymbol{\epsilon}} = \underline{\boldsymbol{\epsilon}}_{\underline{\boldsymbol{e}}} + \sum_{i=0}^{N} f_i \underline{\boldsymbol{\epsilon}}_{\underline{\boldsymbol{p}}}^{\ i} + \sum_{i=1}^{N} f_i \underline{\boldsymbol{\epsilon}}_{\underline{\boldsymbol{T}}}^{\ i}, \tag{4.66}$$

where $\underline{\epsilon_T}^i$ is the strain associated with the transformation from the parent phase to the *i*th variant. As this model relies on a law of mixtures expressed at the level of the elastic strain, it will be called SLM (Law of Mixtures on the elastic Strain) in what follows.

The total Helmholtz free energy and dissipation pseudo potential read:

$$\tilde{\Psi}(\underline{\boldsymbol{\epsilon}_{e}}, T, \boldsymbol{f}, \boldsymbol{\gamma}) = \Psi_{e}(\underline{\boldsymbol{\epsilon}_{e}}, T) + \Psi_{th}(T) + \Psi_{coup}(\underline{\boldsymbol{\epsilon}_{e}}, T) + \sum_{i=0}^{N} f_{i}\Psi_{p}^{i}(\boldsymbol{\gamma}^{i}, T) + \sum_{i=1}^{N} f_{i}\Psi_{transfo}^{i}(f_{i}, T)$$

$$(4.67)$$

$$D(\dot{\boldsymbol{f}}, \dot{\boldsymbol{\gamma}}, \boldsymbol{G}; T, \boldsymbol{f}) = -\Xi(\boldsymbol{G}) + \sum_{i=0}^{N} f_i \Phi^{\star^i}(\zeta \dot{\boldsymbol{\gamma}^i}, T) + \sum_{i=1}^{N} f_i \Omega^{\star^i}(\zeta \dot{f}_i, T).$$
(4.68)

Stationarity conditions

The total power functional is:

$$\Pi(\underline{\boldsymbol{\epsilon}_{\boldsymbol{e}}},\boldsymbol{f},\boldsymbol{\gamma},T;\dot{\boldsymbol{\gamma}},\dot{\boldsymbol{f}},\boldsymbol{G}) = \frac{d}{dt}\Psi(\underline{\boldsymbol{\epsilon}_{\boldsymbol{e}}},T,\boldsymbol{f},\boldsymbol{\gamma}) + \eta\dot{T} + \sup_{\underline{\boldsymbol{\sigma}},\boldsymbol{\tau}} D(\boldsymbol{f},T;\zeta\dot{\boldsymbol{\gamma}},\zeta\dot{\boldsymbol{f}},\boldsymbol{G}).$$
(4.69)

As stated above, the internal variables must satisfy the condition:

$$\Pi^{\diamond}(\underline{\boldsymbol{\epsilon}_{\boldsymbol{e}}},\boldsymbol{f}^{\diamond},\boldsymbol{\gamma}^{\diamond},T;\dot{\boldsymbol{\gamma}}^{\diamond},\dot{\boldsymbol{f}}^{\diamond},\boldsymbol{G}) = \inf_{\boldsymbol{f},\boldsymbol{\gamma}}\Pi(\underline{\boldsymbol{\epsilon}_{\boldsymbol{e}}},\boldsymbol{f},\boldsymbol{\gamma},T;\dot{\boldsymbol{\gamma}},\dot{\boldsymbol{f}},\boldsymbol{G}).$$
(4.70)

The Euler-Lagrange equation associated with the plastic slip γ_j^i is:

$$-f_i \underline{\boldsymbol{\sigma}} : \underline{\boldsymbol{P}_j^i} + f_i \sum_k H_{jk} \gamma_k^i + f_i \zeta \frac{\partial \Phi^\star}{\partial \gamma_j^i} = 0$$
(4.71)

$$\Leftrightarrow f_i(\tau^i_\alpha - \tau^i_{P_\alpha} - \zeta \tau^i_{f_\alpha}) = 0, \tag{4.72}$$

which is the yield criterion written for a single phase. This equation signifies that the plastic activity of each phase is independent from the other phases. This means in particular that there is no inheritance of dislocations.

The optimality condition for a transformed volume fraction $f_i(i > 0)$ is:

$$f_i \zeta d_{f_i} = \underline{\boldsymbol{\sigma}} : (\underline{\boldsymbol{\epsilon}_p}^i + \underline{\boldsymbol{\epsilon}_T}^i) - \Psi_p^i - \Psi_{transfo}^i - \int_0^t (\Phi^{\star^i} + \Omega^{\star^i}) dt = 0.$$
(4.73)

The term $\underline{\epsilon_p}^i + \underline{\epsilon_T}^i$ may be rewritten in the form $\underline{\epsilon} - \underline{\epsilon_{oth}}$ where $\underline{\epsilon_{oth}}$ encompasses the inelastic straining of the other phases. Consequently, it can be said that there is a competition between the reduction in the overall straining in the material and the formation of the new phase.

4.6.5 Law of mixtures on free energies (ELM model)

Power functional expression

This time, the elastic strain is independent for each phase:

$$\underline{\boldsymbol{\epsilon}_{e}}^{i} = \underline{\boldsymbol{\epsilon}} - \underline{\boldsymbol{\epsilon}_{p}}^{i} - \underline{\boldsymbol{\epsilon}_{T}}^{i}. \tag{4.74}$$

However, the Voigt bound for the behaviour of the multiphase material is considered. The total power functional is then:

$$\tilde{\Psi}(\underline{\boldsymbol{\epsilon}_{e}}, T, \boldsymbol{f}, \boldsymbol{\gamma}) = \Psi_{th}(T) + \sum_{i=1}^{N} f_{i} \Psi^{i}_{trans}(f_{i}, T) + \sum_{i=0}^{N} f_{i} \left(\Psi^{i}_{e}(\underline{\boldsymbol{\epsilon}_{e}^{i}}, T) + \Psi^{i}_{coup}(\underline{\boldsymbol{\epsilon}_{e}^{i}}, T) + \Psi^{i}_{p}(\boldsymbol{\gamma}^{i}, T) \right)$$
(4.75)

$$D(\dot{\boldsymbol{f}}, \dot{\boldsymbol{\gamma}}, \boldsymbol{G}; T, \boldsymbol{f}) = -\Xi(\boldsymbol{G}) + \sum_{i=0}^{N} f_i \Phi^{\star^i}(\dot{\boldsymbol{\gamma}^i}, T) + \sum_{i=1}^{N} f_i \Omega^{\star^i}(\dot{f}_i, T).$$
(4.76)

This is equivalent to considering a law of mixtures expressed on the free energy of the material. This justifies the name of the model, ELM (Law of Mixtures on the free Energy).

Stationarity conditions

The optimality condition with respect to a plastic slip γ_j^i is:

$$f_i(-\underline{P_j^i}:\underline{\underline{C}}:\underline{\underline{C}}:\underline{\underline{C}}^i+\tau_{P_j}^i+\tau_{f_j}^i)=0.$$
(4.77)

Again, one obtain a yield criterion for crystal plasticity provided the effective stress acting on a phase is considered to be $\underline{\underline{C}} : \underline{\epsilon_e}^i$.

Considering a transformed volume fraction $f_i(i > 0)$, we get:

$$f_i \zeta d_{f_i} = -\Psi_e^i - \Psi_p^i - \Psi_{transfo}^i - \int_0^t \left(\Phi^{\star^i} + \Omega^{\star^i} \right) dt.$$
(4.78)

The driving force for the transformation in only the total energy variation induced by its formation. It can be understood from this equation that only the phase that is the most energetically favourable will form at a given material point. This motivates the use of a simplex algorithm for the minimisation with respect to transformed volume fractions in the next chapter (see section 5.1.5).

4.6.6 Tangent modulus

Numerical implementation of materials models requires the computation of a tangent matrix \underline{M} such that ($\underline{\epsilon}$ and $\underline{\sigma}$ are written in Voigt notation):

$$\begin{pmatrix} \dot{\boldsymbol{\sigma}} \\ -\dot{\boldsymbol{\eta}} \end{pmatrix} = \underline{\boldsymbol{M}} \begin{pmatrix} \dot{\boldsymbol{\epsilon}} \\ \dot{\boldsymbol{T}} \end{pmatrix} = \begin{bmatrix} C_{\boldsymbol{\epsilon}} & C_{\boldsymbol{c}} \\ C_{\boldsymbol{c}} & C_{\boldsymbol{T}} \end{bmatrix} \begin{pmatrix} \dot{\boldsymbol{\epsilon}} \\ \dot{\boldsymbol{T}} \end{pmatrix}.$$
(4.79)

From Coleman's relations, one obtain:

$$\begin{cases} d\eta = -\frac{\partial^2 \Psi}{\partial T^2} dT - \frac{\partial^2 \Psi}{\partial T \partial \mathbf{X}} . d\mathbf{X} - \frac{\partial^2 \Psi}{\partial T \partial \underline{\epsilon}} : d\underline{\epsilon} \\ d\underline{\boldsymbol{\sigma}} = \frac{\partial^2 \Psi}{\partial \underline{\epsilon}^2} : d\underline{\epsilon} + \frac{\partial^2 \Psi}{\partial \underline{\epsilon} \partial \mathbf{X}} . d\mathbf{X} + \frac{\partial^2 \Psi}{\partial \underline{\epsilon} \partial T} dT, \end{cases}$$
(4.80)

where $X = (\gamma, f)$. It is necessary to know the differential of X in order to determine \underline{M} . Stationarity conditions with respect to internal variables enforce that:

$$\frac{\partial^2 \Psi}{\partial \mathbf{X}^2} d\mathbf{X} + \frac{\partial^2 \Psi}{\partial \mathbf{X} \partial \underline{\epsilon}} : d\underline{\epsilon} + \frac{\partial^2 \Psi}{\partial \mathbf{X} \partial T} dT = \mathbf{0}.$$
(4.81)

This allows the calculation of the quantities in equation (4.79):

$$\begin{cases} \underline{C}_{\epsilon} \equiv \underline{C}_{e} - \frac{\partial^{2}\Psi}{\partial\epsilon\partial \mathbf{X}} : \underline{\chi}^{-1} : \frac{\partial^{2}\Psi}{\partial\mathbf{X}\partial\epsilon} \\ \overline{C}_{c} \equiv \frac{\partial^{2}\Psi}{\partial T\partial\epsilon} - \frac{\partial^{2}\Psi}{\partial T\partial\mathbf{X}} : \underline{\chi}^{-1} : \frac{\partial^{2}\Psi}{\partial\mathbf{X}\partial\epsilon} \\ C_{T} \equiv \frac{\partial^{2}\Psi}{\partial T^{2}} - \frac{\partial^{2}\Psi}{\partial T\partial\mathbf{X}} : \underline{\chi}^{-1} : \frac{\partial^{2}\Psi}{\partial\mathbf{X}\partial T} \end{cases}$$
(4.82)

with $\underline{\chi} = \frac{\partial^2 \Psi}{\partial X^2}$.

4.7 Conclusion on the model formulation

Several approaches were presented for the coupling between thermomechanical and transformatonal behaviours in a metallic material. Most of them are not flexible enough to reproduce all the conditions encountered in the experimental part of this work. Henceforth, choice has been made to resort to a variational methodology.

The material is assimilated to a generalized standard medium. The Helmholtz free energy additively couples the physics by considering the relationship between internal variables and their conjugates. A dissipation potential rules the evolution of the internal variables. All dissipation processes are supposed to be independent.

Two versions of the model are derived. In the first case, the coupling between the behaviour of the phases is established at the level of the elastic strain, in agreement with previous works on martensitic transformations. This model is hoped to reproduce the behaviour of the material under displacive transformation conditions in which the phases form to minimize the overall straining. In the other case, the coupling is made at the level of the free energy in an attempt to better capture diffusive mechanisms. Indeed, only the most energetically favourable phase is authorized to form.

Bibliography

- Christian Miehe and Jorg Schroder. A comparative study of stress update algorithms for rate-independent and rate-dependent crystal plasticity. *International Journal for Numerical Methods in Engineering*, 50(2):273–298, January 2001. 68, 69
- [2] J.R. Rice. Inelastic constitutive relations for solids: An internal-variable theory and its application to metal plasticity. *Journal of the Mechanics and Physics of Solids*, 19(6):433– 455, November 1971. 68
- [3] M S Duesbery and V Vitek. Plastic anisotropy in B.C.C. transition metals. (128):12. 70
- [4] L Stainier. A micromechanical model of hardening, rate sensitivity and thermal softening in BCC single crystals. *Journal of the Mechanics and Physics of Solids*, 50(7):1511–1545, July 2002. 70, 71

- [5] A M Cuitino and M Ortiz. Computational modelling of single crystals. *Modelling and Simulation in Materials Science and Engineering*, 1(3):225–263, April 1993. 71
- [6] Hojun Lim, Corbett C. Battaile, Jay D. Carroll, Brad L. Boyce, and Christopher R. Weinberger. A physically based model of temperature and strain rate dependent yield in BCC metals: Implementation into crystal plasticity. *Journal of the Mechanics and Physics of Solids*, 74:80–96, January 2015. 72
- [7] Ghiath Monnet, Ludovic Vincent, and Benoit Devincre. Dislocation-dynamics based crystal plasticity law for the low- and high-temperature deformation regimes of bcc crystal. *Acta Materialia*, 61(16):6178–6190, September 2013. 72
- [8] P. Franciosi, L.T. Le, G. Monnet, C. Kahloun, and M.-H. Chavanne. Investigation of slip system activity in iron at room temperature by SEM and AFM in-situ tensile and compression tests of iron single crystals. *International Journal of Plasticity*, 65:226–249, February 2015. 73
- [9] Sylvain Queyreau, Ghiath Monnet, and Benoît Devincre. Slip systems interactions in αiron determined by dislocation dynamics simulations. *International Journal of Plasticity*, 25(2):361–377, February 2009. 73
- [10] U. F. Kocks. Laws for Work-Hardening and Low-Temperature Creep. Journal of Engineering Materials and Technology, 98(1):76, 1976. 73
- [11] A. Saai, H. Louche, L. Tabourot, and H.J. Chang. Experimental and numerical study of the thermo-mechanical behavior of Al bi-crystal in tension using full field measurements and micromechanical modeling. *Mechanics of Materials*, 42(3):275–292, March 2010. 73
- [12] M.G. Lee, H. Lim, B.L. Adams, J.P. Hirth, and R.H. Wagoner. A dislocation density-based single crystal constitutive equation. *International Journal of Plasticity*, 26(7):925–938, July 2010. 73
- [13] Jiawa Lu, Wei Sun, and Adib Becker. Material characterisation and finite element modelling of cyclic plasticity behaviour for 304 stainless steel using a crystal plasticity model. *International Journal of Mechanical Sciences*, 105:315–329, January 2016. 73
- [14] P. Franciosi and A. Zaoui. Multislip in f.c.c. crystals a theoretical approach compared with experimental data. Acta Metallurgica, 30(8):1627–1637, August 1982. 73
- [15] R. Madec. Des intersections entre dislocations à la plasticité du monocristal CFC : étude par dynamique des dislocations. Université Paris XI (thesis), 2001. 73
- [16] Namin Xiao, Mingming Tong, Yongjun Lan, Dianzhong Li, and Yiyi Li. Coupled simulation of the influence of austenite deformation on the subsequent isothermal austenite–ferrite transformation. *Acta Materialia*, 54(5):1265–1278, March 2006. 74
- [17] Y.J. Lan, N.M. Xiao, D.Z. Li, and Y.Y. Li. Mesoscale simulation of deformed austenite decomposition into ferrite by coupling a cellular automaton method with a crystal plasticity finite element model. *Acta Materialia*, 53(4):991–1003, February 2005. 74
- [18] Dong An, Shiyan Pan, Li Huang, Ting Dai, Bruce Krakauer, and Mingfang Zhu. Modeling of Ferrite-Austenite Phase Transformation Using a Cellular Automaton Model. *ISIJ International*, 54(2):422–429, 2014. 74

- [19] Fabrice Barbe, Romain Quey, Lakhdar Taleb, and Eduardo Souza de Cursi. Numerical modelling of the plasticity induced during diffusive transformation. An ensemble averaging approach for the case of random arrays of nuclei. *European Journal of Mechanics -A/Solids*, 27(6):1121–1139, November 2008. 75
- [20] Akinori Yamanaka, Tomohiro Takaki, and Yoshihiro Tomita. Simulation of Austenite-toferrite Transformation in Deformed Austenite by Crystal Plasticity Finite Element Method and Multi-phase-field Method. *ISIJ International*, 52(4):659–668, 2012. 75
- [21] M.G. Mecozzi, M. Militzer, J. Sietsma, and S. van der Zwaag. The Role of Nucleation Behavior in Phase-Field Simulations of the Austenite to Ferrite Transformation. *Metallurgical and Materials Transactions A*, 39(6):1237–1247, June 2008. 75
- [22] Amir Sadjadpour, Daniel Rittel, Guruswami Ravichandran, and Kaushik Bhattacharya. A model coupling plasticity and phase transformation with application to dynamic shear deformation of iron. *Mechanics of Materials*, 80:255–263, January 2015. 75, 81
- [23] M.A. Biot. Variational principles in irreversible thermodynamics with application to Viscoelasticity. *Physical Review*, 97:1463–1469, 1955. 77
- [24] Q Yang, L Stainier, and M Ortiz. A variational formulation of the coupled thermomechanical boundary-value problem for general dissipative solids. *Journal of the Mechanics and Physics of Solids*, 54(2):401–424, February 2006. 78
- [25] G. Cailletaud. Une introduction à la plasticité cristalline: interactions avec l'environnement. pages 81–115. EDP Sciences, 2009. 79
- [26] J. Mosler and O.T. Bruhns. On the implementation of rate-independent standard dissipative solids at finite strain – Variational constitutive updates. *Computer Methods in Applied Mechanics and Engineering*, 199(9-12):417–429, January 2010. 80

5

Numerical implementation of a variational model for the simulation of polycrystalline behavior with phase change

The objective of this part is to use the material point model developed in the previous chapter to obtain field data. Implementing it in a finite element code implies the discretization of the model over a time increment so as to solve the thermomechanical problem iteratively. Precautions are taken to maintain a thermodynamically consistent derivation, which leads to an efficient way to obtain plastic slips and other internal variables increments.

A monocrystalline domain is considered first to illustrate the features of the ELM and SLM models. The main disadvantage of both models is that in the absence of initial disparities in the mechanical state of the material, the whole domain transforms at once. Henceforth, it is necessary to introduce hetereogeneities to favour the transformation in certain zones. In the present work, this is achieved through deforming the numerical samples in tension. Two types of tests are investigated: continuous heating for the diffusive ferrite-to-austenite transformation and continuous cooling for the displacive austenite-to-ferrite transformation.

Computation cost has limited the size of the tested microstructures. A reference microstructure containing three grains is chosen to investigate the ability of the models to simulate transformation events. The onset of transformation and the modifications in the strain field it induces are fairly well described. However, the strain field after the transformation does not compare with what is seen experimentally. An attempt is made to simulate experimental microstructures. The experimental microstructures are simplified in order to keep affordable computation costs. Although the advancement of the transformation front is captured, the obtained strain fields after transformation are unsatisfying.

5.1 Discrete form of the material point model

5.1.1 Thermomechanical response of a polycrystal

As mentioned in section 1.1.2, a polycrystal is a set of grains, each possessing its own orientation and properties. Several methods can be employed to obtain the response of the polycrystal. One may, in order of complexity:

- consider only the shapes and orientations of the grains, disregarding their position in space. This philosophy echoes mean-field homogenization methods. They yield the mean response of a set of grains. However, they would fail at capturing strain localizations such as the one highlighted in the experimental part;
- consider the shapes and orientations of the grains and also their position in space. A geometrical representation of the sample is then required. In such a paradigm, grain boundaries are simple geometrical separations between domains in space. However, intragranular effects can be expected to be captured;
- consider the shapes and orientations of the grains, their position in space and come up with a specific representation of the interfaces. Grain boundaries are important defects in the material and a proper modelling of their physiognomy leads to a better representation of the heterogeneities in the polycrystal.

In the frame of the present work, only the first two ways are explored. No work is made on the treatment of grain boundaries because the combination of crystal plasticity and multivariants transformation already makes the computation long. It has to be kept in mind that this constitutes a limitation or at least an area for improvement of the modelling approach. Finite Element computations are performed using the Zorglib finite element library developed by Laurent Stainier at GeM Institute. A discretization of the material model is required to embed the material point model in the Finite Element code. It is performed through the integration of the power functional over a time increment.

5.1.2 Calculation over a time increment

Let us consider an evolution of the system between two instants t_n and t_{n+1} . A shift is performed on temperature for convenience: $\hat{T} = T - T_0$. The incremental form of equation 4.69 can be taken as a starting point:

$$\Pi_{inc} = \int_{t_n}^{t_{n+1}} (\frac{d}{dt} \Psi(\underline{\boldsymbol{\epsilon}}_{\underline{\boldsymbol{e}}}, \hat{T}, \boldsymbol{f}, \boldsymbol{\gamma}) + \eta \dot{T}) dt + \int_{t_n}^{t_{n+1}} D(\zeta \dot{\boldsymbol{f}}, \zeta \dot{\boldsymbol{\gamma}}, \boldsymbol{G}; \hat{T}(t), \boldsymbol{f}(t)) dt \qquad (5.1)$$

$$\Leftrightarrow \Pi_{inc} = \Psi_{n+1}(\underline{\boldsymbol{\epsilon}}_{e_{n+1}}, T_{n+1}, \boldsymbol{f}_{n+1}, \boldsymbol{\gamma}_{n+1}) - \Psi_{n}(\underline{\boldsymbol{\epsilon}}_{e_{n}}, T_{n}, \boldsymbol{f}_{n}, \boldsymbol{\gamma}_{n}) + \eta_{n}(T_{n+1} - T_{n})$$

$$+ \Delta t \left\langle D\left(\frac{T_{n+1}}{T_{n}}\frac{\Delta \boldsymbol{f}}{\Delta t}, \frac{T_{n+1}}{T_{n}}\frac{\Delta \boldsymbol{\gamma}}{\Delta t}, \boldsymbol{G}(\hat{T}_{n+1}, \hat{T}_{n}); \hat{T}(t), \boldsymbol{f}_{n+v}\right) \right\rangle_{t_{n}}^{t_{n+1}},$$

$$(5.2)$$

where Ψ_n designates the value of the Helmholtz free energy at time t_n , $\Delta t = t_{n+1} - t_n$ and <> is the average operator. v is a numerical parameter such that $\mathbf{f}_{n+v} = v\mathbf{f}_n + (1-v)\mathbf{f}_{n+1}$. v ranges between 0 and 1.

The optimality of the internal variables leads to the definition of:

$$\Pi_{inc}^{eff} = \inf_{\Delta \gamma, \Delta f} \Pi_{inc}.$$
(5.3)

The functional in equation (5.3) is used for the finite element implementation of the model. The nodal variables are the displacement and the temperature. They can be obtained through optimization of the total incremental energy functional:

$$\inf_{\Delta \boldsymbol{u}} \sup_{\hat{T}_{n+1}} \int_{B} \Pi_{inc}^{eff} dV + \int_{B} r \frac{T_{n+1}}{T_{0}} dV + \int_{\partial B \setminus \partial B_{1}} \rho \boldsymbol{F}_{\boldsymbol{0}n+1} \cdot \boldsymbol{\Delta \boldsymbol{u}} dS - \int_{\partial B \setminus \partial B_{2}} \Delta t \frac{T_{n+1}}{T_{0}} \boldsymbol{q}_{\boldsymbol{0}n+1} \cdot \boldsymbol{n} dS, \quad (5.4)$$

where the dependence in \boldsymbol{u} of the strain is implicitly assumed.

5.1.3 Consistent update of dissipation

A straightforward implementation of equation (5.2) would be to express the average of dissipation over a time step in the form:

$$\left\langle D\left(\frac{T_{n+1}}{T_n}\frac{\Delta \boldsymbol{f}}{\Delta t}, \frac{T_{n+1}}{T_n}\frac{\Delta \boldsymbol{\gamma}}{\Delta t}, \boldsymbol{G}; \hat{T}(t), \boldsymbol{f}_{n+\upsilon}\right) \right\rangle_{t_n}^{t_{n+1}} \simeq D\left(\frac{T_{n+1}}{T_n}\frac{\Delta \boldsymbol{f}}{\Delta t}, \frac{T_{n+1}}{T_n}\frac{\Delta \boldsymbol{\gamma}}{\Delta t}, \boldsymbol{G}; \hat{T}_{n+1}, \boldsymbol{f}_{n+\upsilon}\right).$$
(5.5)

However it has been proven that this expression leads to a non consistent formula for heat equation [1]. Henceforth, according to Stainier et al., the following expression has to be considered:

$$\left\langle D\left(\frac{T_{n+1}}{T_n}\frac{\Delta \boldsymbol{f}}{\Delta t}, \frac{T_{n+1}}{T_n}\frac{\Delta \boldsymbol{\gamma}}{\Delta t}, \boldsymbol{G}; \hat{T}(t), \boldsymbol{f}_{n+\upsilon}\right) \right\rangle_{t_n}^{t_{n+1}} \simeq \frac{T_n}{T_{n+1}} D\left(\frac{T_{n+1}}{T_n}\frac{\Delta \boldsymbol{f}}{\Delta t}, \frac{T_{n+1}}{T_n}\frac{\Delta \boldsymbol{\gamma}}{\Delta t}, \boldsymbol{G}; \hat{T}_n, \boldsymbol{f}_{n+\upsilon}\right) + \frac{\Delta T}{T_{n+1}} D\left(\frac{T_{n+1}}{T_n}\frac{\Delta \boldsymbol{f}}{\Delta t}, \frac{T_{n+1}}{T_n}\frac{\Delta \boldsymbol{\gamma}}{\Delta t}, \boldsymbol{G}; \hat{T}_{n+\upsilon}, \boldsymbol{f}_{n+\upsilon}\right).$$
(5.6)

Taking the derivative of equation (5.4) with respect to \hat{T}_{n+1} , the discrete form of the heat equation (4.38) is obtained:

$$\Delta \eta = \Delta t \left(\sum_{i=0}^{N} f_{i_{n+\nu}} \frac{\partial \Phi^{\star^{i}}}{\partial \hat{T}_{n+1}} \left(\frac{T_{n+1}}{T_{n}} \frac{\Delta \gamma}{\Delta t} \right) + \sum_{i=1}^{N} f_{i_{n+\nu}} \frac{\partial \Omega^{\star^{i}}}{\partial \hat{T}_{n+1}} \left(\frac{T_{n+1}}{T_{n}} \frac{\Delta f}{\Delta t} \right) \right) - \frac{\nabla q_{n+1}}{T_{n}} + \frac{r}{T_{n}}, \quad (5.7)$$

where we have used the relation $\eta_{n+1} = -\frac{\partial \Psi_{n+1}}{\partial \hat{T}_{n+1}}$.

5.1.4 Mechanical internal variables update

The example of the update of mechanical internal variables is chosen to underline the advantages of the expression (5.3). Indeed, it is well-known that the plastic slips cannot be uniquely determined from the rate-independent Schmid law. The addition of a rate dependence or a specific numerical treatment are required to reach convergence. Borja and Wren [2] came up with the so-called ultimate algorithm where systems are treated one by one. This algorithm was established for a hardening of the type presented in equation (4.10). Slip increments are determined at each iteration on the system that is the furthest from the yield surface:

$$\alpha = \underset{\text{systems }\beta}{\operatorname{argmin}} \{ \kappa(\beta) \in \mathbb{R}^+ \},$$
(5.8)

where the $\kappa(\beta)$ are calculated according to:

$$\underline{\underline{P}}_{\underline{\beta}} : (\underline{\underline{\sigma}}_n + \kappa(\beta) \underline{\underline{C}}_{\underline{\epsilon}} : \underline{\underline{\Delta}}_{\underline{\epsilon}}) - (\tau_n + \kappa(\beta) H_{\alpha\beta} \sum_{\gamma} \sum_{\delta} G^{-1}{}_{\gamma\delta} \underline{\underline{P}}_{\underline{\delta}} : \underline{\underline{C}}_{\underline{\epsilon}} : \underline{\underline{\Delta}}_{\underline{\epsilon}}) = 0, \quad (5.9)$$

with \underline{G} the Jacobian matrix associated with slips. The associated slip increment is then:

$$\Delta \gamma_{\alpha} = \kappa(\alpha) \sum_{\beta} G^{-1}{}_{\alpha\beta} \underline{P_{\beta}} : \underline{\underline{C^{ep}}} : \underline{\Delta \epsilon}$$
(5.10)

In this algorithm, the indeterminacy regarding slips is lifted through the order by which they are considered. This means that the numerical treatment of crystal plasticity influences the results, which is not desirable. Schmidt-Baldassari [3] explored two other possibilities to obtain plastic slips. The general idea is to explicitly enforce the yield criteria Γ_{α} in the principle of maximum dissipation. The two possibilities are:

- a penalized expression of the mechanical dissipation in the form:

$$\underline{\boldsymbol{\sigma}}: \underline{\boldsymbol{\epsilon}_{p}} + \boldsymbol{\tau}.\dot{\boldsymbol{\gamma}} + \frac{1}{2} \sum_{\alpha} (\max(0, \iota \Gamma_{\alpha}))^{2},$$
(5.11)

where ι is the penalty factor.

– an augmented Lagrangian approach. Positive variables z_{α} are introduced so that the yield criterion becomes $\tau_{\alpha} - \tau_{P_{\alpha}} - \tau_{f_{\alpha}} + z_{\alpha}^2 = 0$. The augmented Lagrangian associated with this formulation is:

$$\underline{\boldsymbol{\sigma}}: \underline{\dot{\boldsymbol{\epsilon}_p}} + \boldsymbol{\tau}. \dot{\boldsymbol{\gamma}} + \sum_{\alpha} \left(\lambda_{\alpha} (\Gamma_{\alpha}(\underline{\boldsymbol{\sigma}}) + z_{\alpha}) + \frac{1}{2} \iota (\Gamma_{\alpha}(\underline{\boldsymbol{\sigma}}) + z_{\alpha})^2 \right), \quad (5.12)$$

where λ_{α} are Lagrange multipliers.

It is shown in [3] that these formulations are equivalent to considering rate dependent constitutive crystal plasticity laws. In particular, the penalty expression boils down to a Perzyna viscoplastic behaviour. The variational formulation developed in this work also resorts to a viscoplastic behaviour. However, the increments of plastic slips are obtained from the minimization of the power functional (5.3) without the need for the introduction of multipliers. Besides, the activation of some specific systems recovers physical meaning in terms of the minimization of the total energy of the system.

5.1.5 Minimization strategy

Staggered scheme

The model provides an efficient and elegant way to obtain slip systems. However, it needs to be articulated with the minimization with respect to transformed volume fractions. Two strategies can be considered: either a monolithic strategy in which all internal variables are optimized at once or a staggered strategy in which the functional is minimized with respect to each internal variable in turn. The monolithic approach is numerically efficient but may prove less robust in the sense that the system to be solved can become badly conditioned when there are important discrepancies between the gradients with respect to the different internal variables. In this work, choice is made to treat variables in a staggered scheme because it allows dealing with more complex loadings. This leads to a fixed-point procedure that takes the following form:

- 1. Minimization of Π_{inc} with respect to transformed fraction
- 2. Minimization of Π_{inc} with respect to plastic slips taking into account the new transformed fractions
- 3. *Convergence check:* If the increments of plastic slips are superior to tolerance, go to 2.
- 4. *Convergence check:* If the increments of internal variables are superior to tolerance, go to 1.
- 5. Update all variables

Dealing with transformed fractions

The minimization with respect to the transformed fractions requires the enforcement of the condition $f_0 + \sum_{i=1}^{N} f_i = 1$. Two approaches are developed, one for each of the models presented in section 4.6.

SLM model An augmented Lagrangian approach is preferred in this context. Following Bertsekas [4], the problem (5.3) is rewritten as the minimization of the Lagrangian:

$$\mathcal{L}_{c_k}(\underline{\boldsymbol{\epsilon}}, \hat{T}, \boldsymbol{f}, \boldsymbol{\gamma}) = \prod_{inc}(\underline{\boldsymbol{\epsilon}}, \hat{T}, \boldsymbol{f}, \boldsymbol{\gamma}) + \mu_k g_k^+(\boldsymbol{f}, \mu_k, c_k) + \frac{c_k}{2} ||g_k^+(\boldsymbol{f}, \mu_k, c_k)||^2.$$
(5.13)

k is the current iteration index. The n and n + 1 subscripts have been dropped for convenience. μ_k is a Lagrange multiplier and c_k is the parameter that rules the penalization of the inequality $\sum_{i=1}^{N} f_i \leq 1$. g_k^+ reads: $g_k^+(f, \mu_k, c_k) = \max\left(1 - \sum_{i=1}^{N} f_i, -\frac{\mu_k}{c_k}\right)$. The minimization procedure is the following:

- 1. Initial values for the augmented Lagrangian parameters: $c_0 = 0.1$, $\mu_0 = 0.0$.
- 2. Minimize \mathcal{L}_{c_k} with respect to the transformed fractions.
- 3. If the increment of transformed fractions is small enough and such that $\sum_{i=1}^{N} f_i \leq 1$, stop computations.
- 4. $\mu_{k+1} = \mu_k + c_k g_k^+$. 5. $c_{k+1} = \begin{cases} 10c_k \text{ if } ||g_{k+1}^+|| > \frac{||g_k^+||}{4} \\ c_k \text{ otherwise} \end{cases}$ 6. Go to 2.

ELM model In the context of the ELM model, only the most energetically favourable phase forms. This leads to a treatment of the determination of the transformed volume fractions in the form of a simplex minimization. The simplex array has the form shown in Table 5.1 at the first iteration. A classical simplex minimization algorithm is then used [5].



Table 5.1: Simplex array at the first iteration.

5.2 Application of the ELM model to the ferrite-to-austenite transformation

5.2.1 Material properties

Examples of polycrystalline simulations will be presented, first for the ELM model and then for the SLM model. In both cases, the material properties used in the simulations are the one displayed in Table 5.2. The cubic elastic constants are taken from [6]. The thermal properties are the same as in section 2.4.2.

This set of properties is in the order of magnitude of what could be observed for pure iron. A fitting on some experimental data could have been performed to improve the accuracy of the final results. However, the main objective of this chapter is only to illustrate the capabilities of the model to reproduce qualitatively the behaviour of a metallic material undergoing an allotropic phase change. Besides, a long-term perspective of the present work is that properties related to crystal plasticity models will be fitted on finer scale simulations, such as dynamic of dislocations simulations.

In the case of the diffusive ferrite-to-austenite or austenite-to-ferrite transformations, the mechanical effect of the phase transformation can be considered to be a purely spherical contribution. The transformation strain tensor is then written as:

$$\underline{\boldsymbol{\epsilon}_{T}} = \begin{pmatrix} -0.0043 & 0 & 0\\ 0 & -0.0043 & 0\\ 0 & 0 & -0.0043 \end{pmatrix},$$
(5.14)

so that the associated change in volume matches the calculations of section 1.4.1.

5.2.2 Transformation behaviour of a monocrystal

Let us take the example of a unit cubic domain representing a monocrystal of given orientation. It is tested under the following conditions:

Crystal plasticity (BCC phase)																
Dissipation						Hardening					Evolution law					
E_k	(J)	$\dot{\gamma}_k$	$\gamma_{k_0} (s^{-1}) = \tau_0 (MPa)$		$s_0 (MP)$	$Pa) = U_{edg}$	$(J.m^{-1})$) b	b(mm)		$\rho_0 \ (mm^{-2})$))	κ (mm)	
1.12 1	0^{-19}	1.	$1.0\ 10^{-4}$ 1.0		2.38	1.	$31 \ 10^{-9}$	2.5	$57 \ 10^{-7}$	0.01	10^{6}		2.	3 1.09	0.10^{-4}	
Crystal plasticity (FCC phase)																
Dissipation					Hardening			Evolution law								
		m	$\dot{\gamma}_0 (s^{-1})$) $\tau_0 (MPa)$		$\tau_{f_0} (MP)$	$a) \qquad b(n)$	nm)	$\rho_0 (mr$	$n^{-2})$	K	$y_c (mr)$	n)			
			1.0	$1.0 \ 10^{-4}$		1.0	60	2.57	10^{-7}	10	6	30	2.0 10-	-6		
[Transformation Elasticity													1		
ĺ	Latent heat						Dissipation Elastic constants (T in K)						1			
	$L_T (kJ.kg^{-1}) = \Theta_{transf} (K)$		$\dot{f}_0 (s^{-1})$) $d_{f_0}(J)$	$(.mm^{-3})$	р	C11 (C	(Pa)	C12	(GPa)	C44	(GPa)	1			
	16.0			1180	0.0	1.0	1.0	10^{-3}	1.0	243-0.0	55T	147-0	0.034T	122-	-0.022T	

Table 5.2: Material properties.

- Mechanical loading No mechanical solicitation is applied on the sample. Rigid body motion is blocked.
- Heat source A heating source is introduced in the same way as in 2.3.1. The domain is heated continuously under the effect of a constant current of 50 A.
- Transformation One variant of ferrite transforms into one variant of austenite.

The values of the transformed volume fraction when it first exceeds zero are presented in Figure 5.1. The domain transforms all at once from ferrite to austenite. Indeed, the model considers no initial heterogeneity in the mechanical state of the material.



Figure 5.1: Transformational response of a monocrystalline cubic domain under continuous heating. The cube transforms all at once. The green line indicates the edge on which a force will be applied.

A solution to induce an heterogeneous response of the sample would be to introduce numerical perturbations in the initial state of the sample. However it seems natural to resort to a mechanical solicitation so as to better exploit the capacity of the model to couple mechanical and transformational effects. In the case of a polycrystal, even pure tension causes heterogeneities in the material mechanical response. As far as the monocrystalline domain is concerned, the loading itself has to be heterogeneous. For instance, the cubic domain is tested under the following conditions:

- Mechanical loading Constant force with a magnitude of 350 N applied in the y direction on the edge highlighted in green in Figure 5.1.
- Heat source Constant Joule source associated with a current of 50 A.
- Transformation One variant of ferrite transforms into one variant of austenite.

The repartition of the elastic free energy after the mechanical loading and at the onset of transformation is shown in Figure 5.2a. The distribution of the transformed volume fraction at the same timestep is shown in Figure 5.2b. It can be seen that the transformation initiates where the material stored the most mechanical free energy.

The following test is now performed to illustrate the capacity of the model to deal with several variants:

- **Mechanical loading** Uniform loading in tension along the y axis up to a longitudinal strain of 0.1%.
- Heat source Constant Joule source associated with a current of 50 A.
- Transformation One variant of ferrite transforms into three variants of austenite.


imposing a linear vertical force on one edge of the sample.





All three variants are associated with the same distortion matrix as in section 5.2.1 but with different rotations, namely a 45° rotation around the (100) axis, the (010) axis and the (001) axis of the parent crystal respectively. This corresponds to a Bain Orientation Relationship (OR) being preserved between the parent and the product grain. As underlined in section 3.3.2, this assumption is not verified in the case of diffusive transformations and a more complex set of rotations should be considered to better represent the formation of several variants during a reconstructive transformation.

Again, the domain would transform all at once without any heterogeneity in the mechanical state of the material. Besides, all three variants would be equally encouraged to grow and the variant that would form would be determined randomly by the algorithm. However, the application of the tensile loading leads to a selection of one of the variants, as shown in Figure 5.3.

5.2.3 Transformation behaviour of a polycrystal

A microstructure with several grains is used to gain more insight on the transformation mechanisms occuring at grain boundaries. A numerical sample is generated using Neper software [7], that relies on a Laguerre tessellation for the construction of the polycrystal geometry. The size of the domain of study is 0.1 mm per 0.1 mm per 0.1 mm. Only three grains are considered in order to limit the number of possible nucleation sites. The geometry used for the computations presented in this section is shown in Figure 5.4.

This sample is submitted to the following testing conditions:

- Mechanical loading Uniform loading in tension along the y axis up to a longitudinal strain of 0.1%.
- Heat source Constant Joule source associated with a current of 50 A.
- Transformation One variant of ferrite transforms into three variants of austenite.

The map of transformed variants is shown in Figure 5.5. The domain consists in the final state of a mixture of the first and third variants. The strain magnitude after the transformation is also represented in Figure 5.5. It is defined as:

$$||\underline{\epsilon}|| = \sqrt{\underline{\epsilon} : \underline{\epsilon}} \tag{5.15}$$





Figure 5.3: Repartition of transformed fractions after the diffusive transformation for the three variants considered in this example.



Figure 5.4: Microstructure generated for the numerical validation of the model.

The model fails at reproducing the strain localizations that are observed experimentally. Instead, the strain field is homogeneous once the material has transformed and it only reflects the mean response of the domain. To complement the present observations, tests will now be performed on microstructures extracted from the experimental measurements.

5.3 Response of a polycrystal under continuous heating

5.3.1 Main challenges brought by dealing with experiment-like samples

The final goal of the modelling work carried on here is to use the numerical tool for a direct comparison with experimental results. However, conducting calculations on realistic microstructures is difficult because of the presence of disparities in the grain sizes that require a very fine meshing and critically increase the number of nodes. The experimental microstructure may be simplified to circumvent this issue, which will be done in section 5.3.2.



Figure 5.5: Transformational response of a polycrystal cubic domain under continuous heating.

However, the coupled problem remains difficult to be solved. First, the minimization process described in section 5.1.5 is conducted at each material point in addition to the global minimization process to search for the global equilibrium. Second, there is a number of issues that are raised by the nature of the problem itself:

- the number of internal variables is high. Several slip systems are considered for several variants which implies that two mechanical states can be energetically close at a given time. Taking into account dissipation constitutes a regularization of the problem but it does not discard all situations when the mechanical state of the material oscillates between two local minima in the total incremental energy landscape;
- at given time, a material point represents a portion of the sample that contains, the parent phase, a variant of the product phase or a mixture of several variants. Each variant is more or less deformed depending on the loading applied to the sample. Consequently, the range of mechanical states in which the material points can be is extremely large. This may lead to a bad conditioning of the global tangent matrix;
- likewise, the local Hessian may be badly conditioned due to heterogeneities in the internal variables values.

These difficulties require to conduct the calculations with small timesteps. The finite element code is parallelized using OpenMP to improve performance. The parallelization is made at the level of the update of material points and of the assembly of the tangent matrix. Computations can eventually be run on a single node with 24 cores. Typical computation time for a simulation of transformation during heating is 80h for 10000 degrees of freedom (dofs). This clearly limits the number of grain that can be treated. Two solutions may be considered if one wants to conduct tests on large scale samples:

- consent to a reduction in the resolution of the results by using homogenization methods. Information on slip systems activity for instance may be retrieved but each grain is reduced to a material point. As a result, intragranular localizations cannot be captured;
- reshape the finite element and material behaviour codes to improve their performance. In particular, the possibility to run the code on several nodes could reduce the computation time, although a high number of nodes would be required to cut it by one order of magnitude or more. This solution has not been explored in this work. Calculations are thus performed on rather coarse meshes with an awareness on the limited precision of the final results.

5.3.2 Construction of the numerical microstructure

A methodology has been implemented to build meshes from the EBSD data. First, grains boundaries are computed using the MTEX toolbox. Small grains (whose size is inferior to 50 pixels) are removed not to introduce non-physical singularities in the computation results. Each grain in the EBSD map is assigned its mean orientation. An image of the grain structure is then saved and used as the basis for the mesh construction algorithm.

The mesh construction algorithm is fairly simple. A reference length d_r is chosen. A set of points Γ_P pertaining to the grain boundaries is initialized with the top left-hand corner of the EBSD image. From each point in Γ_P , a circle of radius d_r is drawn and all the points at the intersection of the circle and grain boundaries are added to Γ_P . This procedure is iterated until the whole image is treated. The points are linked together to form grain boundaries. No information is known relatively to the shape of the grains in the thickness of the samples. Consequently, a simple extrusion of the 2D mesh is performed. A comparison between an EBSD map and the geometry used for computations is drawn in Figure 5.6. The orientation of each grain in the numerical microstructure is the same as the mean orientation obtained from EBSD measurements. The dimensions of the numerical sample are 10.5 mm per 4 mm per 1 mm.



z

(a) EBSD representation of the grain orientations

(b) Geometry extracted from the EBSD map

Figure 5.6: Comparison between the initial EBSD data used for the generation of the microstructure and the obtained numerical sample.

5.3.3 Estimation of the macroscopic response of the sample

Basics of homogenization

The objective of this part is to provide an illustration for what can be obtained when embedding the material point model into an homogenization scheme. Let us consider the example of a sample mechanically loaded under athermal conditions. Homogenization hinges on the idea of building a bridge between the properties of individual grains, whose strain and stress tensors are $\underline{\epsilon}^{gr}$ and $\underline{\sigma}^{gr}$ respectively, and the properties of the macroscopic medium, associated with \underline{E} and $\underline{\Sigma}$:

$$\begin{cases} \underline{\epsilon^{gr}} = \underline{\underline{A}^{gr}} : \underline{\underline{E}} \\ \underline{\sigma^{gr}} = \underline{\underline{\underline{B}^{gr}}} : \underline{\underline{\Sigma}} \end{cases}$$
(5.16)

The relation between local and macroscopic moduli is derived from these equations [8]:

$$\underline{\underline{C}_{\epsilon}} = <\underline{\underline{A^{gr}}} : \underline{\underline{C}_{\epsilon}^{gr}} > \tag{5.17}$$

However, equation (5.17) was established for an elastic behaviour. When dealing with plasticity or other sources of non linearities, linearisation of the material behaviour has to be performed. An incremental approach based on the work of Hill [8] consists in regarding only the instantaneous behaviour: $\underline{\dot{\sigma}}^{gr} = \underline{C}^{gr}$: $\underline{\dot{\epsilon}}^{gr}$. At the macroscopic level, a similar expression rules the system behaviour: $\underline{\dot{\Sigma}} = \underline{C}_{\underline{\epsilon}} : \underline{\dot{E}}$. Different linearization methods may otherwise be employed:

- a secant method [9] where a relation between stress and strain is directly assumed. This
 method is particularly used for viscoplastic models in which the relation can be derived
 from the creep law;
- an affine method [10] where local developments of the fields are used to come up with a continuous formulation of the behaviour;
- it is also possible to treat homogenization in a variational way, following the ideas of deBotton and Ponte Castaneda [11].

A self-consistent approach is adopted here. The leading idea is to assume that each grain can be modelled as an elliptical inclusion surrounded by a homogenized medium whose properties are the averaged properties of all the other grains. To compute the localization tensor $\underline{\underline{A}}$ that appears in equation 5.17, the following expression is needed:

$$\underline{\underline{A}^{gr}}_{\underline{\epsilon}} = \left(\underline{\underline{C}^{gr}_{\epsilon}}_{\underline{\epsilon}} + \underline{\underline{C}^{gr^{\star}}}_{\underline{\epsilon}}\right)^{-1} : \left(\underline{\underline{C}_{\epsilon}}_{\underline{\epsilon}} + \underline{\underline{C}^{gr^{\star}}}_{\underline{\epsilon}}\right)$$
(5.18)

where $\underline{\underline{C}^{gr^{\star}}}$ is the influence tensor, computed through:

$$\underline{\underline{C^{gr^{\star}}}} = \underline{\underline{C_{\epsilon}}} : \left(\underline{\underline{S^{gr}}}^{-1} - \underline{\underline{I}}\right).$$
(5.19)

 $\underline{S^{gr}}$ is the Eshelby tensor that arises from the resolution of the elliptical inclusion problem. In the case of an isotropic behaviour, analytical expressions for this tensor can be established [12]. However, in the general case, it has to be determined numerically. The method of Gavazzi and Lagoudas [13] is employed in this work.

The algorithm used for the implementation of the self-consistent method is outlined below. Since equation 5.17 has an implicit form, an iterative algorithm is employed:

- 1. Initialization: for each grain, $\Delta \epsilon = \Delta E$.
- 2. Computation of $\underline{\Delta \sigma^{gr}}$ and C_{ϵ}^{gr} in each grain.
- 3. Computation of Eshelby tensor $\underline{S^{gr}}$ in each grain.
- 4. Computation of the influence tensor in each grain using equation 5.19.
- 5. Computation of the localization tensor in each grain using equation 5.18.
- 6. Computation of the macroscopic tangent modulus using equation 5.17.
- 7. Convergence check: If $||C_{\epsilon_{n+1}} \underline{C_{\epsilon_n}}||$ is superior to tolerance, go to 4.
- 8. Update strain increment in each grain: $\underline{\Delta \epsilon^{gr}} = \underline{\underline{A}^{gr}} : \underline{\Delta E}$
- 9. Convergence check: If $||\underline{\Delta \epsilon_{n+1}^{gr}} \underline{\Delta \epsilon_n^{gr}}||$ is superior to tolerance, go to 2.

Mean response of the polycrystal

The volume of each grain in Figure 5.6 is computed and used to weigh the contribution of the grain to the macroscopic mechanical response of the sample. The sample is submitted to a continuous heating test. Figure 5.7 displays the evolution of the mean transformed fraction and of the elastic free energy stored in the sample. As expected, all grains transform at the same time. The elastic free energy exhibits a jump when the transformation occurs. A dilatation behaviour is retrieved, which brings confidence with respect to the possibility to retrieve overall some of the experimental trends thanks to mean-field homogenization. This approach would probably deserve more work in the future.



Figure 5.7: Evolution of the magnitude of strain and of the transformed volume fraction during a continuous heating test.

5.3.4 Mesoscopic behavior obtained with the ELM model

A mesh is built from the geometry presented in Figure 5.6 and the numerical sample is submitted to the following test:

• **Mechanical loading** Uniform loading in tension along the z axis up to a longitudinal strain of 0.1%.

- Heat source The heat source aims at reproducing the temperature conditions encountered in the experiment. A Joule heat source assuming a constant current density along the cross-section (*i.e* the sections parallel to the x-y plane) of the sample is introduced as before. However, a different value for the cross-section is assigned to each point to recall the geometry of the experimental samples. As a consequence, a temperature gradient is present in the simulations.
- Transformation One variant of ferrite transforms into one variant of austenite.

The accent is put on the moment when the maximum of the transformed volume fraction in the sample first exceeds zero. The distribution of the transformed volume fraction is shown in Figure 5.8, as well as the temperature distribution at that moment and the associated distribution of the magnitude of the strain tensor. As expected, there is a linear demarcation between the center of the sample that is transformed and its extremities. Nonetheless, contrarily to what have been observed experimentally, the strain field does not exhibit any kind of localization.





Figure 5.8: Temperature, transformed volume fraction and strain magnitude after the center of the sample has transformed. Fields computed with the ELM model.

5.3.5 Comparison with the SLM model

In order to establish a direct comparison with the results yielded by the ELM model, let us consider the exact same test case solved with the SLM model. The evolution of the transformed volume fraction is presented in Figure 5.9. The main difference between the two models is that in the case of the SLM model, the center of the sample is not fully transformed and there is a gradient of transformed volume fraction that is much smoother than in the case of the ELM model. However, in both cases, the representation of the strain field after the transformation is not satisfying.



(a) Transformed volume fraction.

(b) Strain magnitude.

Figure 5.9: Transformed volume fraction and strain magnitude after the center of the sample has transformed. Fields computed with the SLM model.

5.4 Application of the SLM model to the austenite-to-ferrite transformation

The focus is put on the displacive austenite-to-ferrite transformation. The material properties are the same as in section 5.2.1. For simplicity, a Bain transformation mechanism is considered here. The transformation strain tensors are:

$$\underline{\boldsymbol{\epsilon}_{T_1}} = \begin{pmatrix} 0.25 & 0 & 0\\ 0 & -0.12 & 0\\ 0 & 0 & -0.12 \end{pmatrix} \underline{\boldsymbol{\epsilon}_{T_2}} = \begin{pmatrix} -0.12 & 0 & 0\\ 0 & 0.25 & 0\\ 0 & 0 & -0.12 \end{pmatrix} \underline{\boldsymbol{\epsilon}_{T_3}} = \begin{pmatrix} -0.12 & 0 & 0\\ 0 & -0.12 & 0\\ 0 & 0 & 0.25 \end{pmatrix}.$$
 (5.20)

Each transformation strain tensor is associated with a 45° rotation to account for the change in crystal axes system at the transformation. Let us now consider the cooling of a cubic monocrystalline domain. When no mechanical solicitation is applied to the material, no variant is favoured over the others. As a consequence, a balanced mixture of the three is formed to accommodate the overall straining induced by the transformation as shown in Figure 5.10.

Upon the application of a tensile solicitation along the y axis of the cube up to a longitudinal strain of 0.1%, the repartition between variants is changed, as highlighted in Figure 5.11. The strain magnitude distribution after the transformation is also represented in this figure. As for the ELM model, the strain field only represents the mean strain undergone by the material. The SLM model fails at capturing strain localizations occurring during the transformation.



(c) Variant 3

Figure 5.10: Repartition of transformed fractions after the displacive transformation for the three variants considered.



Figure 5.11: Repartition of transformed fractions after the displacive transformation for the three variants considered and corresponding strain magnitude.

5.5 Conclusion on numerical experiments

A strategy for the modelling of metallic materials undergoing allotropic phase changes has been proposed in this work. It is constructed under a thermodynamics-consistent variational framework. Its main features is that transformed volume fractions are treated as internal variables and that it can be divided into two submodels depending on the transformation mechanisms that are at stake.

This work has not resulted in a full comparison between experimental trends and numerical simulations. However, there are signs that it is on track:

- the macroscopic behaviour during the experimental tests is well reproduced;
- the SLM and ELM models are suited for the transformation cases they have been derived for, *i.e.* displacive and diffusive transformations respectively.

Nonetheless, two points are disappointing with regard to the possibility of using the model to explain experimental behaviours:

- computation times deter from the investigation of complex microstructures;
- the shear strain field after the transformation is much more homogeneous in the simulations than in the experiments. The effect of the mesh size has not been investigated enough and it may be that very fine meshes are necessary to account for small scale variations of the strain field. However, it is likely that other adjustments will need to be made. A derivation of the model in finite transformations to include the effect of rotations and a proper consideration of interfaces could be keys to progress in this area.

Bibliography

- L. Stainier. Consistent incremental approximation of dissipation pseudo-potentials in the variational formulation of thermo-mechanical constitutive updates. *Mechanics Research Communications*, 38(4):315–319, June 2011. 89
- [2] Ronaldo I. Borja and Jon R. Wren. Discrete micromechanics of elastoplastic crystals. *In*ternational Journal for Numerical Methods in Engineering, 36(22):3815–3840, November 1993. 89
- [3] Martin Schmidt-Baldassari. Numerical concepts for rate-independent single crystal plasticity. *Computer Methods in Applied Mechanics and Engineering*, 192(11-12):1261– 1280, March 2003. 90
- [4] D.P. Bertsekas. *Constrained optimization and Lagrange multiplier Methods*. Academic Press, 1982. 91
- [5] G.B. Dantzig. *Linear Programming and Extensions*. Princeton University Press, 1963. 91
- [6] John Leese and A. E. Lord. Elastic Stiffness Coefficients of Single Crystal Iron from Room Temperature to 500°C. *Journal of Applied Physics*, 39(8):3986–3988, July 1968.
 92
- [7] R. Quey, P. Dawson, and F. Barbe. Large-scale 3D random polycrystals for the finite element method: Generation, meshing and remeshing. *Computer Methods in Applied Mechanical Engineering*, 200:1729–1745, 2011. 94
- [8] R. Hill. Continuum micro-mechanics of elastoplastic polycrystals. *Journal of the Mechanics and Physics of Solids*, 13(2):89–101, April 1965. 98
- [9] R.A. Lebensohn and C.N. Tomé. A self-consistent anisotropic approach for the simulation of plastic deformation and texture development of polycrystals: Application to zirconium alloys. Acta Metallurgica et Materialia, 41(9):2611–2624, September 1993. 98

- [10] André Zaoui and Renaud Masson. Micromechanics-based modeling of plastic polycrystals: an affine formulation. *Materials Science and Engineering: A*, 285(1-2):418–424, June 2000. 98
- [11] G. DeBotton and P. P. Castaneda. Variational Estimates for the Creep Behaviour of Polycrystals. *Proceedings of the Royal Society A: Mathematical, Physical and Engineering Sciences*, 448(1932):121–142, January 1995. 98
- [12] T. Mura. Micromechanics of defects in solids. Martinus Nijhoff Publishers, 1987. 98
- [13] A. C. Gavazzi and D. C. Lagoudas. On the numerical evaluation of Eshelby's tensor and its application to elastoplastic fibrous composites. *Computational Mechanics*, 7(1):13–19, 1990. 98

6

Conclusion

The general goal of the present work was to show that the study of the ferrite-to-austenite and austenite-to-ferrite transformations in iron is a promising benchmark case accessible to both high-performance numerical mechanical simulation tools and to modern experimental equipments.

The problem was addressed from both a numerical and an experimental standpoints. At the experimental level, the main contribution of the present work is the design of a novel experimental set-up to study phase changes at the mesoscopic scale. The methodology chosen to study the ferrite-to-austenite and austenite-to-ferrite transformations in iron is to track the perturbations they induce on kinematic fields, which are computed using the Digital Image Correlation (DIC) technique. The experimental device allows taking high-resolution (around 1.6 μ m) images of the surface of a sample without any deterioration in image quality even above the transformation temperature. It is used to submit high-purity samples to heating and cooling ramps so that they go through a ferrite-to-austenite-to-ferrite transformation cycle. This procedure is complemented by the acquisition of the ferrite grains sizes and orientations in the initial and final states using Electron BackScattered Diffraction mappings.

When the ferrite-to-austenite transformation occurs, strong strain localizations are observed as a result of the accommodation of the volume misfit between the two phases. The Maximum Shear Strain (MSS) quantity is shown to be the best quantity to compute to post-process the obtained kinematic fields. It takes non zero values only after the transformation begins and it quantifies the severity of the misfit between the phases. Two behaviours are characterized due to the geometry of the sample. In the center, the temperature is the same everywhere and the MSS peaks are markers of nucleation events. In the absence of plastic deformation of the material, it is confirmed that triple points act as favourable points for nucleation. On the sides of the samples, there is a gradient of temperature. A transformation front progressively develops and advances in the material. The MSS values are more homogeneous in this zone, which indicates that the temperature gradient favours growth over nucleation. A thresholding on the MSS values permits to extract the transformation front.

These trends are confirmed by the study of the ferrite microstructure in the final state. Grains are elongated on the sides of the samples. In the center of the sample, small grains with various orientations are formed to accommodate the orientation incompatibilities when the two fronts join at the end of the austenite-to-ferrite transformation. The main disadvantage of the experimental procedure is that the information on the austenite phase morphology is lost. A method has been developed to retrieve this information. It is based on the comparison for a given point between its orientation in the final state and the orientations it could have following pre-defined transformation paths. The low error levels in the orientations comparison support the hypothesis of a nucleation-growth for the ferrite-to-austenite transformation, with a nucleated germ having at least one coherent boundary with one adjacent ferrite grain, followed by a displacive austenite-to-ferrite transformation.

At the numerical level, the main challenge to overcome was to develop a model that could couple the physics involved in the study of the ferrite-to-austenite transformation: thermodynamics of phase changes, thermics and mechanics of the polycrystal. Besides, it only incorporates parameters that can be determined from simulations at finer scales in order to stay in line with the driving motivation for the present work. The originality of the approach is that modulo an adaptation in the treatment of the mixture of phases, diffusive and displacive transformation mechanisms are modelled under the same variational formalism.

Simulations were conducted on representative microstructures with a few grains and on simplified microstructures extracted from experimental measurements. It is necessary to solicit the material mechanically first in order to introduce heterogeneities in the grain structure before thermal loading. The model allows to capture the importance of grain boundaries on the nucleation process. Finally, one has access to the final microstructure after heating. Another advantage of the model that is not exploited to its full extent in this work is the possibility to extract plastic slips on the different slip systems of each crystal to study defects reorganization. However, only strain fields that were too homogeneous to compare with experimental trends were obtained so far at the end of the simulation of an allotropic transformation in a polycrystalline material.

Despite some encouraging trends evoked in the previous paragraphs, some footsteps are thus necessary for the experimental and numerical ways to meet. In particular, the following perspectives can be highlighted:

- at the experimental level, it remains to dissociate plastic strain from transformation strain in the post treatment of the kinematic fields obtained by DIC;
- the thermomechanical treatment of the iron samples could be improved to obtain even coarser grains. The grains would then be almost 2D and it would be easier to interpret the obtained kinematic fields;
- the simulation work hit the barriers it tried to overcome in the sense that the computations on complex microstructures or microstructures with grain sizes heterogeneities are too long to be performed on a single computation node. It is then necessary to improve the performance of the code;
- once this limitation is overcome, what could be done to improve the model is to provide a better description for the role of grain boundaries. Either a misfit term could be added or transformed fractions could be treated as order parameters so that mixed terms between the parameters would provide the interface energy in the fashion of a phase field approach. This could improve the representation of strain fields after transformation;
- the transformation physics could be more developed. In particular, a model that would dissociate nucleation and growth could be of interest in the case of the ferrite-to-austenite transformation.

Acknowledgments

I would like to express my gratitude to Éric Charkaluk and Bertrand Wattrisse who accepted to review my thesis. Their valuable comments broadened the scope of the issues addressed in this work and gave it a whole new dimension. I am also extremely grateful to Brigitte Bacroix and Guillaume Kermouche for being President of the Jury and Examiner respectively. The expertise of the Jury led to fruitful discussions and opened very interesting perspectives regarding the *in situ* observation of transformations.

I would like to point out that this thesis could not have existed without the contribution of many people, among who stand out my three supervisors. First, I would like to thank Michel Coret. Michel, thank you for taking the time to answer my questions, as silly as they can be, in so many occasions and for always being willing to share your immeasurable knowledge. I will always keep in mind your sense of curiosity and the desire to tackle complex problems in order to reveal their richness little by little. Thank you also to Laurent Stainier for his guidance throughout the modelling developments of this thesis. Your inspiring vision of the thermomechanics of metallic materials and your rigor will hopefully long remain instilled in my work. Finally, I would like to thank Bertrand Huneau for sharing with me his contagious enthusiasm for the study of metallic materials and the evolutions within their microstructure. This work owes a lot to his analysis of these phenomena but also to his ability to gather people around their study.

Among these people, I would like to thank Emmanuel Bertrand for his precious help regarding the implementation of the thermomechanical treatments for grain coarsening. My thanks also go to Marilyne Mondon who courageously went through the hard task of making largescale EBSD maps and contributed to the quality of the images presented in this work.

I am extremely grateful to all the people who participated in the design and usage of the experimental device described in this document. In the first place, many thanks to Emmanuel Bon without whom this bench would not have its present form. I also thank Pierrick Guégan and Franck Pasco who kindly welcomed me in the CRED and helped me with technical issues. I am grateful to Alexandre H and Raphaël who made me forget the daunting nature of test campaigns and made each test a special moment. Thank you to the students who contributed to these campaigns: Alexandre J, Olivier, Anaël, Vincent.

This thesis was conducted in the PMM (Process and Mechanics of Materials) group of the GeM Institute. It was a very pleasant experience. I cannot name everyone but I want each member of this group reading these lines to feel acknowledged for making me feel at home in the cool atmosphere of the lab. I would like to make a special dedication to the OSDM team, to the T226 team, to the coffee team, to the badminton team, to the tennis team... Actually, it all boils down to enthralling and funny moments and discussions with you guys. In this regard, I address thanks to Angel and Marie for all the comforting end-of-the-day discussions.

I am also thanking my dear friend Imadeddine for his support all along those three years of PhD. Finally, I would like to thank my family, and especially my sister for showing me how to keep things going and my parents for their enduring support. I wish that they will find in these lines a testimony of my gratitude and my affection.

UNIVERSITE BRETAGNE SCIENCES LOIRE POUR L'INGENIEUR



Titre : Couplage entre plasticité et transformation de phase dans le Fer

Mots clés : Transformation allotropique, Corrélation d'Images Numériques, Formalisme variationnel

Résumé : Les propriétés mécaniques des alliages de largement conditionnées par fer sont leur microstructure et la population de défauts locaux qu'elle contient. Comme il s'agit d'un moment de forte interaction entre ces deux éléments, l'étude des transformations α -y et y- α du fer mérite une attention particulière. Outre le passage d'une structure cristalline cubique centrée à une structure cubique faces centrées - donc de compacités différentes -, elles sont associées à une réduction des paramètres de maille. Le changement de volume correspondant est responsable de déformations mécaniques locales autour des sites de germination.

La technique de corrélation d'images numériques (CIN) s'est montrée fiable pour ce qui est de calculer des champs cinématiques à l'échelle de quelques grains. Dans ce travail, elle est adaptée à la capture de localisations de la déformation pendant les transformations allotropiques.

Un banc expérimental est conçu pour obtenir des images haute résolution avec un contrôle fin des sollicitations thermiques. Des essais de validation sont d'abord effectués sur du fer industriel. Puis des échantillons de fer haute pureté sont ensuite soumis à des cycles de transformation α -y- α et les champs de déformation correspondant sont calculés par CIN. Associés à l'acquisition des orientations initiales et finales, ils sont utilisés pour valider les mécanismes de transformation proposés dans la littérature. parallèle, un modèle, écrit En en petites déformations, est construit en incorporant des composants liés à la transformation dans une fonctionnelle dont les conditions de stationnarité sont équivalentes au problème thermomécanique à résoudre. Les incréments des variables internes, alissements plastiques incluant et fractions volumique transformées, sont obtenus en minimisant cette fonctionnelle.

Title: Coupling between plasticity and phase transformation in iron

Keywords: Allotropic transformation, Digital Image Correlation, Variational Framework

Abstract: Mechanical properties of iron-based alloys are largely conditioned by their microstructure and the population of local defects inside this microstructure. As it is a moment of massive interplay between these two elements, the study of the α - γ and γ - α transformations in iron is of particular interest. Besides a change from a body-centered cubic to a face-centered cubic crystal structure - and thus a change in compacity -, they lead to a lattice parameter reduction. The corresponding change in is responsible volume for local mechanical deformations around transformation sites.

Digital Image Correlation (DIC) technique has been proven reliable to compute kinematic fields at the scale of a few grains. In the present work, an adaptation of this technique to the observation of strain localizations induced by the formation of a new phase is proposed.

A home-made device is designed to obtain highresolution images and to control heating and cooling. Tests are first conducted on industrial iron to assess the viability of the procedure. High-purity iron samples are then submitted to α - γ - α transformation cycles and the associated strain fields are computed. In combination with the acquisition of initial and final orientations they are used to validate transformation mechanisms proposed in the literature.

In parallel, a model, written under the small strain format, is built by incorporating transformationrelated components into a power functional whose stationarity conditions are equivalent to the thermo mechanical problem. In accordance with variational principles, the evolution of internal variables, including plastic slip increments and fraction of the material locally transformed, are computed through the minimization of the functional.