



Listening to materials : in search of the unseen

Stéphanie Deschanel

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Mémoire d'Habilitation à Diriger des Recherches

Listening to materials

Institut National des Sciences Appliquées de Lyon et l'Université
Claude Bernard LYON I

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October 7, 2020

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Foreword

I have heard that writing the habilitation thesis is an easy and enjoyable task. Indeed, it is the opportunity to look back over the past years of research and teaching and then look forward to new research and academic projects. Somehow, I had trouble starting it, maybe because there are so many ways to do it and a decision should just be made, almost to echo the beauty and difficulties of the researcher/teacher profession where the variety and possibilities are virtually endless. The freedom to choose what you want to work on and who you collaborate with is one of the nicest things about academia. But, like anything truly worth doing, we all know that being your own boss comes with its own set of unique challenges! And it is also about opportunities appearing all along and about the financial supports you may... or may not get (but this is an other issue that could take an entire thesis to write on!). Anyhow, I am happy to try to outline the main scientific results obtained during the last decade or so of my career, and it's about time! I will report about 90% of my research from 2007, when I got my position at MatéIS, until today ; research that is focused on the study of plasticity and damage of materials monitored by Acoustic Emission (AE) and the dynamics of fatigue and fracture precursors, at the interface between mechanics and physics.

The wonder now is how to proceed? There is no specific template to write an Habilitation à Diriger des Recherches (HDR) and there are as many HDR manuscripts as there are associate professors writing one.

Although this manuscript will be read by no more than a handful of readers (and I don't blame all the others! "but still!"), I did not want to juxtapose articles and I thought that this document could be profitable to young researchers starting in the field. It is with this in mind that I wrote chapter 1 as an introduction, in a sensory journey, to experimental techniques, methods and analyses used and developed in the field and more specifically to the AE method, the driving force of my research, adding here and there personal reflections on research and teaching approaches. To quote the first article of the decree of 23rd November 1988 on HDR : "L'habilitation à diriger des recherches sanctionne la reconnaissance du haut niveau scientifique du candidat, du caractère original de sa démarche dans un domaine de la science, de son aptitude à maîtriser une stratégie de recherche dans un domaine scientifique ou technologique suffisamment large et de sa capacité à encadrer de jeunes chercheurs". HDR is a degree which accredits to supervise alone PhD students, so it might be right to think this way. Incidentally, reading the last words of the article, isn't it ironic to ask the applicants to have already conducted research in order to pass the habilitation to conduct research?

Otherwise, one might wonder about the interest of an HDR in terms of academic career when in any case the opportunity of a professor's promotion tends towards zero in the economic-population pyramid-university autonomy etc. (!) context.

Without forgetting global warming! Should we just rely on Darwin?

Anyhow, even if it is a necessary step in the carrier of an associate professor, writing an HDR is above all a reflection for yourself and an opportune time to take stock of the past research and define a scientific perspective. Moreover, the HDR being an official degree, this manuscript is also written keeping in mind that the first (and perhaps the only!) readers and reviewers will be the members of the jury (I do thank them for that).

Chapters 2 and 3 discuss to the life of a material from its pristine state, through plasticity, growing damage, up to rupture. The last chapter 4 is dedicated to my research project. I also included in a very last chapter my Curriculum Vitae.

Otherwise, how could I not mention this Covid-19 lockdown period during which I am writing this HDR? On the teaching side, it has been quite hectic with an overload of work to prepare the tutorials lectures and practicals (!) in distance learning. Was it a good time to revisit the whole educational mode? I am not sure as we have had to prepare as quickly as possible and do our best to administer an education worthy of the name. However, I believe we will have to take advantage of this period and think about changing our too ascending vision of our teaching and favor the reverse teaching. On the research side, well, I must confess that when the teaching gave me time, being locked down, it has been a perfect forced time of reflection!

Acknowledgements

"So Long, and Thanks for All the Fish"

Hitchhiker's Guide to the Galaxy, Douglas Adams.

... And

THANKS

to all my colleagues with whom I had the opportunity to collaborate all along my journey, without whom the research presented in this document would not have been possible,

to the PhD students who came on the road, for their beautiful energy that motivated my research,

to the Mateis's technicians and assistants for their support,

to the members of the jury (in particular the reviewers), for accepting my invitation,

to the brave souls who proofread all or part of this manuscript,

to my beloved family and friends, for boosting me on this journey,

and to my dear favorite cook, for all those delicious dishes prepared with love during this writing!

Note en français

La rédaction de l'Habilitation à Diriger des Recherches (HDR) est une tâche facile et agréable, m'a-t-on dit. En effet, c'est l'occasion de revenir sur les années de recherche et d'enseignement passées et d'envisager ensuite de nouveaux projets de recherche et d'enseignement. D'une certaine manière, j'ai eu du mal à débiter la tâche, peut-être parce qu'il y a tant de façons de le formuler et qu'il faut juste prendre une décision, presque pour faire écho à la beauté et aux difficultés de la profession d'enseignant/chercheur où la variété et les possibilités sont pratiquement infinies. La liberté de choisir les sujets sur lesquels on souhaite travailler et avec qui on collabore est l'une des plus belles facettes du monde universitaire. Mais, comme pour tout ce qui vaut vraiment la peine d'être fait, nous savons tous qu'être son propre patron comporte son lot de défis uniques ! Et c'est aussi une question d'opportunités qui se présentent tout au long de votre carrière. Quoi qu'il en soit, je suis heureuse de pouvoir présenter les principaux résultats scientifiques obtenus au cours de la dernière décennie de ma carrière, et il était temps ! Je rapporterai environ 90% de mes recherches depuis 2007, date à laquelle j'ai obtenu mon poste au laboratoire MatéIS, jusqu'à aujourd'hui ; des recherches qui sont axées sur l'étude de la plasticité et de l'endommagement des matériaux suivis par Emission Acoustique (EA) et sur la dynamique de la fatigue et des précurseurs de la rupture, à l'interface entre la mécanique et la physique.

La question est maintenant de savoir comment procéder. Il n'existe pas de modèle spécifique pour rédiger une HDR et il y a autant de manuscrits d'HDR qu'il y a de chercheurs qui en rédigent un. Bien que ce manuscrit ne sera lu que par une poignée de lecteurs, je n'ai pas voulu juxtaposer les articles et j'ai pensé que ce document pourrait être profitable aux jeunes chercheurs débutant dans le domaine et en particulier utilisant l'EA. C'est dans cet esprit que j'ai rédigé le premier chapitre comme une introduction, au cours d'un voyage sensoriel, aux techniques expérimentales, aux méthodes et aux analyses utilisées et développées dans le domaine et plus particulièrement à la méthode d'EA, moteur de mes recherches. En effet, nous appréhendons le monde, qui est fait de matière, grâce aux cinq méthodes de perception, ou sens, traditionnellement reconnues : le son, la vue, le toucher, l'odorat et le goût. En outre, j'ai rajouté ici et là quelques réflexions générales sur le monde universitaire, car ce manuscrit est également l'occasion d'analyser les différentes façons d'aborder les activités de recherche et d'enseignement.

Même s'il s'agit d'une étape nécessaire dans la carrière d'un chercheur, la rédaction d'une HDR est avant tout une réflexion pour soi-même et un moment opportun pour faire le point sur les recherches passées et définir une perspective scientifique. De plus, l'HDR étant un diplôme officiel, ce manuscrit est également rédigé en gardant à l'esprit que les premiers (et peut-être les seuls !) lecteurs et évaluateurs seront les membres du jury (je les en remercie). Les chapitres 2 et

3 suivent les mécanismes multi-échelles gouvernant le comportement mécanique des matériaux du début de la déformation plastique jusqu'à la rupture. Le chapitre 4 est consacré à mes différentes perspectives de recherche pour les prochaines années. J'ai également inclus dans un ultime chapitre, mon curriculum vitae.

Voici un bref aperçu de mon parcours. Après avoir obtenu mon diplôme d'ingénieur en 2002, j'ai senti que mon goût pour les sciences n'était pas tout à fait satisfait. C'est pourquoi j'ai commencé mon voyage vers la recherche avec une thèse de doctorat, soutenue en 2005, au sein de deux laboratoires, le Laboratoire de Physique (ENS Lyon) et le GEMPPM (anciennement MatéIS, INSA Lyon), qui relie la physique à la science des matériaux. L'objectif était d'étudier la dynamique des précurseurs de la rupture des matériaux hétérogènes avec une application aux mousses polymères vitreuses. Juste après, j'ai eu l'occasion d'aller aux États-Unis (MIT, Cambridge) pour un post-doctorat portant sur les mécanismes du comportement aux grandes vitesses de polymères nanocomposites : microstructure, mécanismes et comportement mécanique. J'ai ensuite été recruté comme maître de conférences en 2007 au laboratoire MatéIS. J'ai fait partie de l'équipe ENDV (Evaluation Non Destructive et Durabilité) de MatéIS jusqu'en 2012. J'ai co-encadré avec Nathalie Godin (MatéIS) mon premier doctorant sur l'identification des sources d'endommagement dans les composites thermoplastiques par des analyses d'EA basées sur la reconnaissance de formes.

Dans les premières années de ma carrière, j'ai également pris des responsabilités pédagogiques puisqu'une nouvelle section SCANfirst (Science et Anglais, 1ère année INSA) a ouvert en 2007. J'ai dû mettre en place un cours/TD sur l'électrostatique et l'électromagnétisme, exclusivement enseigné en anglais. Cette section a été étendue en 2013.

Ayant rencontré Jérôme Weiss (LGGE et maintenant ISTerre), j'ai eu la chance de participer à un projet de l'Agence Nationale de la Recherche (ANR) sur l'évolution de la criticalité lors de chargements cycliques comme indicateur de la fatigue. Dans ce cadre, j'ai co-encadré avec lui la thèse de doctorat de Wafa El May (2009-2013) traitant de la dynamique des dislocations collectives et des avalanches lors de la fatigue de l'aluminium. En renouant avec mon premier goût pour les polymères, je les ai réécoutés avec Olivier Lame (MatéIS) et Thomas Monnier (IVA) pour explorer l'initiation de la déformation plastique et pour comprendre et caractériser la plasticité et l'endommagement des polymères semi-cristallins, avec la thèse de Nicolas Casiez. Finalement, je n'ai pas réussi à me chasser de la tête les métaux découverts au préalable et j'ai rejoint l'équipe METAL du laboratoire, fin 2013, pour appliquer mes compétences et mes connaissances en EA au domaine des matériaux métalliques.

En 2014 et 2015, j'ai passé 16 mois à Queen's University, au Canada, pour étudier le phénomène des fractures d'un point de vue totalement nouveau : via la fractoluminescence, et en couplant la technique de détection des particules à la

technique d'EA. "Les émissions lumineuses et acoustiques dans les scintillateurs inorganiques" était en effet le thème de la thèse d'Alexis Tantot que j'ai co-encadré avec Philippe Di Stefano de Queen's et Loïc Vanel de l'ILM.

En 2016, je me suis lancé dans un nouveau thème de recherche : la plasticité gouvernée par les dislocations et plus particulièrement la dynamique collective des dislocations lors de chargements cycliques de matériaux à différentes structures cristallines pour une nouvelle vision de la plasticité. J'ai co-encadré la thèse de doctorat de Gabriel L'Hôte (2016-2019) qui portait sur des monocristaux purs de cuivre, avec Maurine Montagnat (IGE) et Sophie Cazottes (MatéIS). Grâce à l'expertise de Sophie Cazottes, nous avons pu combiner l'EA à des caractérisations microstructurales en utilisant la diffraction des électrons rétrodiffusés (Electron BackScattered Diffraction - EBSD) et les techniques d'imagerie par contraste de canalisations électroniques (Electron Channeling Contrast Imaging - ECCI), ce qui nous a permis d'obtenir de nouvelles informations précieuses.

Mes travaux de recherche actuels se concentrent désormais sur ce thème et sont consacrés à l'étude de la fatigue des matériaux et à la prédiction de la rupture finale grâce aux analyses d'EA que j'ai précédemment mises en oeuvre. Cela se fera, en partie, grâce à la thèse de Théophile Vié qui a débuté en novembre 2019 sur la "Fatigue et Emission Acoustique sur des échantillons d'aluminium corrodés vers la prédiction de la durée de vie" avec Nathalie Godin et Bernard Normand (MatéIS). Récemment, avec Jérôme Weiss, nous avons fait une découverte passionnante sur la détection de signaux d'émission acoustique spécifiques de la propagation de fissures par fatigue dans les matériaux métalliques. Ces signaux, dénommés multiplets acoustiques, se caractérisent par des formes d'ondes quasi identiques, signature d'une source unique, et sont déclenchés de façon répétée sur de nombreux cycles de chargement successifs au même niveau de contrainte. Ils marquent la propagation lente et incrémentale d'une fissure de fatigue à chaque cycle, ou le frottement le long des surfaces de rupture.

Des expériences préliminaires ont ainsi été lancées, combinant l'EA et la corrélation d'images numériques (Digital Image Correlation - DIC), avec Julien Réthoré. Ce travail nous a naturellement conduit à proposer un projet sur la détection précoce de la propagation des fissures, qui mènera en définitive à une rupture globale en fatigue et sur l'étude de la rupture intermittente à l'aide de ces multiplets acoustiques validés en champ complet, projet financé par l'ANR, et que je dirigerai à partir de septembre 2020.

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List of Acronyms

AE Acoustic Emission	i
AFM Atomic force microscopy	24
AI Artificial Intelligence	16
AIC Akaike Information Criterion	14
AM Additive manufacturing	85
ANR Agence Nationale de la Recherche	2
ASL Average Signal Level	10
bcc body-centered cubic	50
BGO bismuth germanate	22
BSE Back Scattered Electrons	20
CSR cyclic stress response	34
dB Decibel	6
DBs disorientation bands	55
DCDC double-cleavage drilled compression	22
DIC Digital Image Correlation	2
DVC Digital Volume Correlation	86
EBSD Electron BackScattered Diffraction	2
ECCI Electron Channeling Contrast Imaging	2
ESP extrasensory perception	29
fcc face-centered Cubic	32
FCG fatigue crack growth	64
GMO genetically modified organisms	87
hcp hexagonal close packed	44
HDR Habilitation à Diriger des Recherches	i
IRT infrared thermography	24
LCF Low Cycle Fatigue	44
LEFM Linear Elastic Fracture Mechanics	67
LTA Long-time average	15

MAE Modal Acoustic Emission	13
MgO magnesium oxide	22
MT martensitic transformation	33
NDT Non Destructive Testing	8
PDF probability density function	47
PE Polyethylene	32
PMMA Poly(methyl methacrylate)	78
PMT photomultiplier	72
PSBs Persistent Slip Bands	19
PU Polyurethane	21
ROI region of interest	18
R-ECCI Rotational Electron Channeling Contrast Imaging	2
RMS Root Mean Square	10
RSS resolved shear stress	
RUL remaining useful lifetime	17
SAXS Small-angle X-ray scattering	37
SEM Scanning Electron Microscopy	19
SHM Structural Health Monitoring	83
SNR Signal-to-Noise Ratio	6
STA Short-time average	15
SIF Stress Intensity Factor	18
TDCB Tapered Double Cantilever Beam	78
TEM Transmission Electron Microscopy	20
TOA time of arrival	13
UT Ultrasonic testing	16
US ultrasonic	6
UV ultraviolet	22
WF Waveform	

A sensory journey

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1.1 My journey as associate professor

Let me give you a quick look back at my journey to put you in the picture. After I graduated from my engineering school in 2002, I felt that my *taste* for science was not quite fulfilled. That is why I began my *journey* into research with a PhD thesis, defended in 2005, within two laboratories, Laboratoire de Physique (ENS Lyon) and GEMPPM (former Mateis, INSA Lyon), connecting physics to material science. The objective was to investigate the dynamics of the fracture precursors of heterogeneous materials with an application to vitreous polymer foams. Right after, I had the opportunity to go to the USA (MIT, Cambridge) for a postdoctoral fellowship dealing with high rate behavior of polymeric nano-composites : microstructure, mechanisms, and mechanical behavior. I was subsequently recruited as Associate Professor in 2007 at Mateis Laboratory. I was in the ENDV (Non Destructive Evaluation and Durability) team of Mateis until 2012. I co-supervised with Nathalie Godin (Mateis) my first PhD student on the identification of damage sources in thermoplastic composites through [AE](#) analyses based on pattern recognition.

In the first years of my career, I also took pedagogic responsibilities as a new section SCANfirst (Science and English, 1st year INSA) opened in 2007. I had to

set up a "2 in 1" course/tutorial on electrostatics and electromagnetism, exclusively taught in English. This section was expanded in 2013.

Having met Jérôme Weiss (LGGE and now ISTerre), I had the chance to participate in an Agence Nationale de la Recherche (ANR) project on the evolution of criticality in cyclic loading as an indicator of fatigue. Within this context, I co-supervised with him the PhD thesis of Wafa El May (2009-2013) dealing with collective dislocation dynamics and avalanches during fatigue of aluminum. Remembering my first taste for polymers, I listened to them anew with Olivier Lame (Mateis) and Thomas Monnier (LVA) to explore the initiation of plastic deformation and to understand and characterize plasticity and damage of semi-crystalline polymers, with Nicolas Casiez's PhD thesis. Eventually, I couldn't get the beforehand discovered metals out of my head and I joined the laboratory's METAL team, end of 2013, to apply my skills and knowledge in AE to the field of metallic materials.

In 2014 and 2015, I spent 16 months at the Queen's University, Canada, to study the fracture phenomenon from a completely new point of view : via fractoluminescence, and by coupling the particle detection technique and the AE technique. "Light and sound emissions in inorganic scintillators" was indeed the theme of Alexis Tantot's thesis whom I co-supervised with Philippe Di Stefano from Queen's and Loïc Vanel from ILM.

In 2016, I embarked on a new research theme : dislocation-mediated plasticity and more specifically collective dislocation dynamics during cyclic loading of materials with different crystal structures for a new vision of plasticity. I co-supervised Gabriel L'Hôte's PhD thesis (2016-2019) who focused on pure single crystals of copper, with Maurine Montagnat (IGE) and Sophie Cazottes (Mateis). Thanks to Sophie's expertise, we were able to combine AE to microstructural characterizations using Electron BackScattered Diffraction (EBSD) and Electron Channeling Contrast Imaging (ECCI) techniques¹ proving valuable new information.

My current research work is now focused on this theme and is dedicated to the study of material fatigue and the prediction of final failure through AE analyses that I previously implemented. This will be done, in part, through the thesis of Théophile Vié who started in November 2019 on "Fatigue and AE on corroded aluminum samples towards lifetime prediction" with Nathalie Godin and Bernard Normand (Mateis). Lately, together with Jérôme Weiss, we made an exciting discovery of acoustic emissions specific to fatigue crack growth in metallic materials, the so-called AE multiplets. Preliminary experiments were thus launched, combining AE and Digital Image Correlation (DIC), with Julien Réthoré. This work naturally led us to propose a project on the early detection of fatigue and intermittent failure using full-field approved AE multiplets, which has been granted by the ANR, and which I will be managing starting in September 2020.

1. A novel approach have been proposed for dislocations imaging : Rotational Electron Channeling Contrast Imaging (R-ECCI).

1.2 In search of the sixth sense

I will open this [HDR](#) report with a sensory journey through the five traditionally recognized methods of perception, or sense : sound, sight, touch, smell, and taste, through which we apprehend the world, which is made of matter. As a matter of fact, our senses help us feel matter. This little sensory walk will allow me to present the different experimental techniques, methods and analyses used and developed in my research. I hope that this section will be useful for future master's or PhD students who will start a research activity on a related theme and in particular using [AE](#). Besides, I will add here and there few general reflections on academia, as this manuscript is also an opportunity to analyze the different ways of approaching research and teaching activities.



FIGURE 1.1 – Let's use our five senses for research !

1.2.1 Sound : Acoustic Emission, a probe of the unseen

I will begin with sound (as every baby starts its own life) because I relate this sense to the Acoustic Emission technique, which is going to be the common thread to the different studies I conducted : the main technique that will help answer the different questions raised.

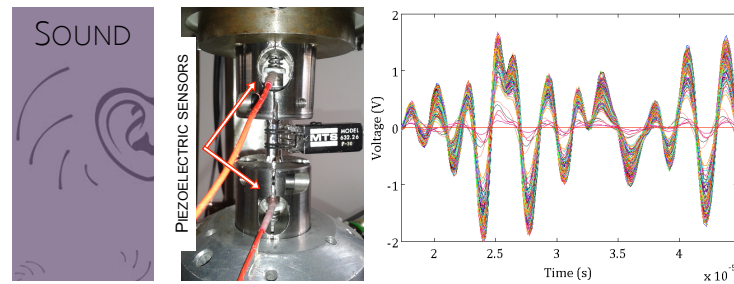


FIGURE 1.2 – SOUND. Acoustic Emission sensors recording AE waveforms

1.2.1.1 Overviews

Acoustic emission is a phenomenon of energy release in the form of transient elastic waves resulting from any sudden local change of irreversible strain [[Malen &](#)

[Bolin 1974](#)] within a material. This phenomenon occurs in many materials when they are subjected to mechanical, thermal or chemical stresses.

The application of a load and/or the presence of an aggressive environment produces internal modifications such as the evolution of cracks, local plastic deformation, corrosion and, in some cases, phase transformations dissipating energy, a fraction of which, often weak, is released in the form of elastic waves. The waves propagate through the material/structure and are then detected by means of specific sensors (usually piezoelectric transducers placed on the surface of the structure/specimen) that allow the conversion of the surface movements of the material into electrical signals. The voltage response of the sensors is then amplified and stored digitally (as AE waveforms) for analysis, giving information on the internal behavior of the materials under consideration.

I will now introduce a bit of history to set the scene. Leonardo da Vinci (1452-1519) wrote in its Notebooks [[Vinci 1880](#)] (edited by Jean Paul Richter in 1880) : "If you want to know where a mine runs, place a drum over all the places where you suspect that it is being made, and upon this drum put a couple of dice, and when you are over the spot where they are mining, the dice will jump a little on the drum at every blow which is given underground in the mining." In 1638, Galileo Galilei, said in his final book and scientific testament (Dialogues Concerning Two New Sciences [[Galilei 1638](#)]) : "This is a beautiful experiment enabling us to distinguish individually the waves which are produced by the vibrations of a sonorous body, which spread through the air, bringing to the tympanum of the ear a stimulus which the mind translates into sound. " After all, they are already foreseeing the transient elastic waves and the future piezoelectric sensors detecting the surface vibration and translating it in the form of an electrical signal. It also relates to geophysics with what is happening underground. Indeed, the AE technique is similar to seismology except that AE is in the scale of engineering structures. When AE occurs in geologic structures, it is often referred to as micro-seismic activity.

A little later, one can find in the Posthumous works of Robert Hooke (1705) [[Hooke 1705](#)] : "There may be also a Possibility of discovering the Internal Motions and Actions of Bodies by the sound they make, who knows but that in the Watch we may hear the beating of the Balance, and the running of the Teeth, and Multitude of other Noises; who knows, I say, but that it may be possible to discover the Motions of the Internal parts of Bodies, whether Animals, Vegetables, or Minerals, by the sound they make, that one may discover the Works performed in the several offices and shops of a Man's Body, and thereby discover what Instrument or Engine is out of order, what Works are going on at several Times, and Lies still at others, and the like; that in Plants and Vegetables one might discover by the noise the Pumps for raising the Juice, the Valves for stopping it, and the rushing of it out of one Passage into another, and the like." We will learn so much about materials if we listen to them carefully.

Back in the 8th Century, the alchemist Jabir ibn Haya (Geber) noticed that

"Jupiter (tin) produces a rough sound, a crack and that Mars (iron) is very sonorous during forging" [Drouillard 1996]. The noise emitted by iron is probably due to the formation of martensite during cooling. The audible "screaming" or "crackling" sound, "tin cry", was attributed to twinning by J. Czocharalski in 1916 [Czocharalski 1996], turning it the earliest known AE phenomenon in material science. In France, Albert Portevin et François Le Chatelier evoked in 1923 a "little dry sound" [Portevin & Le Chatelier 1923], during the formation of slip bands in aluminium alloys.

Since then, research on AE has been developed in different domains, on different materials (composites e.g. [Godin *et al.* 2018, Prosser *et al.* 1995, Morscher 1999, Maillet & Morscher 2015, Mills-Dadson *et al.* 2017], concrete e.g. [Ohtsu 2015, Aggelis *et al.* 2013, De Sutter *et al.* 2017], metals e.g. [Wadley *et al.* 1980, Eitzen & Wadley 1984, Heiple & Carpenter 1987a, Heiple & Carpenter 1987b, Rogers 2005, Aggelis & Matikas 2012], polymers e.g. [Eitzen & Wadley 1984, Galeski *et al.* 1987], ...) and for several applications (structure diagnosis [Ono 2011, Rogers 2001, Rogers 2017], aerospace [Holford *et al.* 2017], geotechnical fields [Lengliné & Marsan 2009], etc.)

If enough energy is released, audible sounds are produced. The cracking of timber subjected to loads near failure produces audible sounds which can be used as indicators of impending failure of wooden structures. The more sensitive our hearing, the weaker the sounds we can detect. The AE technique helps extend our hearing by detecting sounds of higher frequencies and lower intensities than human sense. Crack propagation, phase changes, and dislocation movements in metals are all examples of processes we can now listen to. AE measurements are usually performed in the ultrasonic range between 50 kHz and 2 MHz. Signals at higher frequencies would be damped by the material leading to too weak signals, when signals at lower frequencies might be combined to the background noise.

Acoustic emission is a passive technique : detection requires a source mechanism to be active (i.e. when local micro-displacements take place : damage must be growing, it is not dependent on defect size). Non evolving defects, such as voids and non-growing cracks for instance, do not yield AE. The transducers only "listen" to what the material is willing to tell and do not generate the acoustic waves that the transducers used in ultrasonics do.

Acoustic emission is a volumetric method, an entire structure can be evaluated in a single test when adequate sensor placements cover it. The technique allows concurrently global monitoring and localization of the source position using a distributed array of sensors. Unlike ultrasonic pulse-echo technique, inspection path is not predefined by applied stimulus and no volumetric scanning is needed. It can provide in situ, continuous monitoring.

Besides, the information coming from the material still need to be interpreted and related to possible physical sources. Objective correlation between AE observa-

tion and direct knowledge of the AE sources is not easy to establish. Other drawbacks of AE are high attenuation of waves in some materials and Signal-to-Noise Ratio (SNR). K. Ono made an exhaustive review in [Ono 2018] on damping and attenuation for different materials (metals, polymers and composites) concerning guided waves (Rayleigh Waves, Lamb waves, longitudinal and transverse bulk waves), on large metallic structures and laboratory attenuation measurements, over a range of frequencies. To get a rough idea, in steels, AE waves are attenuated at $0.1 - 1 \text{ dB/m}^2$, but in concrete this jumps to 45 to 118 dB/m i.e., amplitude decreases by 200 to 10^6 times after traveling 1 m . Polymers also exhibit strong acoustic damping [Jarzynski *et al.* 2003] with attenuations ranging from 1 dB/cm up to 100 dB/cm at 5 MHz . We have ascertained in [Casiez 2015] that the attenuation of ultrasonic (US) waves in semi-crystalline polymers is of the order of 100 to 1000 times greater than that commonly observed in metals. The term attenuation includes energy loss due to scattering and reflection as well as absorption, hence it depends on specimen thickness and experimental configuration.

1.2.1.2 Continuous vs discrete Acoustic Emission

A distinction can be made between two types of AE : continuous emission and discrete emission (or by bursts).

- The continuous AE is a slowly evolving background noise, the stress wave bursts are unresolvable (figure 1.3(b)). The amplitude of this background noise increases when several sources are activated at the same time. This type of emission can be associated with uncorrelated dislocation movements during the plastic deformation of metals.
- The discrete AE is related to sudden events of short duration and much higher amplitude and energy in which the individual stress wave bursts can be distinguished. The discrete AE signal called burst (or hit) has the general appearance of a damped sinusoid (figure 1.3(c)). It occurs when sources of higher energy are operating. It can be associated, for instance, to crack growth, fiber breaks, stress corrosion or collective dislocation movements (avalanches).

To illustrate the two kinds of AE, figure 1.3(a) represents a raw signal recorded during one second of the first cycle of a fatigue test on Copper : a background noise, sometimes with voltage peaks which are associated with the discrete AE (figure 1.3(c)). The continuous background contains information on the surrounding background noise and the possible dislocation movements.

1.2.1.3 Acoustic waves in elastic media

Different kinds of mechanical waves can be distinguished by considering how the motions of the particles of matter are related to the direction of propagation of the

2. The Decibel (dB) is a unit based on twenty times the common logarithm of the ratio of two acoustic voltages.

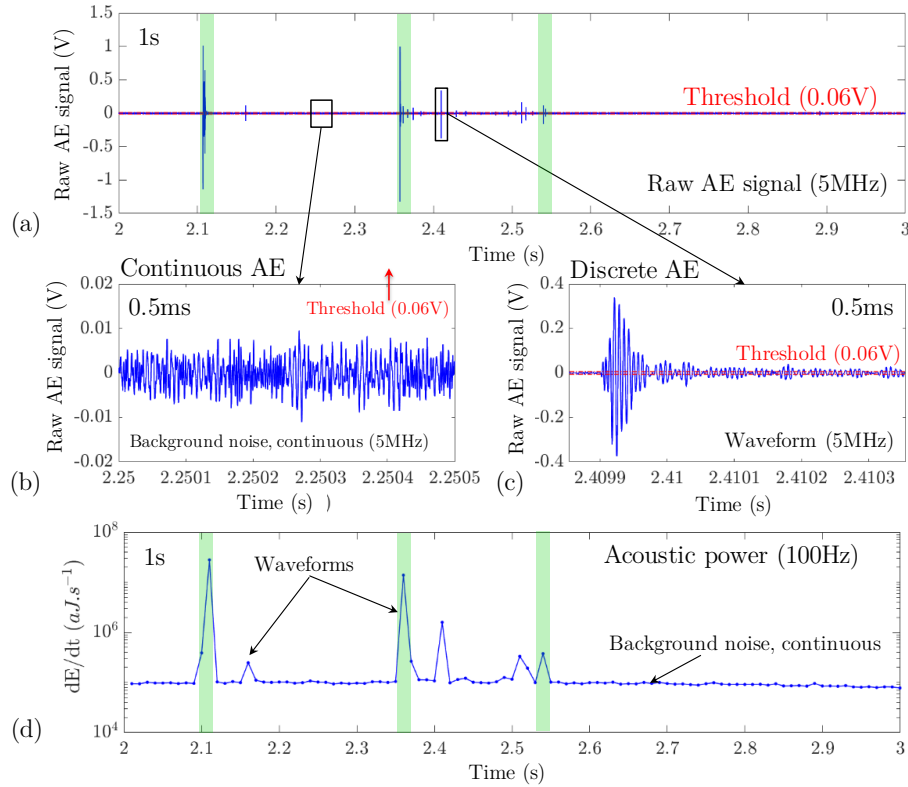


FIGURE 1.3 – Example of AE signal recorded during 1s of one cycle of a fatigue test [L'Hôte 2019]. (a) Raw AE signal (V) sampled at 5 MHz. (b,c) Enlargement of the "continuous" background and the so-called discrete part of the signal ((V) sampled at 5 MHz). Each discrete emission (WF is recorded separately, when the signal exceeds an imposed threshold (here 36 dB / 0.006 V, red). (d) Acoustic power dE/dt (aJ/S) recorded by the system, sampled at 100 Hz. This acoustic power reflects both the continuous and discrete parts of the raw signal.

waves themselves. I will talk about three types of elastic waves : the body waves, which travel through the interior of a material, surfaces waves which travel along the surface of a material and plate waves, which propagate in thin structures like plates.

Body waves

Body waves occur ideally in infinite media and include longitudinal waves and transverse waves. In seismology, body waves are the seismic waves that propagate through the interior of the Earth, and they arrive before the surface waves emitted by an earthquake.

In longitudinal waves, also known as compression waves or primary waves (P-waves), the particles of the disturbed medium are displaced in a direction that is parallel to the direction of propagation of the wave. This type of waves has the highest velocity and is therefore the first to be recorded if present.

In transverse waves, also known as shear waves, or secondary waves (S-waves) -the second wave you feel in an earthquake-, the particles of the disturbed medium are displaced in a direction that is perpendicular to the direction of propagation of the wave. The wave velocities are given by the elastic properties of the medium, and the S-wave velocity will be lower than the P-wave velocity. This result should also be valid for AE sources, and consequently P- and S-wave arrivals should be seen in AE waveforms if in far-field³ condition. But, experimental conditions in AE are not favorable for observing distinct P- and S-waves in a dominant portion of the cases [Unander 2004]. Large distances from sources can separate P- and S-wave components for instance when working on large structures at lower frequency.

Surface waves (mainly the Rayleigh waves)

Surface waves travel along surface of semi-infinite media or a relatively thick solid material (seismic surface waves travel along the Earth's surface). They combine both longitudinal and transverse mode to create elliptical motions, travel at a speed slightly lower than shear waves by a factor which is dependent on the elastic constant of the material, and may exhibit velocity dispersion in actual structures. More importantly, Rayleigh waves decay more slowly with distance than body waves, and are sensitive to surface defects when used for Non Destructive Testing (NDT).

Figure 1.4 shows a vertical component seismogram of the 1989 Loma Prieta earthquake recorded in Finland, showing P- and S-waves as well as surface waves. The time scale would be different in case of an AE waveform recorded in a specimen or structure.

Plate waves (mainly the Lamb waves)

Plate waves only propagate in thin structures like plates (which thickness is of the order of the wavelength) and consist of numerous modes. These modes can be

3. The far-field is where the source-receiver distance r is large enough for the $1/r$ terms in the wave field solution to dominate the terms proportional to $1/r^2$ and $1/r^3$ that also appear in the solution

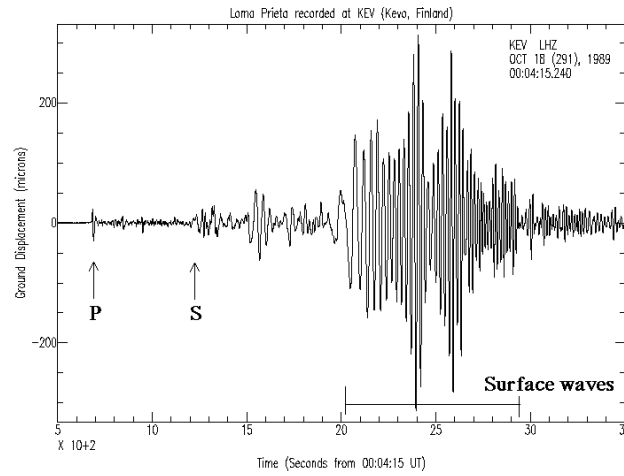


FIGURE 1.4 – A vertical-component seismogram of the 1989 Loma Prieta earthquake in California recorded in Finland. Plot taken from Princeton Earth Physics Project.

categorized into two families, i.e the symmetric modes (extensional modes) and the anti-symmetric modes (flexural modes). In third section, I will also mention the guided Lamb waves that propagate along elongated structures while guided by their boundaries, such as finite plates, cylindrical pipes etc. The prefix "guided" is to recognize that Lamb's infinite plate is, in reality, nowhere to be found.

Lamb wave theory is the prime theory for explaining the signal forms and propagation velocities that are observed when conducting AE experiments because of the shape of the specimens and structures tested.

1.2.1.4 Acoustic Emission recording

Within the framework of the research I have been able to carry out to investigate the behavior of materials under stress, AE has always been continuously monitored during the various mechanical tests performed. In most of the cases, a PCI2 Mistras data acquisition system of Euro Physical Acoustics S.A. (EPA) was used, with a 20, 40 or 60 dB pre-amplification and a $50\text{kHz} - 1.2\text{MHz}$ bandwidth and a 40MHz maximum technical sampling rate. Measurements are usually achieved with two resonant piezoelectric sensors, which are the most commonly used due to their high sensitivity and ease of implementation.

The sensors need some form of coupling medium to transmit the wave between the sensor's wear plate and the surface of the specimen that should be chosen according to the impedance between the two. Silicon grease or silicon glue (requiring 24 hours of drying), or even honey, are examples of wet couplants we use. The sensors are placed on the surface of the specimen or the heads and maintained thanks to Teflon tape or springs in tailor-made grips (as it can be seen in figure 1.2). We do not record the full acoustic signal over the entire tests duration, which can last several hours. Nevertheless, the raw AE signal, sampled at a frequency of 5 MHz (larger

than the resonance frequency of the AE sensors), can be recorded during limited time windows at specific occasions (during individual loading cycles in case of cyclic loading for instance as seen in figure 1.3(a)). We are working instead in an automated detection mode. Transient elastic waves are selected from the background noise above a given amplitude threshold⁴ A_{th} (figures 1.3 and 1.5) which varies depending on the test environment and the pre-amplification used ($\sim$ from 0.005 to 0.11V or from 34 to 40 dB, the conversion being made thanks to equation 1.1).

$$A(\text{dB}) = 20 \times \log_{10}\left(\frac{\text{Amp}(V)}{V_{ref}}\right) - G(\text{dB}) \quad (1.1)$$

where V_{ref} is the reference voltage (1μV) and G is the preamplifier's gain (e.g 20, 40 or 60 dB). The choice of this threshold is defined by the mechanical noise inherent to the machine and the surroundings.

The data acquisition system determines the AE signal arrival times, t_1 and t_2 , at sensors 1 and 2 respectively, corresponding to the first threshold crossing of the signal (figure 1.5). These arrival times are known with a precision of 0.1μs with an important consequence in terms of localization accuracy (see 1.2.1.7. and 3.2.3). Several parameters determined from the waveforms are recorded for each burst : maximum amplitude A_0 in dB, energy E_{AE} , rise time RT , counts C (number of threshold crossings), duration D , etc. (figure 1.5). The amplitude distribution covers the range between A_{th} and 10V, and the energy E_{AE} is obtained by integration of the squared signal divided by the reference resistance R (10 kΩ) over the duration of the WF :

$$E_{AE} = \frac{1}{R} \int_t^{t+D} V^2(t) dt \quad (1.2)$$

An ultrasonic transducer is used to generate well-known pulses, or a pencil lead break procedure [Nielsen 1980, Hsu & Breckenridge 1981] to simulate AE signals, in order to calibrate the tests, in particular to set up the acquisition parameters for our materials : peak definition time (PDT), hit definition time (HDT), hit lock time (HLT), time constants to individualize the waveforms.

Besides, it is important to notice that the recorded waveforms $W(f)$ result from the convolution of the source signal $S(f)$ and of transfer functions in the frequency domain depending on the acquisition chain : $W(f) = S(f) \times G(f) \times D(f)$. $G(f)$ describes the effects of wave propagation within the medium (usually studied using a Green's function approach) and $D(f)$ describes the characteristic response of the detection system (i.e. sensor and detection electronics).

A time-driven dataset is also recorded, where the total acoustic energy (aJ), the Root Mean Square (RMS) voltage (V) and the Average Signal Level (ASL) (dB) are sampled continuously throughout the tests at a sampling frequency of 100 Hz maximum. Hence, the data correspond to the sum of the raw signal over a fixed time

4. AE waveforms are sampled at a frequency varying from 1 to 5 MHz

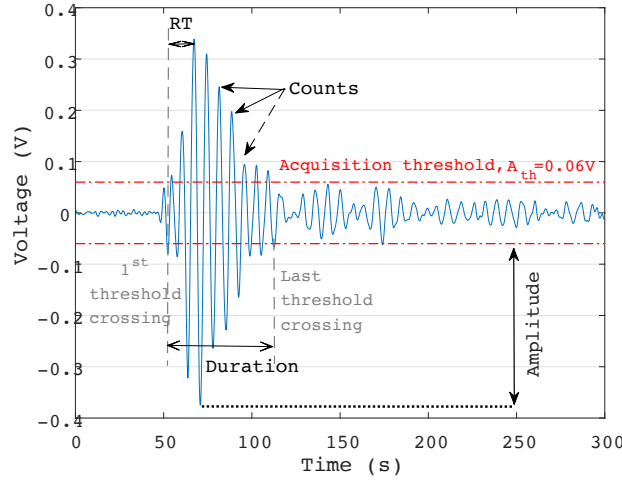


FIGURE 1.5 – Main parameters measured on an AE waveform

window ($dt = 10\text{ms}$ being the minimum time window possible by the acquisition system). The energy dE is defined by the integral of the squared voltage divided by the reference resistance ($10\text{ k}\Omega$), over the time window dt . The corresponding acoustic power dE/dt is thus defined : figure 1.3(d) shows the acoustic power associated with the raw signal of figure 1.3(a). As dt is large ($10\text{ms} - 1\text{s}$) compared to the duration D of detected individual bursts ($0.1\text{ms} - 10\text{ms}$), this AE power encompasses both continuous and discrete AE (if any individual burst arises during the time dt), the latter being, however, smoothed by the low sampling frequency.

In the crack growth experiments performed on scintillators at Queen's university, we do not use this system defining the WFs above a fixed threshold but the raw signal is recorded and well-separated events are determined when the power of the signal overcomes an adaptive threshold above the baseline.

1.2.1.5 Sensitivity of the AE technique

The sensitivity of the AE technique is high but determining the smallest detectable damage is not an easy task as it depends on a large number of conditions : the sensor itself, the impedance between the sensor's wear plate and the surface of the material, the material that is damping more or less the signal, the nature of the source, its orientation, the background noise over the frequency range of the detecting instrumentation, etc. Therefore, it is difficult to obtain an absolute figure for detection sensitivity for AE tests.

However, back in the 80's, Scruby and Wadley [Scruby & Wadley 1983] discussed the detectability of deformation and fracture processes in steels and mentioned a smallest detectable displacement of 10^{-14}m . Then, they deduce the weakest detectable source thanks to simple source models. For instance, stating that the elastic wave amplitude from the motion of dislocation is proportional to the distance moved

a and mean velocity v , if the detector is positioned at a distance h from the source, and has a displacement sensitivity threshold of x then $a.v > 10^{14} h.x \text{ m}^2\text{s}^{-1}$ for the dislocation to be detected. It means that, with a threshold of 10^{-13} m , the smallest detectable dislocation loop, at a depth of 100 mm, must have $a.v > 1 \text{ m}^2\text{s}^{-1}$. Even if the velocity were $\sim 3000 \text{ ms}^{-1}$ (the theoretical limit), the dislocation must propagate $\sim 0.3 \text{ mm}$ for detection. This is much greater than the distance usually moved by a single dislocation and can therefore be discounted as a potential emission source in most structures. Nevertheless, the cooperative motion of groups of dislocations is recorded : Mathis and Chmelik [Máthis & Chmelik 2012] estimated that a minimum of 10 – 100 dislocations must simultaneously move to produce sufficient energy to be detected.

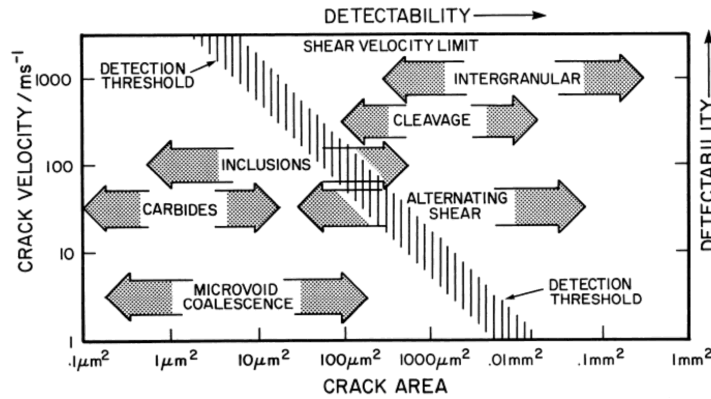


FIGURE 1.6 – Detectability of fracture events in steels. Detection threshold assumes transducer compression wave sensitivity of 10^{-13} m , source-transducer distance 0.1 m, stress 500 MNm^{-2} . Figure reproduced from [Scrubby & Wadley 1983].

Scrubby and Wadley [Scrubby & Wadley 1983] also discussed the case of microcrack growth, (with the same detection limit of 10^{-13} m displacement and a stress of 10^9 Nm^{-2}), crack advance at the shear wave velocity approximately of 3000 ms^{-1} in steel, would be detectable if the new crack area exceeded $\sim 2 \mu\text{m}^2$. At a more realistic velocity for a brittle advance, 500 ms^{-1} , the minimum detectable crack increment would be $\sim 10 \mu\text{m}^2$. It might therefore be possible to detect crack advance of about $1 \mu\text{m}$ in low alloy ferritic steels provided the advance takes place over more than $10 \mu\text{m}$ of crack front. But, we have to bear in mind that, during monitoring, ambient noise levels and hence detection thresholds may be far higher and more generally, there is likely to be a range of crack areas and velocities generating detectable emission. Scrubby and Wadley made an attempt to represent these in the form of a map that visually indicates the likelihood of detectability for steels (figure 1.6). All the estimates assumed an isolated microcrack.

1.2.1.6 Modal Acoustic Emission

An attempt to provide a better theoretical background for AE testing is Modal Acoustic Emission (MAE) [Gorman 1991, Prosser *et al.* 1995, Morscher 1999] : under conditions such that plate theory is applicable, it has been shown that plate waves are useful for understanding AE phenomena. A specimen, such as a plate, is considered thin if its thickness is of the order of the wavelength of the appropriate AE waves. AE waves propagate in a variety of modes whose separation at the sensors allows to extract the exact information about the source that produced the waves. For example, sound travels longitudinally (extensional component) through the sample or in flexure which produces different modes of the waveform. Figure 1.7 shows a simple schematic diagram of the two modes of travel through a material [Morscher 1999].

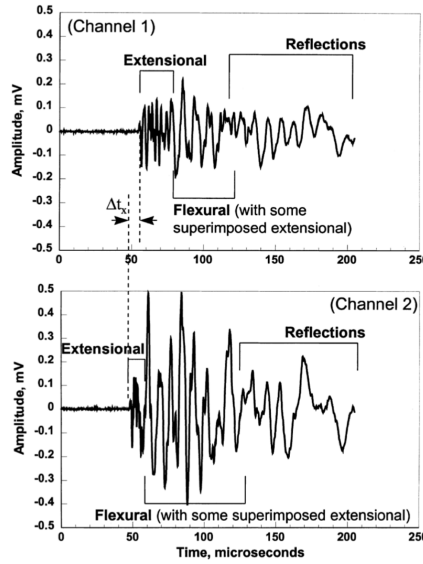


FIGURE 1.7 – WF produced from a lead break on a composite sample, nearest sensor 2 and received on the two channels (sensors). Figure reproduced from [Morscher 1999].

The initial extensional (propagating with a faster velocity) and flexural modes (slower velocity) of the waveform are thus exploited for identifying the source of the emission and its location.

I haven't used MAE as of yet but I wanted to present this approach as I intend to apply it in future studies of fatigue crack growth and for localization improvement.

1.2.1.7 Localization of AE sources

It is possible to determine where the release of acoustic energy took place thanks to the localization of the emissive sources using two or more sensors. Localization is classically performed from triangulation based on the arrival times of the same signal at the different sensors (time of arrival (TOA) approach). During the different

studies I undertook, only two sensors were used, in that case, only a 1D-localization is possible. The AE signals detected by the two sensors can be located along the specimen length when the difference of arrival time between sensor 1 and sensor 2, $\Delta t = t_1 - t_2$, is smaller than d/v (d being the distance between the two sensors and v the wave propagation velocity in the material). The position of the sensors being known, the location of the source is given by $p = \Delta t \frac{v}{2} + \frac{d}{2}$ on an axis connecting the two sensors.

An important parameter for the localization is the wave propagation velocity, which is measured thanks to artificial sources simulating AE sources : with an ultrasonic transducer generating well-known pulses or the rupture of a graphite pencil lead (Hsu-Nielsen source [Nielsen 1980, Hsu & Breckenridge 1981]). The mean wave velocity v is measured, for each sample studied, at room temperature (as examples, aluminum : 5000m.s^{-1} , 304L stainless steel : 5800m.s^{-1} , Cu alloy and Pure Cu : 3570m.s^{-1} , Polyethylene : 2000m.s^{-1}). Besides, the wave propagation velocity may change during a test with respect to the material damage [Morscher 1999, Morscher & Gyekenyesi 2002, Godin *et al.* 2018].

Another key issue for the TOA localization is the determination of the Δt , hence the measurement of the signal arrival time [Eaton *et al.* 2012] which strongly depends on the pre-set threshold as the system determines the arrival time with the point at which the signal amplitude exceeds the threshold level (figure 1.5). In some cases, this might be a problem as multiple signal peaks in the WF may exist prior to this point. In addition, the localization accuracy is impacted by the $0.1\mu\text{s}$ time precision of our system. To improve the accuracy on Δt , Grabec proposed, about 40 years ago, to cross-correlate the signals of a same event received by different transducers [Grabec 1978].

In the TOA approach, the signal propagation is assumed to be within a 2-dimensional plane and in our case, we assume the source to sensor path to be direct and uninterrupted and the propagation velocity to be constant in all directions and regions of the specimens (at least for the metal samples studied ; the case of semi-crystalline polymers is discussed in 2.1). It might be more complicated for complex materials and geometries and novel solutions for AE source location in geometrically complicated structures are developed, as for instance novel Δt mapping techniques (using artificial sources) that do not require knowledge of the sensor position or wave velocity [Baxter *et al.* 2007, Eaton *et al.* 2012, Holford *et al.* 2017].

A range of approaches to determine the arrival time of the signal and detect the first peak above background noise has been investigated as an alternative to the threshold crossing technique. A method based on high-order statistics [Lokajíček & Klíma 2006] (use of the derivative of the 6th order statistical moment of data within a sliding window) determine precise arrival time although the use of a threshold is still required. The threshold-independent technique based on the Akaike Information

Criterion (AIC) [Akaike 1974] to determine signal arrival times has been shown to be very reliable [Hensman *et al.* 2010, Kurz *et al.* 2005, Sedlak *et al.* 2009, Maillet & Morscher 2015]. The AIC-based technique compares signal entropy prior and after each time t (or data point) in a time series. The resulting function, called AIC function, reaches a minimum value at the time of the onset of the waveform when the similarity is a minimum between the high entropy uncorrelated noise prior to onset and low entropy structured signal after the onset. Figure 1.8 [Hensman *et al.* 2010] compares the AIC arrival time estimation (green line) with the first threshold crossing arrival time estimation (red line).

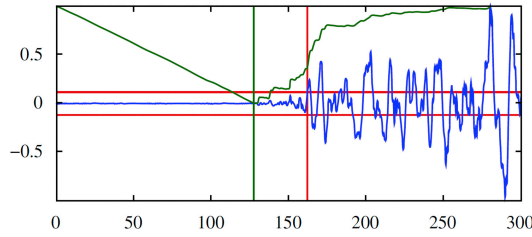


FIGURE 1.8 – Example of signal onset estimation using an AIC based approach. The horizontal and vertical red lines indicate the threshold level and the threshold based arrival time respectively. The green trend is the AIC function and the vertical green line indicates the estimated arrival time at its minimum. Figure reproduced from [Hensman *et al.* 2010].

Different approaches were also used for phase arrival determination such as ratio of Short-time average (STA) to Long-time average (LTA) [Baer & Kradolfer 1987], travel-time differences of the P- and S-wave onsets [Jurkevics 1988], neural networks [Zhao & Takano 1999] and wavelet transformations [Anant & Dowla 1997, Hamstad & Gary 2002, Yamada *et al.* 2000, Jiao *et al.* 2004].

Based on the modal nature of AE, the arrival times can be determined on the different modes at one sensor, enabling source location to be performed with a single sensor only. The single sensor studies usually combine several approaches : wavelet transform, dispersion curves of the fundamental Lamb wave modes, reflections from boundaries ... [Jiao *et al.* 2004, Ebrahimkhanlou & Salamone 2017]. Otherwise, new formulations are developed for localizing acoustic sources in a large anisotropic plates using several AE sensors, not requiring the knowledge of the wave velocity [Kundu *et al.* 2012]. And, deconvolution of acoustic emissions can be used for source localization using time reverse modeling [Kocur 2017].

To go back to the studies I undertook, we have used, in most cases, the traditional threshold crossing technique, given by the system, checking each WF when necessary, to determine the exact time of arrival from the time of the first peak, the small amount of data allowing it. Yet, I will also present the limitation of this technique through the study of fatigue of metals and I will present a new way to localize in

section 3.2.3.

1.2.1.8 Ultrasonic versus Acoustic Emission technique

The acoustic emission technique differs fundamentally from the ultrasonic technique. While **AE** is a passive and global technique, Ultrasonic testing (**UT**) is based on the propagation of ultrasonic (**US**) waves in the material tested, controlled by the operator. An elastic wave generated by a transmitter propagates through the material, is reflected and transmitted at impedance contrasts and is finally recorded by a receiver. The operator choose to study the propagation of such or such type of wave (see section 1.2.1.3. For instance, through Nicolas Casiez's PhD thesis [2012-2015] [Casiez 2015, Casiez *et al.* 2017], we used both guided and longitudinal waves to characterize the plasticity and damage of several Polyethylenes (PE) during tensile tests. In the case of **AE**, the operator does not have control over the waves he is trying to detect as they are generated by local micro-displacements occurring in the material.

For **UT**, very short **US** pulse-waves with center frequencies usually ranging from $0.1 - 15\text{MHz}$, and occasionally up to 50MHz , are transmitted into materials to assess the damage due to previously occurred degradation (detect, quantify and characterize defects and heterogeneities present in a structure) or to characterize materials (determine the sound velocity, density, Young's modulus, thickness etc.). The different material properties and information derive from **US** parameters : while the wave propagates through the material, **US** parameters are significantly affected by changes in microstructural or mechanical material properties. These parameters are, firstly, the amplitude of the wave and, secondly, the so-called time-of-flight, i.e. the time the ultrasonic wave needs to propagate from the transmitter to the receiver. Piezoelectric transducers can be used to generate **US** waves : a couplant is used in order to improve the mechanical contact between transducer (in which the wave is generated) and component. The transducers are capable of exciting different wave modes, selected depending on the application.

We have used the combination of the two techniques **AE** and **UT** to obtain new information about the initiation of plastic deformation and damage of semi-crystalline polymers (see section 2.1).

1.2.1.9 When **AE** meets Artificial Intelligence (**AI**)

AI is everywhere nowadays [the term often used to describe machines (or computers) that mimic "cognitive" functions that humans associate with the human mind, such as "learning" and "problem solving"]. However, if you repeat a word enough times, it eventually loses all meaning. For many, the phrase "artificial intelligence" might have fallen apart in this way as it is used endlessly. Nevertheless, **AI** is in fact a very powerful tool to overcome both the complexity and/or the large amount of data to be treated. For **AE** it means developing new algorithms able to

automatically define, compute, detect phenomena linked with AE. At Mateis lab, several AE approaches have already been developed without using the term "artificial intelligence" but with the same ambitions : for instance, to discriminate the AE signatures of different source mechanisms [Shaira *et al.* 2008] ; in AE pattern recognition and signal source mechanism identification [Godin *et al.* 2004, Moevus *et al.* 2008, Sibil *et al.* 2012, Morizet *et al.* 2016] and towards rupture time prediction [Maillet *et al.* 2012, Loutas *et al.* 2017, Godin *et al.* 2019].

I will present in chapter 4, different future projects where AE technique is combined with AI. One where the objective is to predict the lifetime of corroded samples under fatigue loading : building a new model to estimate the remaining useful lifetime (RUL) of corroded samples or to predict the number of fatigue cycles before corrosion is initiated. And another project where one of the objectives is to propose a new, reliable non-destructive on-line monitoring of the onset of fatigue cracking based on machine learning algorithms during mechanical tests or within structures in-service. To overcome these challenges, and inspired by recent developments in seismology, we will explore data mining and machine-learning methodologies.

1.2.2 Sight : electromagnetic waves

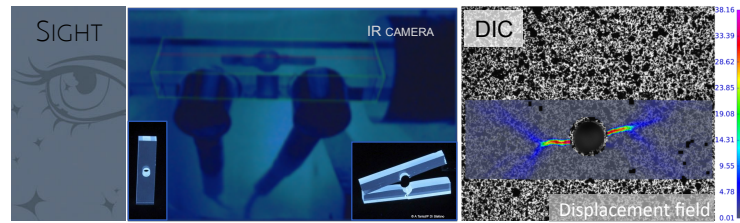


FIGURE 1.9 – SIGHT

The sense of sight, is based on the transduction of light stimuli received through the eyes which contributes to visual perception. This sense might be the one everyone think of first to observe phenomena. Besides, how many times have we read the expression "to shed some light on" in scientific articles ? "To reveal information or details about something ; to clarify or help people understand something" : isn't it the whole idea of research ?

I will use this sense to present the techniques related to electromagnetic waves (light) or any direct observation that I employed during my different studies. Unlike for AE (related to pressure waves) which sets the guiding thread of my research, I will not explain in detail the different techniques, I will only mention them instead. The techniques are always operated simultaneously with the AE technique.

1.2.2.1 Digital Image Correlation

DIC has been performed on the one end to measure specimen strain during fatigue tests (over the study of dislocation dynamics during cyclic loading in copper

single crystal, Gabriel L'Hôte's PhD thesis [2016-2019] [L'Hôte 2019], chapters 2 and 3), and on the other hand to monitor fatigue crack growth (figure 1.9 on the right, chapter 4) .

DIC enables displacement field measurements $u(x)$ to be performed at the specimen surface, assuming conservation of the optical flow, between an image of reference and a deformed image. Compared to traditional strain gages and extensometers, the amount of information gathered about the fine details of deformation during mechanical tests is increased due to the ability to provide both local and average data using DIC.

During Gabriel's PhD thesis, DIC was used as a strain gauge [L'Hôte *et al.* 2019a] (figure 1.10). Indeed, because of the high ductility of copper single crystal, classical contact extensometers could not be used, as their use would induce localized damage on the surface as well as acoustic noise that could be recorded by the AE set-up. A region of interest (ROI) of $\sim 3.3 \times 3.8 \text{ mm}^2$ was defined as the gauge, with the strain tensor being the expected result. Images were recorded using a camera (mv-BlueFOX 124C, MATRIX VISION) at a frequency of 3.2 Hz . Only the mean axial strain E_{yy} (average of the values in the ROI) is exploited. The strain resolution was approximately 0.002%. To attain sufficient contrast, a speckle pattern was painted on the specimen surface using black and white paint.

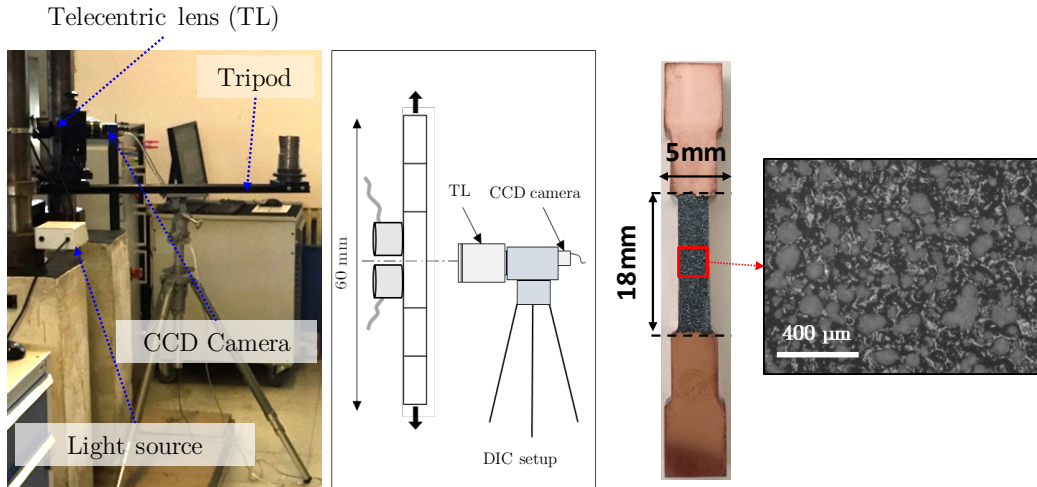


FIGURE 1.10 – DIC setup and speckle pattern [L'Hôte 2019]

DIC also enables the detection of cracks (of length a), the follow-up of their propagation and the computation of the Stress Intensity Factor (SIF) $K_I = \alpha \sigma_a \sqrt{\pi a}$ at crack tip [Réthoré 2015]. K_I is the SIF for mode I loading, applied to the crack opening mode, with α a geometric correction factor and σ_a the applied stress. And, actually, DIC is largely exploited for crack growth monitoring, damage measurement, etc. and is often used with AE (e.g. [Verbruggen *et al.* 2015, Aggelis *et al.* 2016, De Sutter *et al.* 2017] for structural analysis or monitoring of failure mechanisms for instance.

1.2.2.2 Microscopy

Through Wafa El May's PhD thesis [2010-2013] [El May 2013], on the study of plasticity and damage of aluminum during low cycle fatigue, DIC hasn't been implemented (classical extensometer were used to measure and impose strain) but we have used Scanning Electron Microscopy (SEM) for the examination of the surface of aluminum samples (figure 1.11). SEM observations of the surface were performed during cyclic strain-controlled tension-compression tests, interrupted at various stages of fatigue life. The different stages of cyclic behavior could be observed [Weiss *et al.* 2018] : from slip lines on few grains, the activation of additional slip systems, well-defined Persistent Slip Bands (PSBs), up to signs of crack nucleation, crack propagation and even fatigue striations on crack surfaces [Deschanel *et al.* 2017]. I will present the monitoring of different fatigue stages with AE in section 1.3.2.

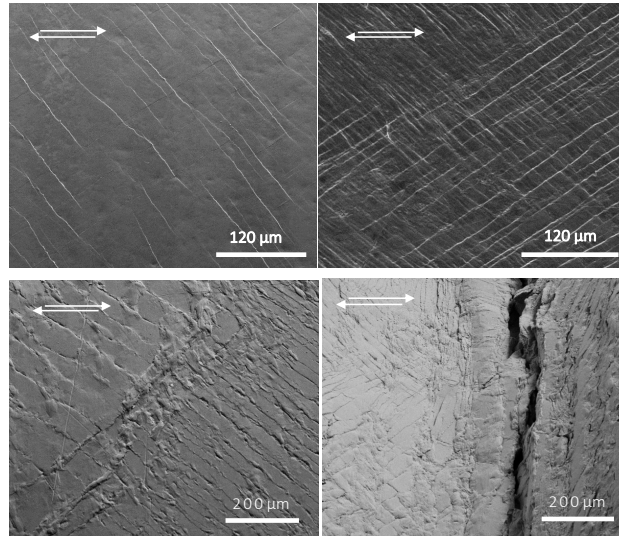


FIGURE 1.11 – SEM observations of the surface of an Al sample during a strain-imposed fatigue test ($\Delta\epsilon = 0.95\%$) interrupted at various stages : (a) 10 Cycles (slip lines on few grains, in a single-slip configuration) ; (b) 100 cycles (double-slip within few grains) ; (c) 1100 cycles (crack initiation along PSBs) and (d) 2100 cycles (fatigue cracking). The white arrows indicate the tension-compression loading axis.

Through Gabriel's PhD thesis, we used EBSD and ECCI techniques to perform microstructural characterizations [L'Hôte *et al.* 2019a]. As it can be seen through an example in figure 1.12, the EBSD technique provides information on the evolution of crystal disorientation (in terms of degree of disorientation and deformation localization) in the studied samples during fatigue tests. The ECCI enables to observe dislocations on the surface of the samples and thus to know the dislocations arrangement formed and to determine their characteristic sizes (for example, the mean

free path⁵).

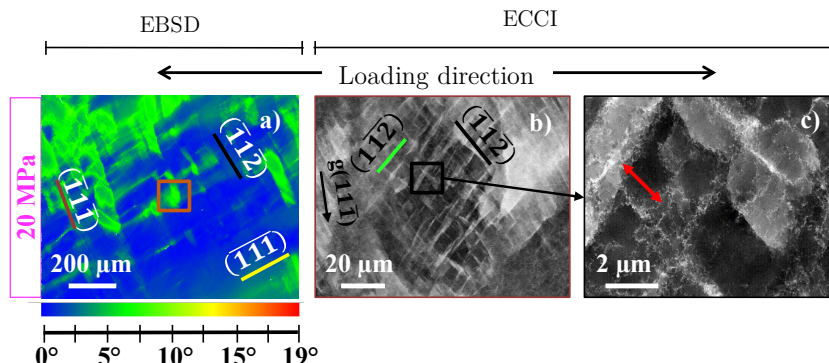


FIGURE 1.12 – Microstructure characterization during a fatigue test on pure Cu at imposed stress [L'Hôte 2019]. a) Texture component from EBSD maps. b and c) Dislocation structure from ECC-Images (dislocations appear in white). The red arrow shows how the mean free path is measured.

ECCI is a technique performed in a SEM, that we have used for dislocation characterization of bulk samples [Coates 1967, Zaefferer & Elhami 2014]. This technique is increasingly used because of the simplified and non-destructive sample preparation compared with that for Transmission Electron Microscopy (TEM) analysis. For both TEM and ECCI, dislocations are visible when the crystal is slightly deviated from the "two-beam" condition orientation. This condition consists of fulfilling the Bragg condition for one family of planes by tilting and rotating the sample relative to the electron beam. As this orientation can be difficult to achieve, we developed during Gabriel's PhD thesis, a novel approach based on the rotation-acquisition of Back Scattered Electrons (BSE) images : R-ECCI. In such an image series, specific angles should result in adequate two-beam conditions to observe dislocations. EBSD maps were obtained using an Oxford Instrument Symmetry detector. Dislocation observations were conducted on a classical SEM (Gemini I column, Carl Zeiss Microscopy GmbH, Germany)). The EBSD acquisition and R-ECCI observation conditions are described in the article "Rotational-Electron Channeling Contrast Imaging analysis of dislocation structure in fatigued copper single crystal" [L'Hôte *et al.* 2019b] that I will not develop in this HDR manuscript. In this configuration, the spatial resolution achievable by ECCI is 10 – 20nm [Zaefferer & Elhami 2014].

1.2.2.3 X-ray tomography

One of the best methods for imaging a damage process is synchrotron X-ray tomography which shows a non-destructive image in the bulk and without bias due to surface preparation and/or surface stress state [Buffière *et al.* 1999, Babout

5. distance over which a dislocation can travel before being stopped by dense dislocation arrangement

et al. 2004]. Tomography is widely developed and exploited at Mateis lab, in particular, the potential of X-ray tomography has been evidenced for studying the mechanical behavior of structural materials through in situ experiments in various experimental conditions [Buffiere *et al.* 2010]. Dedicated experimental devices were developed, allowing deformation and/or temperature changes to be applied to various types of materials while tomography is performed. High resolution synchrotron X-ray micro-tomography have also shown much promise for the study of fatigue cracks in metals with a fatigue machine used for in situ cycling of samples [Buffiere *et al.* 2006].

Moreover, we have already performed in the past, simultaneous AE and fast tomography experiments during tensile tests on Polyurethane (PU) foams [Deschanel *et al.* 2009] where we could relate directly AE events to specific physical events. AE and fast tomography have also been combined without stopping the tensile test, helping to obtain a more complete understanding of the damage evolution leading to the rupture of the sample [Maire *et al.* 2007].

1.2.2.4 Fractoluminescence : emission of light from fracture

Through Alexis Tantot's PhD thesis [2012-2015] [Tantot 2015] and over my stays at the Department of Physics, Queen's University, Canada (January-August 2013 and September 2014-March 2015), we studied fractoluminescence, by coupling scintillation particle detection techniques to AE : correlation of acoustic and light emission during fracture of materials (note that one wave relies on matter and not the other one). [Tantot *et al.* 2013, Tantot *et al.* 2016]. Fractoluminescence has its rightful place in this section related to the sense of sight as the luminescence is the emission of light by a substance which is not caused by incandescence (black-body radiation). Multiple forms of luminescence exist, they are sorted based on the origin of the light emission. The photoluminescence is a type of luminescence due to the absorption of photons. It can be divided into two phenomena : the fluorescence and the phosphorescence. The fluorescence is the "instant" emission of light when a substance is exposed to an exciting light source, this process lasts about ten nanoseconds. The phosphorescence is the "persistant" emission of light after the exposure of the substance to an exciting light source, it can last from a few milliseconds up to several hours. The phosphorescence is observed in everyday-life since all the objects which glow in the dark rely on this phenomenon. We are interested in another type of luminescence : the mechanoluminescence - the emission of light by a solid subject to mechanical stress.

The first scientific report of mechanoluminescence goes back to the 17th century. In 1605, Francis Bacon reported that sugar emits light when scrapped with a knife [Bacon 1605]. Thus, the phenomenon of mechanoluminescence came quite early to the attention of the scientific community (in the late Renaissance) but it was probably already known for centuries by the workers processing sugar canes in South Asia. From this first report until now, mechanoluminescence has been observed in a large variety of materials [Jha & Chandra 2014]. And, as an example, Dubernet

et al. evidenced a linear dependence of the mechanoluminescence intensity with the mechanical power [Dubernet *et al.* 2015]. Since multiple types of mechanical excitations can lead to the emission of light, the mechanoluminescence is divided into several categories. Our study focuses on a specific type : the fractoluminescence, the emission of light caused by the fracture of a material.

Fractoluminescence has been reported in multiple material such as sucrose [Dickinson *et al.* 1984], ice [Mizuno & Mizuno 2002], magnesium oxide (MgO) [Langford *et al.* 1987], mica [Obreimoff & Kapitza 1930] or pressure-sensitive tape [Camara *et al.* 2008]. According to the survey literature by P. Jha [Jha & Chandra 2014], 50 % of all organic molecular solids and inorganic salts exhibit fractoluminescence. This phenomenon adds a new level of complexity to the fracture mechanism which is not described by common fracture theories. Back in 1930, Obreimoff -a contemporary of Griffith - observed that light was emitted by electrical discharges during the failure of mica at ambient pressure [Obreimoff & Kapitza 1930]. When carrying out the same experiment in vacuum ($10^{-4} - 10^{-5}$ mbar), he indirectly observed the emission of X-rays. X-rays photons are an evidence that highly energetic processes occur at the crack tip.

On the atomic scale, the fracture can be compared to a cataclysm. In MgO for example, as the crack tip goes forward, a cloud of electrons, ions and neutral particles is generated and photons are emitted [Langford *et al.* 1987, Dickinson *et al.* 1986]. These emissions are called fracto-emissions. The phenomenon of fracto-emission is generally not limited to the moment of failure, it also extends after the failure. It is the testimony of the highly excited states of the freshly created fracture surfaces. Mechanical activities such as dislocation motion, microcracking or defect production can also involve fracto-emission prior to failure in some materials [Langford *et al.* 1987].

Our study especially focuses on the fractoluminescence as a part of the fracto-emission. As it has been shown with experiments in MgO, the time distribution of the light emission demonstrates the complexity of the mechanism : the fractoluminescence is not directly related to the creation of new surfaces because light is observed prior to and after fracture [Langford *et al.* 1987]. The fractoluminescence is the combination of multiple processes which depends strongly on the material and are not fully-understood yet.

Our article "A multi-channel setup to study fractures in scintillators" [Tantot *et al.* 2016] describes in detail the home-made experimental apparatus, fully dedicated to the study of fractoluminescence. I will discuss the study of sound and light emission from fracture in an inorganic scintillator bismuth germanate (BGO) ($Bi_4Ge_3O_{12}$, brittle material) in section 3.3. As a matter of fact, a scintillator is a material which emits light when excited by ionizing particles (ultraviolet (UV), X-rays, γ , etc). We can see a BGO specimen under UV light in figure 1.9 in the center. Its scintillation emission maximum is situated at 480nm, in a range going from 350nm to 650nm.

The multi-channel setup (figure 1.13) is built around samples of double-cleavage

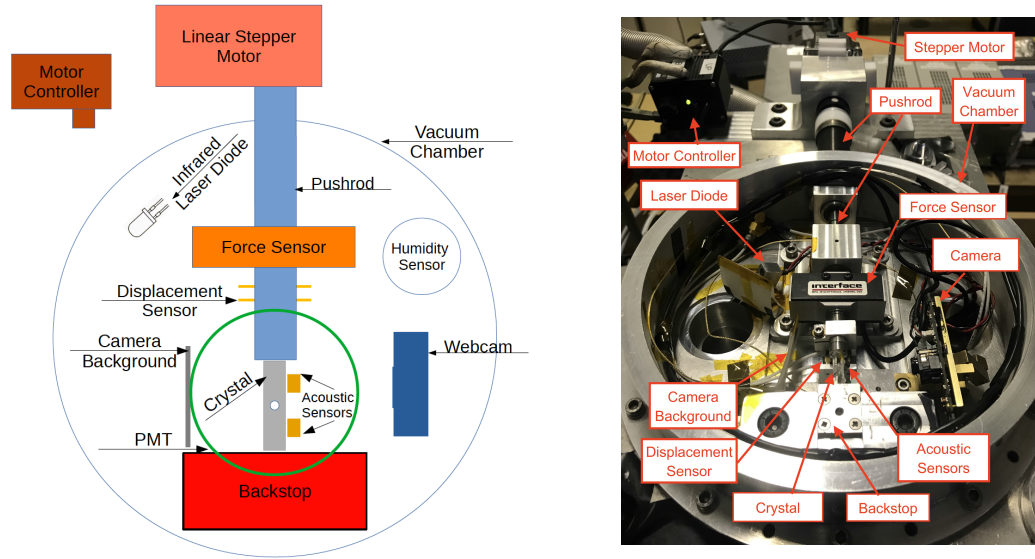


FIGURE 1.13 – Overview of the multi-channel setup used to compress DCDC samples [Tantot 2015].

drilled compression (DCDC) geometry, in order to better control the rupture process [Janssen, Ch. 1977], in a controllable atmosphere (vacuum, nitrogen ...). The DCDC geometry consists of a rectangular bar with a circular hole in the middle of the larger face. Uniaxial compression is applied on both bar ends. Because of the hole, the compression induces tension forces on both sides, which propagate a crack along the length of the bar. The combined effects of the restoring force⁶ between the crystal sides, and the compressive stress applied, allow to control the crack velocity up to a critical length, at which point, the restoring force is too low and the specimen breaks abruptly.

The setup, represented in figure 1.13, allows the continuous digitization over hours of various parameters, including the applied load, and the compressive strain of the sample (measured by a custom-made displacement sensor), as well as the AE. A photomultiplier above the sample measures the visible light emitted, with nanosecond resolution. Simultaneously, the propagation of cracks in the sample is observed using a near-infrared camera and a near-infrared light source (figure 1.9 in the center) (filtered and not visible by the photomultiplier).

In order to allow accurate light measurement and to control the atmosphere, the sample, and most of the instruments, are located in a sealed chamber. This enclosure is light-tight, with the exception of a quartz viewport for the photomultiplier tube, and has been tested at vacuum pressures down to 10^{-5} mbar.

6. We call "restoring force" the elastic force between both sides of the specimen ($20 \times 2.5 \times 3\text{cm}^3$) which tends to close the created cracks.

Clearly, there are a large number of other techniques but my point is not to draw up an exhaustive list here. I will only mention in addition the infrared thermography (IRT) technique. IRT has been successfully used in combination with AE to monitor mechanical damage in structural materials and follow crack propagation during fatigue tests [Kordatos *et al.* 2012, Kordatos *et al.* 2013, Barile *et al.* 2016]. It was also used to investigate the thermal response and dissipative mechanisms under ultrasonic fatigue loading [Torabian *et al.* 2016, Torabian *et al.* 2017]. In the case of the irreversible process of fatigue damage, part of the total energy is dissipated in heat which contributes to a local rising of the temperature (frictional heating as a result of differential movement at cracks). I related IRT to the sense of sight as it is a graphical representation of heat : temperature distribution on the surface of the specimen. It is also an electromagnetic emission, as visible light. Note that it could also be related to the touch sense, as we are able to feel heat as we touch matter. There is a chance that we will use this full-field technique in the future, being particularly suitable for the study of damage development during fatigue loading and for the quantification of the energy emitted during damaging processes.

1.2.3 Touch : roughness

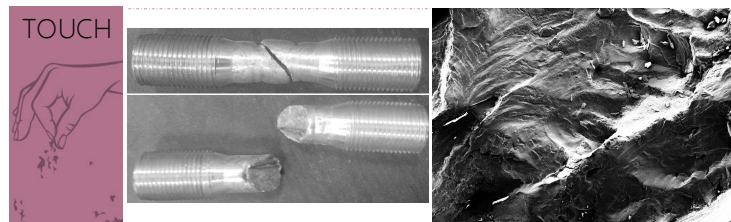


FIGURE 1.14 – TOUCH

Touch is perhaps the most overlooked sense although every one of us receive tactile information about the world around us every second of the day. The one I think of, relevant to material science, is related to the surface texture/ topography and surface roughness. (I didn't use Atomic force microscopy (AFM) where the information is gathered by "feeling" or "touching" the surface with a mechanical probe.) Fractography can be really useful in fracture studies as it analysis the fracture surfaces in order to determine the relation between the microstructure and the mechanism(s) of crack initiation and propagation and, eventually, the root cause of the fracture. Fracture surface analysis can also identify the amount of residual stress, if any, on the surface during fracture, and the existence or not of environmental slow crack growth before catastrophic failure.

Fractographic studies have played (and continue to play) a pre-eminent role in understanding fatigue crack growth [Pelloux *et al.* 1979, Putra & Schijve 1992, Lynch 2017, Chai *et al.* 2017, Wang *et al.* 2019]. Figure 1.15 illustrates an example where specific markings were correlated with specific variable-amplitude loading

events [Cottell 1954].

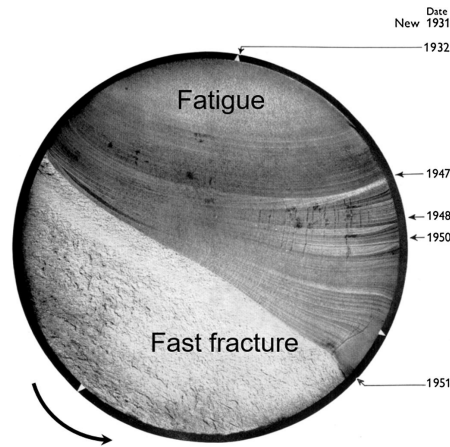


FIGURE 1.15 – Macroscopic view of fatigue fracture surface of a steel shaft that failed in 1951, showing progression markings, some of which could be dated to specific loading events, followed by fast fracture. [Cottell 1954].

As examples, progression markings are now commonly used to determine crack-growth rates in structures where load histories are known [Barter *et al.* 2009]. There was also recognition in the early fatigue literature that the relative areas of fatigue and fast fracture, and their respective geometries, gave an indication of the stress-level and stress-mode (e.g. plane bending, reversed bending, rotating bending, tension-compression) [ASM 1986]. More recently, as an example, the transition from stage IIa to IIb in the Paris [Paris 1961] region of fatigue crack growth has been identified thanks to fractographic observations and by the change of slope in AE cumulative count and cumulative energy vs. the SIF range ΔK [Chai *et al.* 2017].

During Wafa’s PhD thesis, we have studied fracture surfaces of pure Al subjected to cycling loading. The appearance of the fracture surfaces is reminiscent of the ductile nature of the pure Al being studied (figure 1.14). We monitored the slow, incremental propagation of a fatigue crack at each cycle, leaving striations on crack surfaces (figure 1.16). Those fatigue striations spacing gives information about the crack length increment and can be correlated with the local crack growth rate da/dN [Williams *et al.* 2011].

Furthermore, as examples, Daniel Bonamy studied the possible sources of crack surface roughening and the structure the growing crack creates in brittle amorphous materials and brittle heterogeneous materials [Bonamy & Ravi-Chandar 2005, Bonamy 2009]. He also studied the surface fracture patterns depending on crack velocity [Hattali *et al.* 2012] and the microcrack post mortem patterns to understand fast macroscale fracture [Guerra *et al.* 2012]. Also, Laurent Ponson *et al.* showed that statistical properties of crack roughness could be used as a probe of the microscopic failure processes if their complex geometry can be deciphered [Ponson 2016]. They use the statistical description of the roughness of cracks for exploring the dissipative

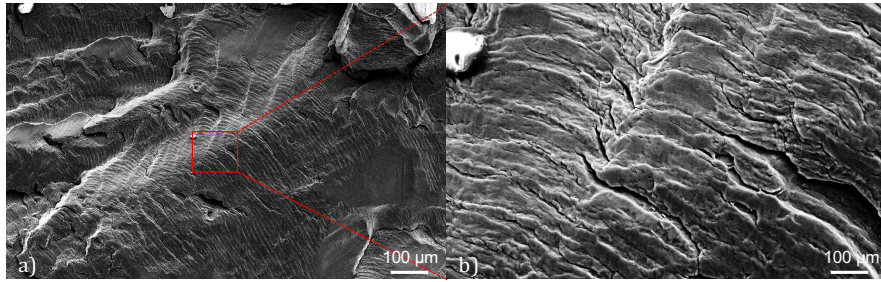


FIGURE 1.16 – Fatigue striations, unevenness and roughness of the surfaces on a fracture surface after 3000 cycles (fatigue test on pure Al at $\Delta\epsilon = 0.95\%$) [El May 2013].

failure mechanisms and tracing back the history of the failure of a material. They also present that the statistical characterization of ductile fracture surface roughness may be used for a post-mortem estimate of fracture toughness [Ponson *et al.* 2013].

1.2.4 Smell : in need of a keen nose

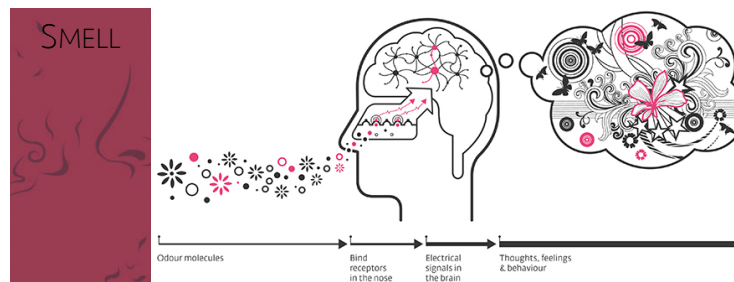


FIGURE 1.17 – SMELL

Smell is chemistry which can definitely be used to characterize materials, however I did not use it in my research. The sense of smell manifests through the nose. "To be nosy" : "given to or showing an intrusive curiosity about the affairs of others" ; "mettre son nez partout !" in French. Not to mention the intrusive side, being curious is of utmost importance in research.

"To have a keen nose" is an expression meaning to have intuition, which is also very necessary in research, although it is not always straightforward to trust one's intuition and it does not always lead to what we were expecting. But is it a bad thing? The nature of any analysis undergone is that one question leads to another. And little by little, with the help of the literature, by chance or determination we sometimes understand a small part and we give our contribution to the field. Anyhow, questions always need to be asked and formulated clearly to set the research objectives.

Similarly, the role of intuition is very important in the teaching process. I realized it is not an easy task to supervise PhD or master's students and my idea is to find the proper balance between letting them work independently and giving

them strong guidance. As a matter of fact, the idea is to understand and teach the intuitive mind, which always holds interest for me but is not always obvious! And most importantly, the intention is to work with them as a team, trying to erase the sense of hierarchy. From my point of view, supervising a PhD student is to coach that person on how to conduct scientific reasoning and how to map out the steps that lead from an idea to a practical result.

Regarding undergraduate students' teaching, we are usually competent in our specific subject discipline, but, as associate professor, we have never been trained to be pedagogues! The calling of teachers is not only to transmit knowledge at any cost but to adapt an educational approach tailored to each student respecting their difference. However; in the French education, it is not easy to do so with groups of 25 during one or two hours of tutorials. Hence, a key point could also be to develop autonomy. It is very important that students should make mistakes in order to learn and that they do not just follow a detailed correction. For this reason, we must give them the time to think by themselves first, but it is unfortunately not always possible because of time constraints and overloaded year programs.

In the same vein, practical work is really beneficial for autonomy as the students can apprehend the theory in a different manner with the model/experience confrontation : a first taste of research! And actually, intuition has been proven to be a valid source of knowledge acquisition in the field of physics, hence let them look for the problem on their own!

Finally, becoming a good teacher is a learning process that I believe never ends. A teacher is in a continual state of learning, building and refining teaching practices.

1.2.5 Taste ... for research



FIGURE 1.18 – TASTE

It might be obvious but a taste for science and for teaching is necessary for an associate professor. It is a fascinating profession : the more we know, the more we want to know but also the more we learn, the more we discover how little we know! The figure 1.19 represents a graph of how confidence to speak on a subject develops depending on experience and knowledge. It is such a privilege to keep

on observing, monitoring, classifying, hypothesizing and seeking "scientific truth". Most of the researchers are passionate about their job, which also means to me that it is difficult to step out of it : the project I am working on, this HDR I am writing, that article I want to finalize... it is all in my head all of the time !

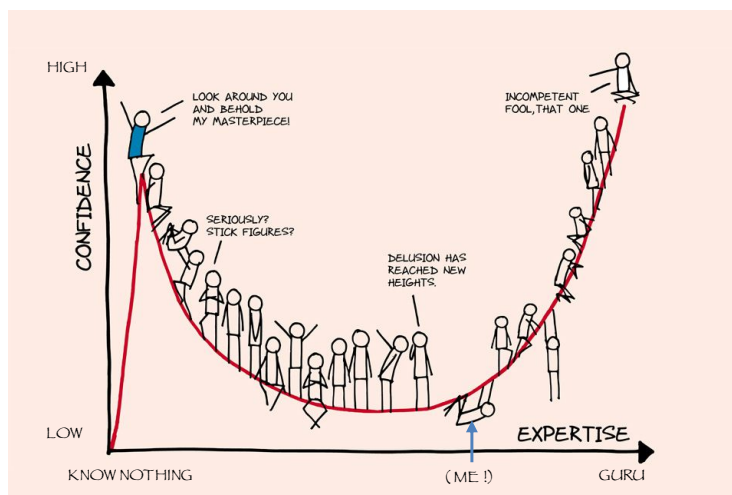


FIGURE 1.19 – Diagram representing the learning curve of a competence in relation to the self-assessment in this competence. According to the Dunning-Kruger effect [Kruger & Dunning 1999], people tend to overestimate their cognitive ability until/unless their competence increases to the point where they become aware of their shortcomings.

Some people have a vague idea of what the profession of "teacher-researcher" can be like. Many people seem to fantasize about how great a boss-less life must be and it's true that, in academia, you mostly have the opportunity to choose which field to work on and whom to work with, you can manage your own schedule and workload (on the research side only, as teaching does impose, for obvious reasons, a schedule). you are part of a team among your peers. There are also a couple of downsides that come along for the ride. It might seem that you don't have anybody telling you what to do and when to do it. However, you are sometimes evaluated with odd and suboptimal criteria (such as the h-index). This leads to the "publish or perish" crisis which is detrimental to good research.

Speaking of "publish or perish", I would like to mention the lucrative scientific publishing market that prospered on that race for publication. I think that the mechanism of publication, which originally allows knowledge to circulate and improve over time, has sadly become a real economic preserve : that of scientific publishers (a few big names share a juicy market and privatize scientific works, most of them financed by public funds). I really like the French video [#datagueule63](#) presenting this issue, although it is quite old (4 years ago). This is slowly changing today though, as many researchers criticize this publishing scheme and advocate open access journals. As a matter of fact, my colleagues just opened a scholarly

journal in the field of Solid Mechanics, [JTCAM](#), provided on a Fair Open Access basis, without any cost neither for readers nor authors. Moreover scientific institutes refuse more and more to pay outrageous fees to publishers for articles their own researchers wrote. I know there are pros and cons on the subject and that it is a perplexing question but I want to believe that Open Access returns us to the values of science.

On the teaching side, I would like to provide the undergraduate students with the opportunity to experience a taste of research through a method of discovery, solving problems, trial and error approaches, experiments etc. so that they develop their taste for science! We should stimulate their curiosity and let them make mistakes but also we should not be afraid to make mistakes ourselves as a teacher, which can be scary if we don't have the tutorial under control. But again, I do think we are not trained enough for teaching. What strikes me, event after 10 years, is that our teaching system operates on a voluntary basis (responsibility of this course module or that semester) but little acknowledgement. How lucky are the faculties to have such dedicated employees!

Furthermore, it might be complex not to have someone you can lean on for help and inspiration, especially at the beginning of the career when you try to get fundings ,while trying to cope with all the new teaching you need to produce and master. That is why I think we would all benefit from a mentorship in the first years of our careers to overcome theses difficulties.

After all these considerations on the profession, I would like to add a quick note related to the sense of taste as it necessarily brings us back to food and I must admit, I am really passionate about cooking, younger, I was really curious about molecular gastronomy, the sub-discipline of food science that seeks to investigate the physical and chemical transformations of ingredients that occur in cooking. I want to give two names : the French INRA chemist Hervé This whose book I loved and Ferran Adrià, the famous head chef of ElBulli (the famous Michelin 3-star restaurant near the town of Roses, in Spain, closed in 2011 and reopened as a creativity centre in 2014). During my PhD thesis, I was dying to go there but you need to book 2 years in advance and who knows where one is going to be two years ahead ?? Not me, even more so when I was a PhD student. Why am I saying all this? Maybe because it is part of my fascination for science and research, which is like finding the ultimate recipe, adding a little bit of salt, a little bit of scallions or honey to reach the perfect *taste*.

1.2.6 The sixth sense

After having talked about the five senses to carry out a research theme, we could wonder if there is a sixth sense driving us.

The sixth sense is defined as a power of perception seemingly independent of the five senses, i.e an extrasensory perception ([ESP](#)) including claimed reception of

information not gained through the recognized physical senses, but sensed with the mind. The term was adopted by psychologist J. B. Rhine to denote psychic abilities such as intuition, telepathy, psychometry, clairvoyance, and their trans-temporal operation as precognition or retrocognition. The sixth sense is indeed synonymous with keen intuition, which I already mentioned earlier in section 1.2.4 and which I think is an important tool in conducting research. Or, the sixth sense could be an unconscious integration of all five senses : the recipe for successful research ?

I can see from here the drop of sweat forming on the brow of the scientific reader, fearing that I might lead him into a supernatural dimension with psychic considerations but fear not, it won't be long ! Besides, talking about "precognition", an ongoing quest in material science is fracture prediction, lifetime prediction... and in geophysics, earthquake prediction ... and combined with the six sense, it reminds me of this belief that animals could sense the coming of an earthquake, while human beings and their instruments commonly could not. I will just cite quickly two articles "Earthquake Prediction by Animals : Evolution and Sensory Perception" [Kirschvink 2000] and "When the snakes awake : animals and earthquake prediction" [Tributsch 1982]. Helmut Tributsch, a physical chemist collected anecdotal reports of abnormal animal behavior during 78 earthquakes [Tributsch 1982]. For example, cats picked up kittens and left houses ; dogs barked madly ; pigs bit each other ; fish jumped out of water ; and snakes awoke from hibernation, crawled from their burrows and, in some cases roze to death on snow-covered surfaces. He also describes observations of several other phenomena thought to precede earthquakes -fog, lights and sound- and discusses various hypotheses. On the basis of the available circumstantial evidence, he makes a strong argument for the attribution of these phenomena to an increase in the number of electrostatically charged particles in the atmosphere, generated by the tectonically stressed crust.

According to Chi-Yu-King in its note in Nature in 1983 [King 1983], "the book contains some simplified geophysical statements that may not be rigorous enough for the experts. Seismologists, for example, will probably be annoyed by Tributsch's frequent use of the words "force", "intensity" and "strength" interchangeably with "magnitude" in describing the size of earthquakes ; volcanologists may disagree with the statement that "volcanic eruptions are special cases of earthquake activity" ; and physicists will be dissatisfied by the absence of direct evidence for the postulated release of charged aerosols from the crust and by the absence of a credible mechanism to account for such a process".

Intrigued by those reports of animal behavior, Kirschvink talks about the evolutionary mechanism of exaptation (the process by which features acquire functions for which they were not originally adapted or selected) [Kirschvink 2000]. "The first step is to evolve a vibration-triggered early warning response which would act in the short time interval between the arrival of P and S waves. Anecdotal evidence suggests this response already exists. Then if precursory stimuli also exist, similar evolutionary processes can link an animal's perception of these stimuli to its P-wave triggered response, yielding an earthquake predictive behavior. " He also presents a brief review of possible seismic precursors and recommends that additional magnetic,

electrical, tilt, and hygro-sensors be incorporated into dense monitoring networks in seismically active regions.

After all, the animals' ability to detect impending earthquakes is interesting and amazing, but has nothing to do with ESP or that sixth sense *or my research activity*! Anyways, animals just have much higher sensitivity. The fact that animals have keener senses than humans is well-documented. Dogs have a remarkable sense of smell, birds can migrate using celestial cues, and bats can locate food with echoes. Elephants can detect faint vibrations and tremors (such as other elephants' footsteps) from fantastic distances. Animals are better sensors! At the end of the day, we all are transducers in a way as every measurement must end up as an electrical signal in our brain. And, eventually, the sixth sense would be to use wisely the five senses : listen fully, observe cautiously, contact judiciously, scent intelligently and taste soundly .. with intuition !

1.3 Focus on the sound of materials

At the end of the day, it appears that the five (or six?) senses are required to carry out a research project. Regarding my research activities, the different methods described above, through the sensory journey, are used, all along mechanical tests, to study plasticity and damage of heterogeneous materials up to global rupture, to investigate the dynamics of dislocations and of fracture precursors and explore cyclic fatigue. With the constant help of AE, developing data analysis, I listened to different materials : from polymers to metals, through scintillators.

So what do we learn from listening carefully to the materials? Applying the Five Ws (Who, What, When, Where, Why) and Silent H (How) to the experimental approach, I try to make a small contribution to the understanding of phenomena I examine and listen to. I try to find relevance in the empirical approach by collecting data, analyzing and interpreting in order to bring insight into the damage process of a material and in fine to test or draw scientific hypotheses, possibly through models and most certainly towards predicting models.

1.3.1 From plasticity to damage, up to rupture

As detection in AE requires a source mechanism to be active, in need of a confession from the material, I had to torture loads of samples to assess damage and follow the different states of degradation preceding failure. Therefore, series of experiments have been performed over a range of deformational regimes : mainly uniaxial monotonic tension and cyclic loading (strain or stress imposed, tension-compression, tension-tension). Historically, the notion of damage was introduced during the study of material fatigue (see section 1.3.2) : different loadings leading to different mechanisms.

In this manuscript, I will focus on materials like

- semi-crystalline polymers (Polyethylene (PE)) to study the initiation of their plastic deformation and damage ;
- metals ; mainly pure metals : 99.95% pure aluminum and 99.999% pure copper single crystal (elaborated using the Bridgman method [Kasap & Capper 2017], at Institut Laue Langevin (ILL), Grenoble), both face-centered Cubic (fcc) crystals, to investigate the dislocation dynamics during cyclic loading, plasticity and damage in fatigue ;
- an inorganic scintillator BGO for the study of fracture.

The microstructure of the material is first characterized in its pristine state. In most cases, initial defects due to elaboration or machining are present. Despite all the precautions taken for pure metals and above all pure single metals (electro-erosion, electropolishing...) initial dislocations are still present in the material. The mechanisms of plastic deformation, the evolution of dislocation structure, of microstructure, the increase in dislocation density, surface degradation, micro-cracks, micro-cavities... are then studied. The macroscopic crystal plasticity is explored considering both uncorrelated dislocation motions (e.g [Rouby *et al.* 1983, Chicois *et al.* 1987, Slimani *et al.* 1992, Vinogradov *et al.* 2019]) and the collective dislocation dynamics (dislocation avalanches e.g [Lebyodkin *et al.* 2013, Richeton *et al.* 2005, Richeton *et al.* 2006, Weiss & Miguel 2004, Hu *et al.* 2018]). Microstructural evolutions are correlated to dislocation patterning (under cyclic loading) on the one hand and the nature of collective dislocation dynamics on the other hand. Through the analyze of collective dislocation dynamics during cycling loading, we also try to link dislocation patterning to cyclic hardening/softening and the intermittency of plasticity.

The physical mechanisms that cause plastic deformation differ significantly with materials. In brittle materials, plasticity is caused predominantly by slip at micro-cracks. In amorphous materials, the discussion of "dislocations" is inapplicable, since the entire material lacks long range order, but crazing⁷ will take place. Among the micro-mechanisms and damage that can simultaneously occur during the plastic deformation of semi-crystalline polymers one can quote shear crystallites, fragmentation of crystalline lamellae, cavitation or martensitic transformation.

As damage progresses, cracking has emerged as an important area of my studies, as I was aiming to : investigate the initiation of micro-cracks ; consider the possible link between fatigue crack initiation, microstructure and plastic avalanches ; follow the propagation of a controlled crack and to listen to a specific signature of fatigue crack growth.

The choice of the different materials has consistently allowed to reduce the possible AE sources to just a few. In the case of pure Cu single crystals and pure Al (with a polycrystalline structure consisting of large elongated grains), the absence of precipitates or phase transformation restricts AE sources to dislocation motion

7. Small-dimension cracks which form ? in a polymeric material and the surfaces of which remain connected by filaments.

and mutual interactions of the dislocations, microcrack nucleation, and abrupt crack advances. More specifically, (see section 2.2.2 for simple AE source models for both) :

- (i) on the one hand, we relate the continuous AE to plastic deformation resulting from the cumulative effect of numerous, small and uncorrelated (in space as well as in time) dislocation rearrangements. They are activated within independent dislocation "units" (cells, channels, ...), and are characterized by a size distribution with a well-defined scale [Rouby *et al.* 1983, Slimani *et al.* 1992]. In our classification, this is a signature of the *mild* plasticity.
- (ii) and on the other hand, we relate the discrete AE to the signature of intermittent dislocation avalanches (collaborative events) that we associated with *wild* plasticity. Discrete AE is also related to cracking phenomena.

During DCDC tests on scintillators, the only possible source is crack growth.

Finally, regarding the semi-crystalline polymers, the possible AE sources are a little more numerous : the formation of micro-cavities (if any, some PE do not exhibit a formation of cavitation), the shearing of crystallites, the fragmentation of crystalline lamellae, the martensitic transformation (MT) and the fracture of fibrils close to global fracture of the material.

Nonetheless, as already mentioned, the detected AE signal is just an image of the real physical source and recent works try to build a quantitative relationship between the AE sources and the detected signal by unravelling the effect of the different stages of the acquisition chain [Hamam *et al.* 2019, Le Gall *et al.* 2018]. Although it is a crucial point, I haven't tried so far to trace back to the "primitive" AE signal. Considering the sensor and the acquisition chain are the same during one test, and having in mind that the propagation medium may change through damage etc., we still can consider that two identical events at the source will be similarly received by the sensor.

Another concern is the acoustic energy recorded by the system : what does it correspond to exactly ? How is it computed ? Can we quantify the conversion of elastic energy into acoustic energy ? And then how much is recorded by the system ? Most of these questions still remain unanswered. Every time there is an irreversible energy release in the material, only a weak part of this energy is released under the shape of a stress or displacement wave which is itself detected by the sensor. In the case of a scintillator, elastic energy is converted into broken bonds, phonons (that are either measured as acoustic emission -a weak part- or decay into heat), and light. Therefore, only a small fraction of elastic energy is converted into acoustic energy. Anyhow, physical size of the source will partially determine the amount of energy release (e.g., bundle of filaments vs. single filament, a small crack increment versus the final failure) and there must be a proportionality factor between the size of the source and the acoustic energy received.

1.3.2 Monitoring of different fatigue stages with AE

I am going to succinctly introduce fatigue at once in this section, before going into more details in the following chapters, as this theme has taken a growing part

in my research over the last few years.

Fatigue, i.e. the failure of mechanical structures under cycling loading, remains a considerable technological challenge as it occurs unexpectedly when the structure is operating apparently in a safe and steady state regime, without external signs of mechanical damage such as a measurable modification of the macroscopic mechanical behavior [Suresh 2001]. AE has thus been proposed as a monitoring tool of fatigue for a long time [Harris & Dunegan 1974]. Among other things, we illustrate that the different stages of fatigue can be differentiated in terms of AE. I will then refer, in the following chapters, to the stages that I will succinctly describe here and I will go into deeper explanations of the emerging phenomena (chapters 2 and 3.

We define five different stages, that I introduce through a cyclic strain-controlled tension-compression ($R_\varepsilon = \frac{\varepsilon_{min}}{\varepsilon_{max}} = -1$; $\Delta\varepsilon = \varepsilon_{max} - \varepsilon_{min} = 0.95\%$) test on a pure Al sample. This test and most of the fatigue tests identified in this manuscript have been performed on a taylor-made machine realized at MATEIS Lab [Chicois *et al.* 1987, Slimani *et al.* 1992], specifically designed to generate little noise compared to other hydraulic machines, as relative movements of mechanical parts are avoided during cycling, thus minimizing noise from friction. Moreover, in order to reduce the influence of external vibrations, the machine is disconnected from the ground vibrations. Finally, the sample is fixed with a specific setup allowing an acoustic confinement from the rest of the machine.

By way of illustration, figure 1.20 include the cyclic stress response (CSR) curve (maximum stress σ vs. number of cycles N), the average number of detected AE bursts per cycle (burst rate, dn/dN), the cumulative energy of bursts and the acoustic power peak value, recorded at plastic yield of each cycle.

An initial, rapid hardening stage (A), lasting $\sim 10 - 20$ cycles for $\Delta\varepsilon = 0.95\%$, is singled out by the development of dislocation entanglements leading to an irregular cell structure [Videm & Ryum 1996]. At this stage, the only detectable structures at the surface are slip lines on few grains, in a single-slip configuration (figure 1.11 a) [Weiss *et al.* 2018]. The AE power peak value, recorded at plastic yield, and the burst rate strongly decrease with the increasing number of cycles, when cyclic strain hardening is important.

A softening stage (B), lasting ~ 100 cycles, shows a considerable reorganization of the initial random dislocation pattern into a regular structure and straight dislocation walls [Videm & Ryum 1996]. Although, the burst rate exhibit very low values (generally less than one detected burst per cycle), some episodic discrete AE bursts do persist at those scales, exhibiting a clear evolution through cyclic loading. Transient softening can be explained by this microstructural rearrangement, which is also characterized by the onset of the activation of additional slip systems, as double-slip configurations were observed within few grains (figure 1.11 b).

Afterwards, a secondary hardening stage (C), which can last thousands of cycles and does not show any stress saturation, is associated with the development of a well-defined, regular wall and ladder structure [(figure 1.11 c). The burst rate reaches another minimum before starting to increase slowly, as does the peak acoustic power.

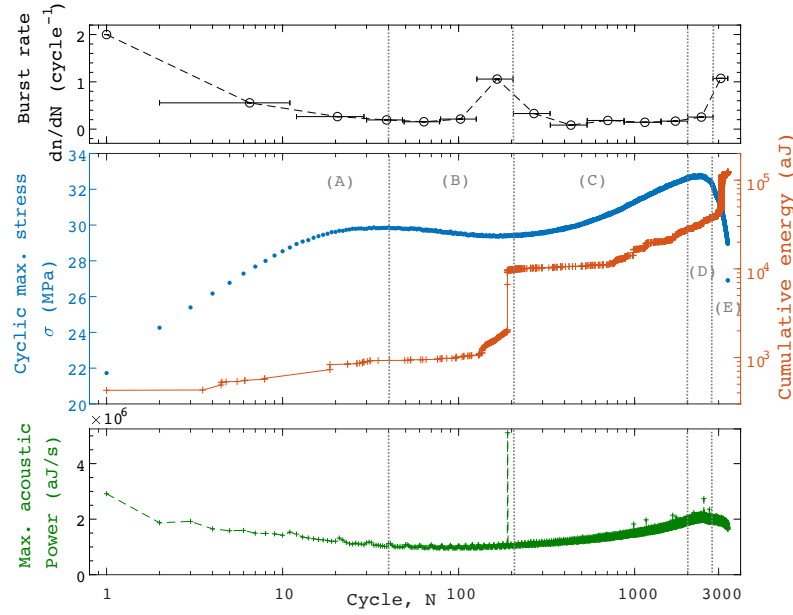


FIGURE 1.20 – CSR curve during a fatigue test at $\Delta\epsilon = 0.95\%$ on pure Al : the different stages of fatigue are clearly differentiated in terms of AE (AE burst rate, cumulative energy, AE power peak value per cycle).

At this stage the first signs of slip irreversibility and crack initiation along PSBs appear at the surface, see (figure 1.11 (c)).

Finally, as the cyclic deformation continues, crack propagation becomes visible perpendicular to the loading axis, indicating stage II of fatigue cracking (figure 1.11 d). The peak acoustic power reaches a new maximum in a fourth stage, onset of secondary softening stage associated to extensive damage (D), while the burst rate rises again. The final softening stage (E) corresponds the final failure of the material with higher energy bursts.

1.4 Be kind, rewind !

This document is organized in the reverse chronological order of the studies I was able to undertake during these past 12 years so that it follows the life-journey of the material under stress until global rupture : starting with the initial damage of materials and the initial stages of cyclic deformation, going through dislocation patterning up to micro-crack initiation, to finally end with extensive damage and final failure.

That is to say, Chapter 2 is devoted to plasticity in different materials : about the onset of plasticity in semi-crystalline polymers ; the coexistence of two kinds of plasticity and about dislocation dynamics during cyclic loading in metals. Chapter 3 focuses on damage from different perspectives : during fatigue of metals, from dislocation patterning, to crack initiation until crack growth and fracture ; and cracks

emitting light in crystals. Accordingly, in the case of metallic materials, by plasticity, I imply phenomena related to dislocations movements and by damage, phenomena implying microcracking and cracking.

In the end, chapter 4 is dedicated to the projects I would like to tackle in the coming years.

Plasticity

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2.1 Initiation of plastic deformation of semi-crystalline polymers during tensile tests

Semi-crystalline polymers constitute an increasingly large part of the polymers used as structural materials (having to withstand mechanical stresses). This is a natural trend that will be reinforced in the coming years as they are essentially recyclable or even biosourced, unlike thermosetting materials. Consequently, the plastic deformation and damage of semi-crystalline polymers have been widely studied during the last decades, allowing to describe the fundamental mechanisms (e.g. shear crystallites, fragmentation of crystalline lamellae, cavitation or phase transformation) leading to the fibrillar microstructure [Galeski 2003, G'Sell & Jonas 1981, Pawlak *et al.* 2014, Lin & Argon 1994, Humbert *et al.* 2010b, Humbert *et al.* 2010a, Xiong *et al.* 2013, Xiong *et al.* 2015]. However, the in situ determination of the initiation of micro-structural plastic events remains problematic and requires complex devices such as Small-angle X-ray scattering (SAXS) or tomography [Humbert *et al.* 2010b, Humbert *et al.* 2010a, Pawlak *et al.* 2014, Xiong *et al.* 2013, Xiong *et al.* 2015]. These materials have a very complex and hierarchical microstructure ranging from the Angström to the micron (coexistence of amorphous and crystalline phases, see figure 2.1). And the study at the mesoscopic scale (from 100nm to

1 μ m) still needs to be expanded as it is at this scale that important deformation mechanisms occur, leading to the collapse of the crystal lattice characteristic of the plasticity of semi-crystalline polymers.

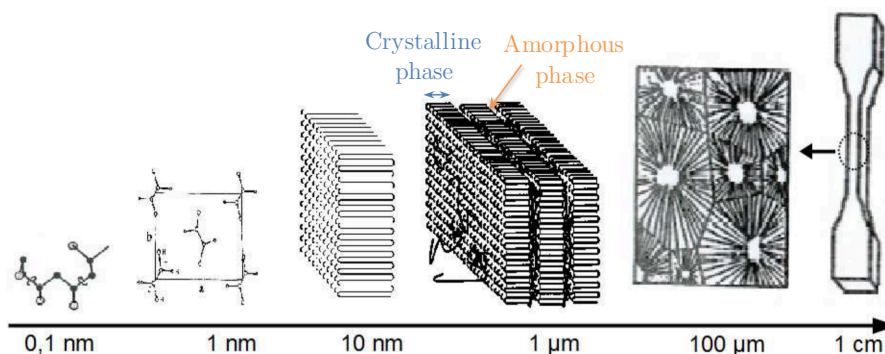


FIGURE 2.1 – Different observation scales of a semi-crystalline polymer.

Therefore, we propose to use the **AE** and **US** techniques to obtain new information about the initiation of plastic deformation and damage of semi-crystalline polymers above their glass transition temperature, during tensile tests. More specifically, we wish to gain a better understanding of the early stages of the mechanisms involved at the local scale in the progressive transformation of the spherulitic structure into fibrillar structure during plastic flow. The influence of the different mechanisms is established, on the one hand, on the AE activity generated [Casiez *et al.* 2014], and on the other hand, on the propagation of **US** waves [Casiez *et al.* 2017]. For the study, several Polyethylenes (**PE**) have been elaborated through different thermal treatments (quenching and isothermal treatment) : **PEs** with varying degrees of crystallinity (ranging from 48% to 79%) -the sizes of crystalline lamellae varying as well-, and different mechanisms of cavitation formation appearing during tensile tests.

The results presented here are largely based on the work carried out as part of Nicolas Casiez' PhD thesis [Casiez 2015]. This PhD thesis was included in the Young Researchers Project ANR (JCJC) "Listening to understand and characterize the plasticity and damage of semi-crystalline polymers" (POLYCOUSTIC), obtained in 2012 with Olivier Lame (Mateis) and Thomas Monnier (Mateis/LVA).

2.1.1 Via the Acoustic Emission technique

The detection of acoustic signals originating from the deformation of **PE** is challenging since polymers strongly attenuate acoustic waves. This could explain the modest number of papers in the literature dealing with AE in such materials! Given this high attenuation in semi-crystalline polymers, we have assumed that the detected **AE** signals cannot come from a single isolated source but from an avalanche of sources on a local scale, allowing the partial addition of the generated waves' amplitude, thanks to the interference phenomenon.

A weak acoustic activity is recorded during the tensile tests on PE. Moreover, these signals are characterized by low acoustic energy. Nevertheless, several AE sources are possible : the formation of micro-cavities, the shearing and fragmentation of crystalline lamellae, the martensitic transformation (MT) and the fracture of fibrils, plus parasitic sources such as machine noise or slippage of the experimental set-up. Hence, it is difficult to conclude about the origin of recorded AE signals, in particular as their localization is not always possible.

However, the use of two different specimen geometries (a dumb-bell and an "anti-sample") and two methodologies based on the elimination and the discrimination of spurious sources has shown that the majority of detected AE signals truly originate from the plastic deformation of materials and/or damage of the material.

This verification allowed us to show that an increase in strain rate leads to greater acoustic activity (number and energy of bursts increase). This behavior is caused by a greater occurrence of plasticity and damage mechanisms leading to a higher amount of acoustic energy being released. In addition, the localization of few signals is possible, along the specimen length, when the strain rate is high, which ascertains that the signals do mostly originate from mechanisms occurring within the material. However, a direct correlation between the AE bursts and the mechanisms is very difficult owing to the weak AE activity. Yet, it would seem that the fragmentation of the crystalline lamellae, cavitation or MT are all responsible for the release of acoustic energy. Besides, since the signals originate from an avalanche of sources, it is very likely that the waves, generated by the different plasticity and damage mechanisms, each contribute to the resulting signal, by partially adding their amplitude, since they appear simultaneously within the material.

The acoustic activity presents three phases during tensile tests, which allowed us to propose a simple model based on the distribution of AE sources on the specimens (figure 2.2).

When they appear, the micro-mechanisms are highly concentrated in the centre of the specimen, which favors the partial addition of wave amplitude by interference. Consequently, the detection of the bursts is easier and the majority of AE signals is recorded, at small strains. In particular, the detection of signals before yielding shows that the initiation of plasticity and/or damage is initiated in the earlier stage of the deformation.

The second phase is characterized by a very weak acoustic activity at medium strains, where the formation of a necking appears and the release of the acoustic energy becomes very diffuse in the samples. Hence, the detection of AE signals is unlikely due to the significant reduction in the phenomenon of interference together with an increase in the spatial extent over which AE sources appear.

Finally, the number of recorded AE signals increases again at high strains, when the necking regions approach the sensors. However, it is difficult to know if these signals, whose energy is relatively high compared to others, come from high energy

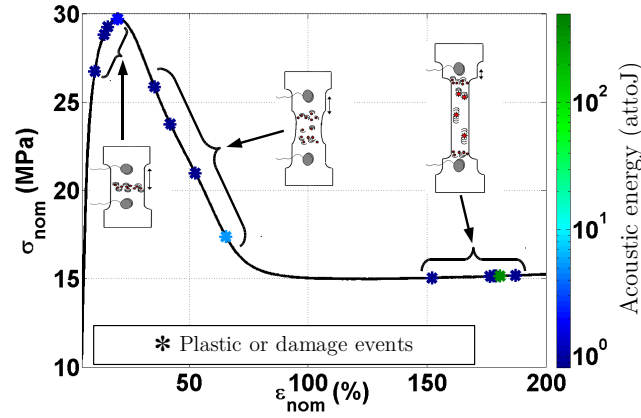


FIGURE 2.2 – Three phases of acoustic activity during a tensile test on PE, with humble AE source allocation model.

damage mechanisms such as fracture of fibrils taking place far from the sensors, in the fibrillar region, or from mechanisms close to sensors and whose energy is weakly attenuated by the short propagation in the material.

The influence of the degree of crystallinity and the mechanisms of formation of micro-cavities has also been studied. The cumulative number of AE signals increases with the crystallinity. This result can be attributed to lower US attenuation, and higher acoustic energy released as the crystallinity of the PE increases.

Only a tiny fraction of the stored elastic energy is released in the form of acoustic energy. The latter increases significantly with the degree of crystallinity, in particular for PE samples exhibiting a formation of micro-cavities. It can actually be suggested that the formation of cavitation is associated with the release of a relative high energy.

Initiation of acoustic activity Acoustic activity is initiated for all PEs before the yield point. Consequently, micro-mechanisms of plasticity and/or damage appear before yielding which is generally considered to be the time when the plastic deformation of the crystalline lamellae is initiated from a macroscopic point of view. Based on the AE data, we have therefore quantitatively defined a plasticity criterion showing that the onset of plastic deformation and damage occurs before the yield point. Two criteria are defined from the stress at which the AE bursts are recorded before the yield point.

- The first criterion, $IniP$, is the ratio between the mean macroscopic nominal stress, at which the AE signals are recorded before the yield point, and the mean of the mechanical nominal stresses from stress/strain curves :

$$IniP = \frac{\bar{\sigma}_{\text{before yield}}}{\bar{\sigma}_{max}}.$$

- The second criterion, $IniP'$, is the ratio of the mean of the nominal macroscopic stresses at which the first AE signal is recorded before the yield point

for all the tests, and $\bar{\sigma}_{max}$:

$$IniP' = \frac{\bar{\sigma}_{first\ signal\ before\ yield}}{\bar{\sigma}_{max}}.$$

Figure 2.3 represents the evolution of those two criteria with the degree of crystallinity.

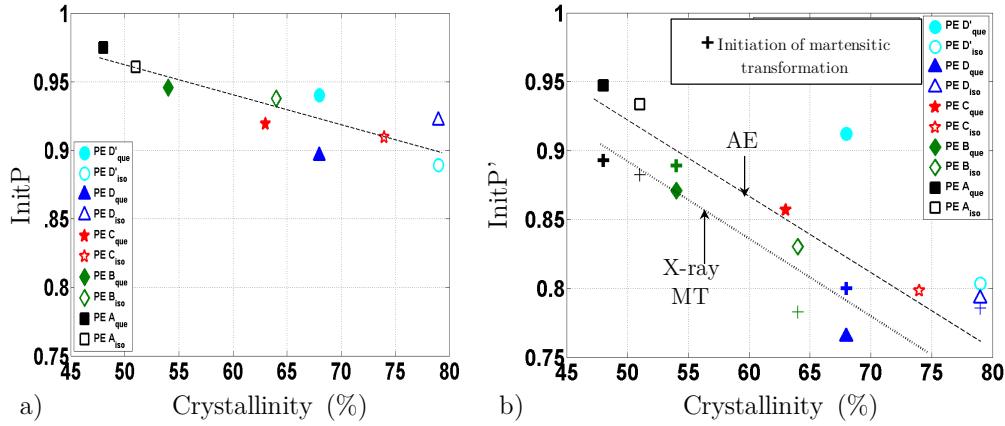


FIGURE 2.3 – Evolution of plasticity criteria with the degree of crystallinity of several PEs (the notations PEA_{que} , PEB_{iso} ... refer to the different thermal treatments [Casiez *et al.* 2014]). In b) : comparison of the initiation of acoustic activity with MT (+) determined from SAXS [Humbert *et al.* 2010b]. Black dashed-lines indicate trends.

The initiation of acoustic activity is all the earlier as the degree of crystallinity is high and can be correlated to the formation of micro-cavities before yielding for the highest crystallinities PE samples. We also found that the acoustic activity recorded before the yield point presents a behavior close to the initiation of MT [Humbert *et al.* 2010b] for the studied PEs (figure 2.3 b), suggesting that this mechanism releases acoustic energy before yielding.

2.1.2 Ultrasonic in situ investigation

We proposed to use complementary US monitoring techniques, still in order to characterize the plasticity and damage of several Polyethylene PE during tensile tests. The proposed methodology uses both guided and longitudinal waves in the ultrasonic frequency range and enables to separate the geometrical effects from those of the material.

US waves propagation in undeformed materials

The propagation of guided and longitudinal waves in undeformed PE exhibits an exponential decay with the propagation distance, and a huge attenuation varying from about 800 to 400 dB/m is observed when the crystallinity increases from $\chi_c = 50\%$ to 80%. When crystallinity increases, the US waves "see" a medium containing a higher proportion of crystalline phase which attenuates less the US

waves than the amorphous phase (US attenuation of the crystal is about 10 times lower than the US attenuation of the amorphous phase). This confirms the impact of the microstructure of PE. In the frequency domain used here, the scattering is negligible and the attenuation is essentially attributed to thermal dissipation. Similarly, we found that the wave velocity increases with the degree of crystallinity. As the material modulus increases with the crystallinity and the wave velocity is related to elastic modulus of material, the observed results were then qualitatively expected. We also showed the strong impact of the sample geometry on the US attenuation since for guided waves, decreasing the thickness increases strongly the attenuation.

Influence of plasticity and damage mechanisms on US wave propagation

In-situ tests were performed during a tensile test, using protocols specifically developed for guided and longitudinal waves. In the elastic regime, as expected,

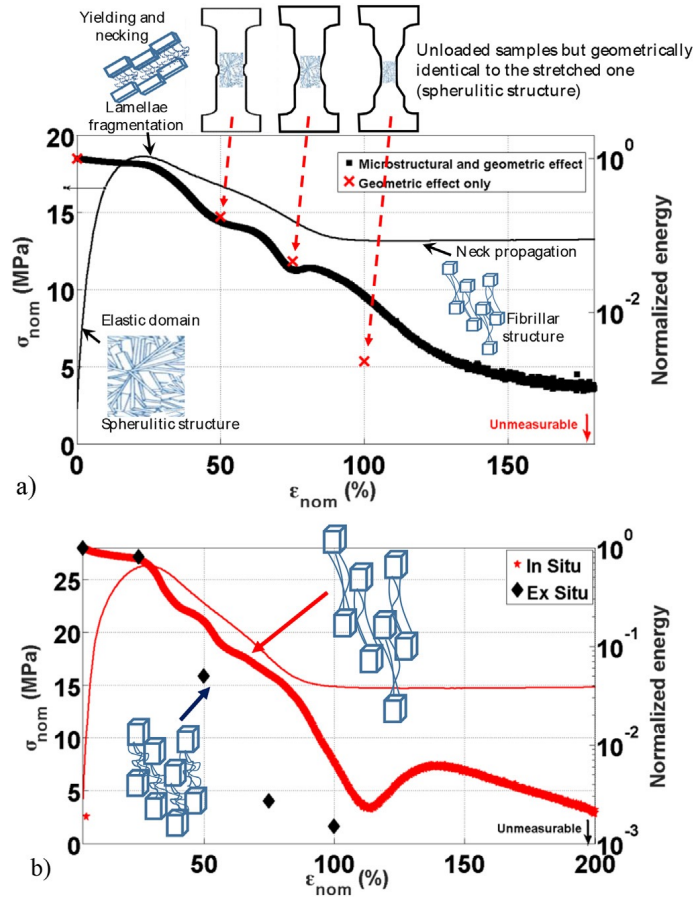


FIGURE 2.4 – a) Separating the geometrical effect from the microstructural effect and the chain extension : attenuation measured for unloaded samples -geometrically identical to the stretched samples- (red cross) vs. attenuation measured in-situ. b) Effect of the chain extension : in situ vs. ex-situ measurements (after unloading, black diamonds).

US parameters remain essentially constant. As soon as the plasticity is initiated, the attenuation coefficient of US waves strongly increases (strong decrease of the transmitted energy), which can be explained by (i) the evolution of the geometry due to necking and (ii) the onset of mechanisms of plasticity and damage within the material. We have shown that the geometrical evolution alone of the specimen under the effect of the the formation of necking causes a significant increase in the attenuation of the guided waves (figure 2.4 a). The destruction of the crystal network, via the fragmentation of crystalline lamellae and cavitation, generates a strong US attenuation increase due to a massive collapse of elastic properties. More surprisingly, we also showed that the progressive alignment of the chains favors the propagation of US guided waves (figure 2.4 b) and can partially compensate the effects of both crystalline network collapse and geometry.

In addition, by comparing guided and longitudinal waves, we showed that the cavitation likely plays a significant role only for longitudinal waves (see figure 2.5). For the longitudinal waves, the attenuation strongly increases with the degree of crystallinity and a strong cavitation is observed for PE samples with the highest crystallinity. The opposite evolution can be observed for the guided waves : the high-crystallinity PE group exhibits a lower attenuation. For longitudinal waves, the cavities are coupled in series with the polymer fibrils whereas they are in parallel for guided waves as illustrated in figure 2.5.

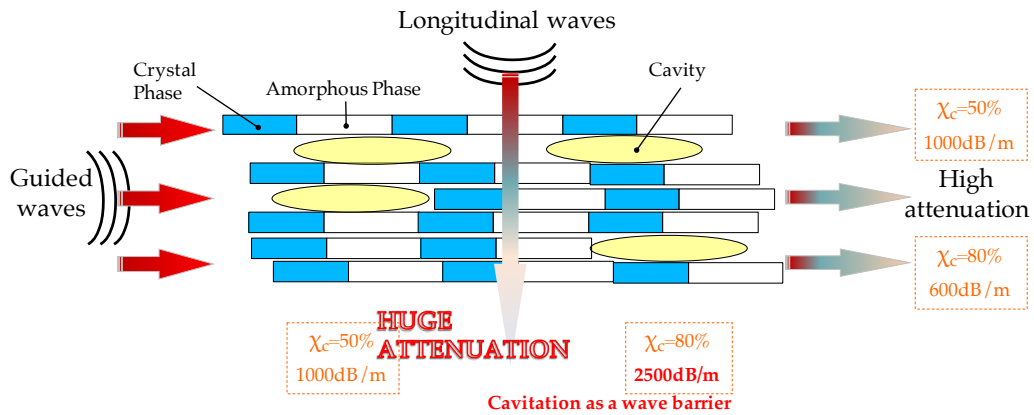


FIGURE 2.5 – Perception of the wave attenuation in a semi-crystalline polymer : anisotropy and cavitation (schematic of the structure with fibrils and voids in plastically deformed PE)

As a consequence, guided US waves propagate essentially using the percolating paths formed by the fibrils, which are weakly disturbed by the presence of the cavities. Finally, it can be noted that the decrease of attenuation originates from the increase in crystallinity since the crystals attenuate much less the US waves than the amorphous phase. It is noticeable that comparable results have been obtained concerning the thermal conductivity. Indeed as the crystalline phase is intrinsically anisotropic, a PE stretched until fibrillar structure, where the crystalline blocks are

essentially aligned, exhibit conductivity that can be 20 times higher in the tensile direction.

Finally, these results show that the [US](#) monitoring technique is an effective method to characterize the microstructural evolution of semi-crystalline polymers along a tensile test, especially revealing the strong anisotropic effect of the cavities on [US](#) propagation. And consequently, [US](#) and [AE](#) techniques may also characterize the degree of fibrillation of a polymer.

2.2 Dislocation-mediated plastic flow

All in all, since the crystalline phase is much less attenuating, I will concentrate on listening to crystalline materials! More specifically, I will now focus on the dissipative process occurring in deforming metals during dislocation-mediated plastic deformation, which has already been widely studied through [AE](#).

2.2.1 Coexistence of two kinds of plasticity

In classical plasticity theory, dislocation-mediated plastic flow is generally assumed to be smooth, with small and uncorrelated strain fluctuations averaged out and thus undetectable at large ($\geq$ mm) scales : an outcome of uncorrelated dislocation motions producing Gaussian fluctuations (*mild* plasticity). An apparently conflicting picture emerged in recent years emphasizing highly correlated dislocation dynamics characterized by power-law distributed fluctuations (*wild* avalanches). And actually, an intermittent character of plasticity in metals, with large fluctuations of the strain rates associated with dislocation avalanches, had been observed a long time ago, especially at the early stages of deformation in the microplastic regime of hexagonal close packed ([hcp](#)) metals [[Becker & Orowan 1932](#), [Tinder & Trzil 1973](#), [Maaß & Derlet 2018](#)].

Based on former [AE](#) measurements in crystals with different symmetries supervised by Jérôme Weiss and our joint work on Low Cycle Fatigue ([LCF](#)) of pure aluminum (during the ANR project EVOCRIT, "Evolution of criticality in cyclic loading as an indicator of fatigue", 2009-2012¹), we showed that intermittent and continuous visions of plastic flow are not incompatible [[Weiss et al. 2015](#)].

In pure metals, the relative contribution of the two types of plasticity depends on the presence of a dislocation structure, a dense structure being assumed to limit the extent and role of dislocation avalanches. The formation of a dislocation structure is in turn related to the crystallographic nature of the material and the loading mode.

Past experiments performed under monotonic loading on both single- and polycrystals of hexagonal metallic materials and ice ([[Miguel et al. 2001](#), [Weiss & Grasso 1997](#), [Weiss & Marsan 2003](#), [Weiss et al. 2007](#), [Richeton et al. 2006](#)]) showed that the plastic deformation processes are not smooth and homogenous in

1. Including Wafa El May's PhD thesis [2010-2013] [[El May 2013](#)]

space [Weiss & Marsan 2003] and time [Weiss & Miguel 2004]. Plasticity is then governed by dislocation avalanches moving through the material in a very heterogeneous and intermittent fashion, generating discrete AE : almost 100% of the plastic deformation takes place in the form of avalanches. This has been confirmed through numerical modeling [Miguel *et al.* 2001, Zaiser 2006]. The formation of a dense and stable dislocation structure is not observed in these crystals. The small number of easy slip planes favors long-range elastic interactions allowing dislocations to self-organize, hence collective effects prevailing the intermittency (signature of a wild plasticity).

On the contrary, the multi-slip character of plastic deformation in fcc materials favors short-range interactions between dislocations such as the formation of locks and junctions that give rise to stable, low energy configurations [Madec *et al.* 2002]. Hence, the build-up of a dislocation microstructure (walls, cells...), which reduces the dislocation mean-free path [Devincre *et al.* 2008], inhibits the collective dislocation dynamics and avalanches, leading to a mild plasticity (continuous AE).

However, even in this mild regime, where most of plastic deformation occurs through small uncorrelated dislocation motions, rare but potentially very large (relative to the characteristic wavelength of the dislocation microstructural pattern) cascades of unlocking events [Devincre *et al.* 2008] can take place, leading to brutal rearrangements of the dislocation substructures. This argues for the existence of crossover regimes where mild (small and uncorrelated) and wild (correlated, power-law distributed) fluctuations can coexist.

To characterize such a crossover, it is convenient to define a parameter measuring the degree of wildness W . A natural choice for such a measure is the fraction of plastic deformation accommodated through dislocation avalanches. In the case of deformation of micropillars, W can be reliably estimated from an analysis of strain and/or stress increments/decrements on the loading curve [Zhang *et al.* 2017]. In the set of experiments presented in this manuscript, focused on the behavior of bulk samples, even the largest avalanches were undetectable from the macroscopic loading curves given the limited sensitivity and the recording frequency of our stress and strain measurements. We therefore define an AE proxy of wildness denoted by W_{AE} , as the fraction of AE energy released through detected AE bursts (discrete AE, hence avalanches), once the instrumental noise has been removed :

$$W_{AE} = \frac{\sum(\text{Discrete AE Energy (aJ)})}{\sum(\text{Total AE Energy (aJ)})} \quad (2.1)$$

As shown in figure 2.6, the measured AE signals and W_{AE} consistently substantiate these differences over a range of deformational regimes (compression creep, uniaxial monotonic tension, tension-compression cyclic loading). Hexagonal crystals, with highly anisotropic slip (ice, Cd, Zn) represent a paradigmatic example of intermittent plasticity with W_{AE} always larger than 95%, almost 100% in ice : AE has a form of an intermittent signal with a negligible continuous background, during compression creep of ice (figure 2.6(a)) and during uniaxial monotonic tension oh hcp cadmium (figure 2.6(b)). Plastic deformation is almost entirely controlled by

dislocation avalanches. Hexagonal crystals exhibit correlated scale-free flows : the bursts are power-law distributed in dissipated energy (figure 2.7), $P(E) \sim E^{-\tau_E}$, with the exponent $\tau_E = 1.40 \pm 0.03$, determined from a robust maximum likelihood methodology [Clauset *et al.* 2009].

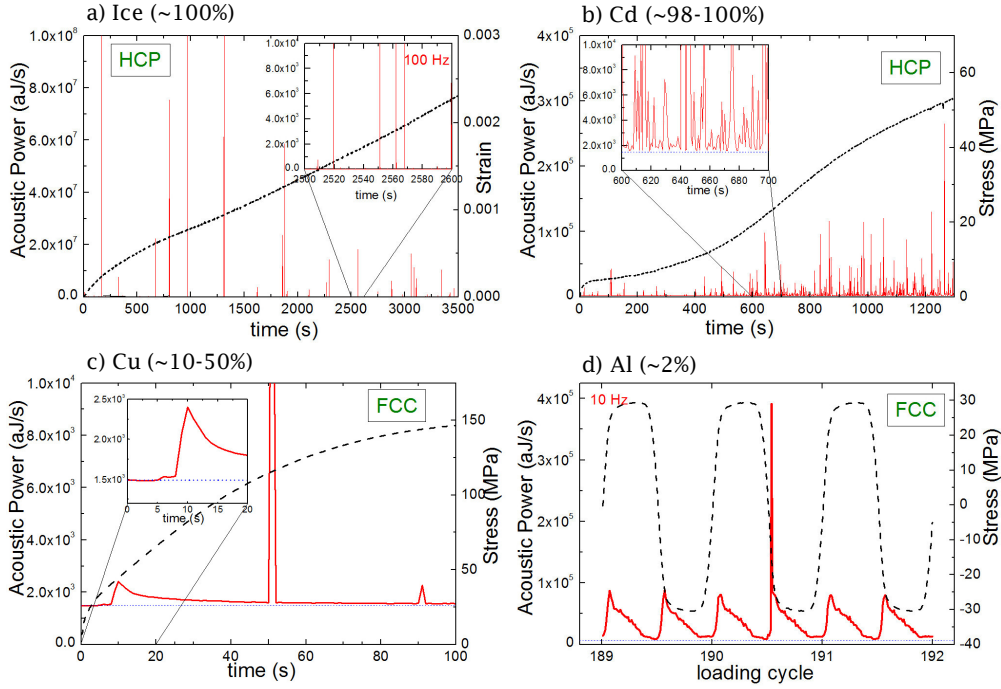


FIGURE 2.6 – Acoustic power recorded during plastic deformation and W_{AE} (%). (a) Single crystal of ice (*H*) under uniaxial compression (creep at constant stress $\sigma = 0.56 \text{ MPa}$, $T = -10^\circ\text{C}$). (b) Cadmium single crystal (*hcp*) under monotonic uniaxial tension ($\dot{\epsilon} = 1.3 \times 10^{-3} \text{ s}^{-1}$, $T = 20^\circ\text{C}$). (c) Copper single crystal (*fcc*) under monotonic uniaxial tension ($\dot{\epsilon} = 1.9 \times 10^{-2} \text{ s}^{-1}$, $T = 20^\circ\text{C}$). (d) Aluminum polycrystal (*fcc*, grain size $\sim 5 \text{ mm}$) under cyclic uniaxial strain control ($\epsilon_{\min}/\epsilon_{\max} = -1$, $\Delta t_{\text{cycle}} = 10 \text{ s}$, $\Delta\epsilon = 0.95\%$, $T = 20^\circ\text{C}$). Red curves : acoustic power sampled at 1 Hz , unless noted otherwise. Black dashed curves : strain (a) or stress (b), (c), (d). Blue dotted lines indicate the level of instrumental noise.

Instead, in the studied *fcc* materials (Cu, Al, CuAl alloys), intermittent and continuous plastic flows coexist. It has been long noticed that AE in plastically deformed crystals may include both continuous background and discrete bursts [Rouby *et al.* 1983, Kiesewetter & Schiller 1976]. Figure 2.6(c) (Cu crystals under uniaxial monotonic tension) shows that acoustic signal is mainly continuous, reaching its maximum at plastic yield, with only occasional bursts above this background. During cyclic loading of Al crystals (Figure 2.6(d)), the acoustic signal is essentially continuous and symmetric in tension and compression, hence revealing its plastic

origin. The continuous noise, however, is interrupted by bursts, on average less than 1 per loading cycle. The continuous component denotes the prevalence of small, uncorrelated dislocation motions confined inside substructural units (cells, labyrinths, etc.) that effectively screen long-range interactions. The large bursts can be attributed to the major autocatalytic cascades of unlocking events. This suggests that the commonly observed quasiequilibrium dislocation patterns in fcc crystals are only marginally stable and their restructuring can be triggered by insignificant changes in the global force balance.

For aluminum (figure 2.6(d)), the contribution due to avalanches is small, reaching under cyclic loading at most a few percent during the first cycles, when the dislocation substructure has not yet fully developed.

The intermediate behavior of Cu (W_{AE} between 10 % and 50 % for Cu single crystals under tension) might be explained by a smaller stacking fault energy compared to Al, favoring the dissociation of dislocations and kinematic hardening. The intermediate position of Copper and CuAl alloys can also be seen through the AE energy probability density function (PDF) : the energy is also power-law distributed but with different scaling exponents (for Cd $\tau_E = 1.45 \pm 0.05$, for Al $\tau_E = 2.00 \pm 0.05$ and for Cu and CuAl alloys $\tau_E = 1.54 \pm 0.08$). Figure 2.7 compares the AE energy PDF in the case of ice, Al and Cu. To relate to the literature on the randomness classification of the scaling exponents [Eliazar & Cohen 2013], we compared the amplitude scaling exponents τ_A ($P(A_0) \sim A_0^{-\tau_A}$). The maximum amplitude of an AE burst, A_0 , is a measure of the incremental strain associated with the dislocation avalanche and scales as $A_0 \sim E^{1/2}$ [Weiss *et al.* 2015, Weiss & Grasso 1997, Rouby *et al.* 1983, Richeton *et al.* 2005, Weiss *et al.* 2000, Weiss *et al.* 2007]. It means that $\tau_A = 2\tau_E - 1$, i.e., $\tau_A = 1.8$ for ice and $\tau_A = 3.0$ for Al. Thus, plastic fluctuations in ice can be qualified as wild with an undefined mean ; for aluminum, with the variance diverging, we are just at the border between wild and mild fluctuations.

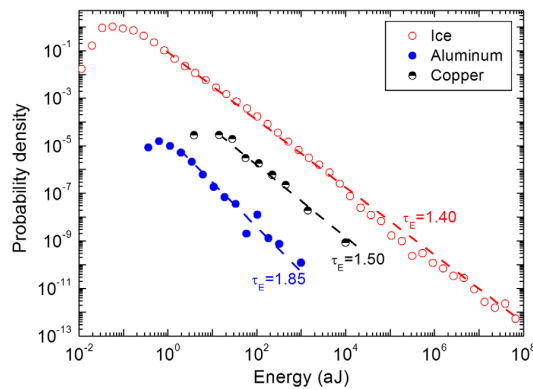


FIGURE 2.7 – AE energy PDF for bursts detected during : a uniaxial compression test on ice (see figure 2.6(a)), a monotonic tension test on copper (see figure 2.6(b)), and a cycling loading test on aluminum under uniaxial tension compression (figure 2.6(d)). The PDFs have been shifted vertically for clarity.

Moreover, the case of cyclic loading of aluminum, for which mild plasticity prevails with few appearance of dislocation avalanches, compared to monotonic loading, indicates that the relevance of the intermittent dynamics in plasticity could also depend on the loading mode. Indeed, cyclic loading is well known to progressively generate well-defined dislocation structures that could embed the avalanches motions and thus decrease the fraction of intermittent plasticity under cycling. I will study the influence of cycling in the following but first, let me present two simple AE source models for intermittent dislocation avalanches on the hand and uncorrelated plastic flow on the other hand.

2.2.2 Acoustic emission source models

Simple AE source models² are used to relate AE characteristics (as energy E_{AE} of bursts or acoustic power dE/dt presented in section 1.2.1.4) to the properties of the source(s), i.e. to the motion of dislocations in the present case.

2.2.2.1 AE source model for discrete AE

I will express here the source model used to interpret the discrete AE (bursts), based on the analysis of Rouby et al. [Rouby *et al.* 1983] and that has been presented in Weiss and Richeton publications [Richeton *et al.* 2005, Richeton *et al.* 2006, Weiss *et al.* 2000, Weiss *et al.* 2007].

The basic assumption is that an AE burst results from the transient and synchronized motion of n dislocations constituting a dislocation "avalanche". *Synchronized* means that the average time delay τ_a between the triggering of individual dislocation motions *within the avalanche* (e.g. between dislocations $i - 1$ and i) is small compared to the characteristic period of the AE transducer, $1/\omega$, where $1/\omega$ is the frequency.

The simple AE source model states that the maximum amplitude of the acoustic wave A_0 , which roughly corresponds to the amplitude at the initiation of the plastic event, is a proxy of the strain associated with the avalanche. It indeed relates directly A_0 to the surface S swept out by dislocations during the avalanche, and to the corresponding strain increment ε_{pl} :

$$A_0 \sim \alpha S b \sim \alpha \varepsilon_{pl} \quad (2.2)$$

with b the Burgers vector and α is damping coefficient.

2.2.2.2 AE source model for continuous AE

To explain the evolution of AE power during cyclic loading of fcc metallic materials, we use a simple model [Rouby *et al.* 1983, Chicois *et al.* 1987, Slimani *et al.* 1992] of uncorrelated plastic flow (mild plasticity). The fundamental hypothesis behind the interpretation of continuous AE is that plastic deformation results from the

2. The models are explained in detail in the supplementary material of [Weiss *et al.* 2007].

cumulative effect of numerous uncorrelated (in space as well as in time) small motions. These independent AE sources are assumed to overlap, and the continuous component of the AE power dE_c/dt (AE power minus the discrete AE) scales as :

$$\frac{dE_c}{dt} \sim b\bar{S}\frac{d\varepsilon_{pl}}{dt} \quad (2.3)$$

where $\bar{S}$ is an average sweeping area for these small dislocation motions, i.e. $\sqrt{\bar{S}}$, consistent with a length, can be associated with the dislocation mean free path³, i.e. $\sqrt{\bar{S}} \approx l$.

Such correspondence between the continuous AE power dE_c/dt and the plastic strain-rate $d\varepsilon_{pl}/dt$ is checked in the case of cycling loading of pure Al and pure Cu, either for strain- or stress-control tests (see figure 2.8 for an example) and has been previously validated for monotonic loading of Al [Kiesewetter & Schiller 1976]. $\bar{S}$ will thus be extracted from equation 2.3 for each cycle of a fatigue test, giving information on the evolution of the dislocation sub-structure from one cycle to another.

Otherwise, the absence of detectable intermittency in the raw AE signal during the cyclic loading of aluminum [Weiss *et al.* 2015], was verified from a multifractal analysis of the AE power record [Lebyodkin *et al.* 2009].

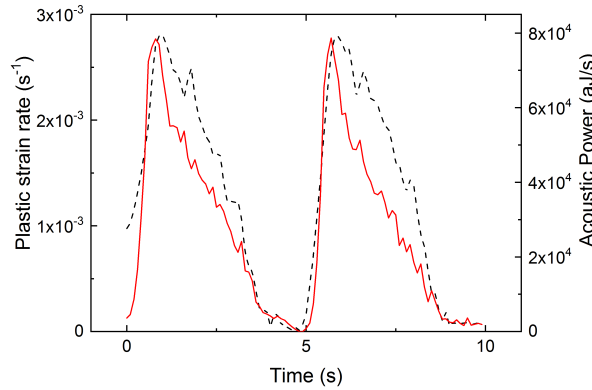


FIGURE 2.8 – Comparison between the AE power recorded during a loading cycle of a cyclic test on a pure Al sample ($R_\varepsilon = -1$; $\Delta\varepsilon = 0.95\%$), and the macroscopic plastic strain-rate measured by extensometry (dashed black line). The instrumental/environmental background noise has been removed from the recorded AE power.

In addition to these two simple models, the article [Weiss *et al.* 2015] proposes a simple theoretical framework compatible with regimes where strongly intermittent events coexist with a Gaussian quasiequilibrium background. The simple stochastic mean-field model, where dislocation flow is represented by a Gibrat-type proportional dynamics [Kesten 1973], is able to capture statistics of avalanches observed in the experiments presented above, which is Gaussian for small events and power law for large events.

3. The dislocation mean free path represents the distance over which a dislocation can travel before being stopped by dense dislocation arrangement (cells, walls, etc.)

2.3 Intermittent plasticity under cyclic loading

The relevance of the intermittent dynamics in plasticity (section 2.2.1) could then depend at the same time on (i) the crystal symmetry (favoring or not the formation of complex dislocation structure) and (ii) the loading mode (monotonic versus cyclic, reinforcing or not the dislocation structure). The evolution of dislocation patterning during fatigue has been extensively studied, and is now well characterized, at least for pure metals [Suresh 2001]. In metals, steels and alloys for which the plastic yield stress is reached at each cycle, strain hardening as well as fatigue crack initiation have been linked with the evolution of dislocation patterns and formation of cell structures during cyclic loading [Laird *et al.* 1986, Mughrabi 2009]. In particular, the development of low-energy dislocation configurations such as PSBs is associated with an irreversibility of slip along different glide planes and plastic strain localization, which serve as precursors to crack initiation [Sangid 2013]. Most of these observations, however, were performed post-mortem (e.g. from TEM), hence capturing only static pictures of dislocation patterns after a given number of loading cycles, while the dynamics of the problem has been somewhat overlooked.

Here, I will question the degree of intermittency of plastic deformation and collective dislocation dynamics when driven by cyclic loading. Namely, the wildness W will be studied through the AE proxy W_{AE} , which is a useful dimensionless measure of the plastic intermittency in bulk samples, especially when one can track its evolution through successive cycles.

A recent study [Zhang *et al.* 2017] has shown that the wild-to-mild transition is controlled, to the first order, by a ratio of length scales, $R = L/l$, with L the external system size and l an internal length scale. The fact that W decreases with R explains how fcc and body-centered cubic (bcc) pure metals characterized by smooth plastic flow at bulk scales may still exhibit power-law distributed strain bursts at μm to sub- μm scales. The internal length scale l can be extrinsically set through alloying, allowing one to counteract the effect of the external size and thus tame intermittency of plastic deformation at small scales.

In pure metals, however, one expects l to intrinsically emerge from short-range dislocation interactions and serve as a characteristic of a dislocation microstructure. In this context, l can be identified as a typical scale of the dislocation pattern which, according to the "similitude principle" [Sauzay & Kubin 2011, Zaiser & Sandfeld 2014], should be inversely proportional to the shear stress τ , $l \sim Gb/\tau$, where G is the shear modulus. Combining this with the Taylor's relation describing strain hardening, $\tau \sim Gb\sqrt{\rho}$, we obtain $l \sim 1/\sqrt{\rho}$ [Zaiser & Sandfeld 2014, Staker & Holt 1972], which defines the boundary between short- and long-ranged (elastic) interactions between dislocations. From these considerations, one can expect the wildness of plastic deformation : (i) to increase with decreasing initial dislocation density ρ_i ; and (ii) to decrease as the material strain-hardens and the forest density

ρ increases.

With all this in mind, some questions can be raised :

- Do dislocation avalanches exist during the whole development of fatigue in fcc metals or plastic intermittency vanishes after only few loading cycles as a result of the build-up of stable, low-energy dislocation substructures ?
- Do the anticipated relations between W , strain-hardening, and the dislocation patterning scale l hold under cyclic loading ?

To answer this question, I will rely on different fatigue tests : cyclic strain-controlled tension-compression tests on pure Al (Wafa's PhD thesis [El May 2013]) and cyclic stress-imposed tension-tension tests on pure Cu single crystals (Gabriel's PhD thesis [L'Hôte 2019]). For the latter, several cyclic loadings were performed by increasing the stress amplitude on the very same [110] copper single crystal specimen (each step lasting 5000 cycles)⁴. While the dislocation motion was dynamically followed using AE, additional microstructural investigations were performed to identify dislocation structures using EBSD and R-ECCI in a SEM.

The materials studied being either single crystal or polycrystal with large grain sizes ($L_g \sim 5 - 10\text{mm} \gg$ internal length scale l), the degree of wildness W will not be affected by the grain boundaries.

2.3.1 Dislocation patterning along fatigue cycles

During strain-controlled cyclic loading, the crystal is deformed at every cycle and cyclic hardening, followed by cyclic softening (in some cases) and saturation of the cyclic stress amplitude are observed. In the case of pure Al we defined 5 stages (see section 1.3.2).

In this section, only the early stages of fatigue are considered (stages A and B of section 1.3.2), i.e excluding stage (C) with first signs of slip irreversibility and crack initiation along PSBs and excluding the stages corresponding to extensive damage and final failure (D and E), that I will develop in chapter 3. Therefore, the AE data can be linked exclusively to dislocation rearrangements. The absence of PSBs and crack initiation was checked during stages A and B. Also, since we are able to identify the onset of stage II for each fatigue test from unambiguous signatures of fatigue crack growth (see section 3.2.1 on AE multiplets), the nonexistence of such crack growth in the early stages is confirmed.

This has also been verified for stress-controlled test on pure Cu. Indeed, even if the stress levels for some fatigue steps are of the order of those required for the formation of PSBs during stress-imposed cyclic loading [Melisova' *et al.* 1997, Jameel *et al.* 2001], no PSBs are observed in our case as the mean stress is always higher than zero, due to the loading ratio ($R_\sigma = 0.1$). Otherwise, for stress-controlled

4. I will present the response to imposed rising alternating stress levels in section 2.3.2.

tests, only cyclic strain hardening followed by saturation is observed.

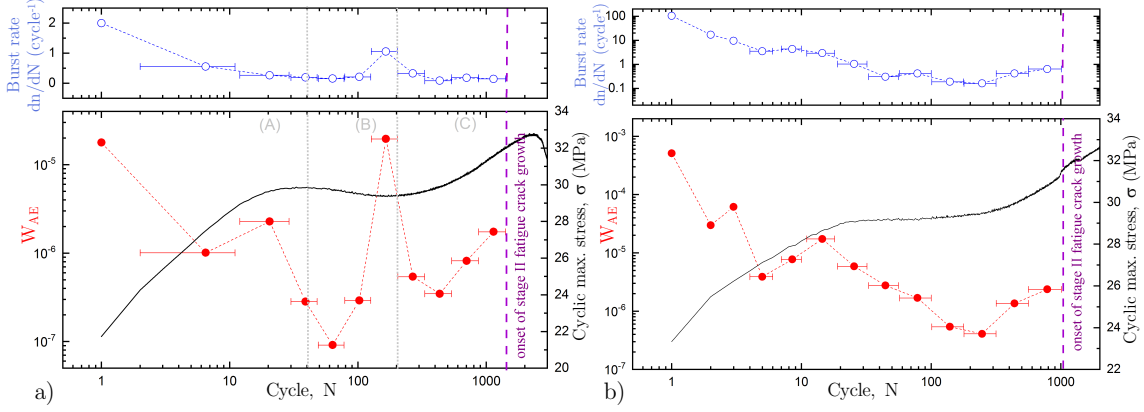


FIGURE 2.9 – Cyclic strain-controlled test ($R_\epsilon = -1$; $\Delta\epsilon = 0.95\%$) on (a) non-annealed and (b) annealed polycrystalline Al sample. Bottom panels : CSR curve (black line) and W_{AE} (red closed circles). Top panels : average AE burst rate (blue open circles).

During the early part of strain imposed tests on Al (stage (A)), both the average burst rate dn/dN and W_{AE} decrease (figure 2.9) : dislocation avalanches are progressively less numerous as a cell structure emerges. Note that both parameters exhibit very low values ($W_{AE} < 10^{-3}$), illustrating the extreme mildness of Al at bulk scales, in agreement with the fact that a wild-to-mild transition takes place in this material for a system size $L \approx 5 - 10\mu\text{m}$ [Zhang *et al.* 2017]. In tests on annealed Al samples (lower initial dislocation density ρ_i) dn/dN and W_{AE} are initially much larger than in non-annealed samples (figure 2.9 (b)), which is consistent with the understanding that annealed samples have a larger initial mean-free path for dislocations (scaling as $1/\sqrt{\rho_i}$).

Nevertheless, some episodic discrete AE bursts do persist at those scales. Gathering all the AE bursts recorded during this initial hardening stage (A), we obtain a power law cumulative distribution of AE energies (cf. figure 3.1(b) in section 3.1).

This means that, as seen in section 2.2.1, two phenomena compete, during this stage : collective dislocation dynamics, governed by long-ranged elastic interactions among dislocations, and the emergence of a self-organizing network controlled by short-range interactions and progressively inhibiting collective effects. If the first process is intrinsically scale-free and associated with power-law distributions of avalanche energies, the second one is characterized by an internal length scale l decreasing as the material hardens, which explains the decrease of the wildness measure in this initial hardening stage. Also, during this stage (A), the AE power peak value, recorded at plastic yield, decreases with the increasing number of cycles when cyclic strain hardening is important (figure 1.20 (bottom)). And so, consistently with the continuous AE source model, the mean free path decreases

(as $d\varepsilon_{pl}/dt$ remains roughly constant).

We find similar results in the case of fatigue of pure Cu single crystals. During constant imposed stress amplitude cyclic loading, while plastic strain mostly occurs during the very first cycles, the mild plasticity ($dE_c/dt \propto d\varepsilon_{pl}/dt$, still in coherence with the continuous AE source model) rapidly vanishes (figure 2.12(a)). This is the signature of strain hardening, due to the rapid multiplication and the subsequent entanglement of dislocations, which is expressed, in this stress imposed mode ($R_\sigma = 0.1$), by a vanishing of plastic strain amplitude $\Delta\varepsilon_{pl}$ over the cycles (figure 2.11(b)). This is directly related to a decrease in AE signals (both continuous and discrete) over the cycles.

Cyclic strain hardening, resulting from the development of a dislocation structure (ECCI images of figure 2.10 shows an example) is accompanied by a decrease of the dislocation mean free path (emerging from dislocation short-range interactions), observed by ECCI and by AE, through $\sqrt{S}^5$ (figure 2.11(c)).

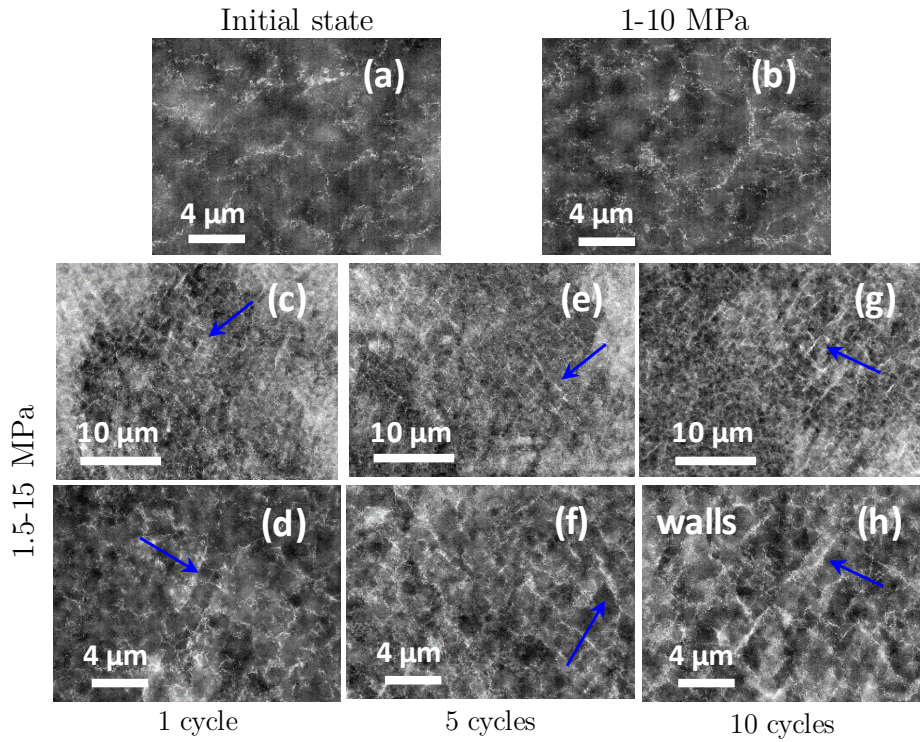


FIGURE 2.10 – Evolution of dislocation microstructure along the cycles of a stress-imposed fatigue test ($R_\sigma = 1$) on pure Cu (oriented in $[110]$ direction). a) Initial state b) After 5000 cycles at 1-10MPa RSS (c-d,e-f,g-h) After 1, 5, 10 cycles at 1.5-15 MPa RSS. Blue arrows indicate the position of a dislocation wall.

The dislocation structure quickly forms and becomes more stable with increasing

5. cf. section 2.2.2.2

cycling (no more plastic deformation nor acoustic activity occur after these 10 cycles, figures 2.11 and 2.13) which shows that the structure no longer evolves once it becomes stable enough to accommodate the applied stress. And, actually, it comes with a decrease of the number of dislocation avalanches and of the wildness of plasticity (W_{AE}) (figure 2.11(a)) over the first cycles.

Note that the values of W_{AE} are higher in the case of pure copper single crystals (at similar values of $\Delta\varepsilon_{pl}$), whose initial dislocation density is much lower. This is also consistent with the intermediate behavior that I presented in 2.2.1 with a clear coexistence of mild and wild plasticity at the beginning of the stress steps (figure 2.12(a)).

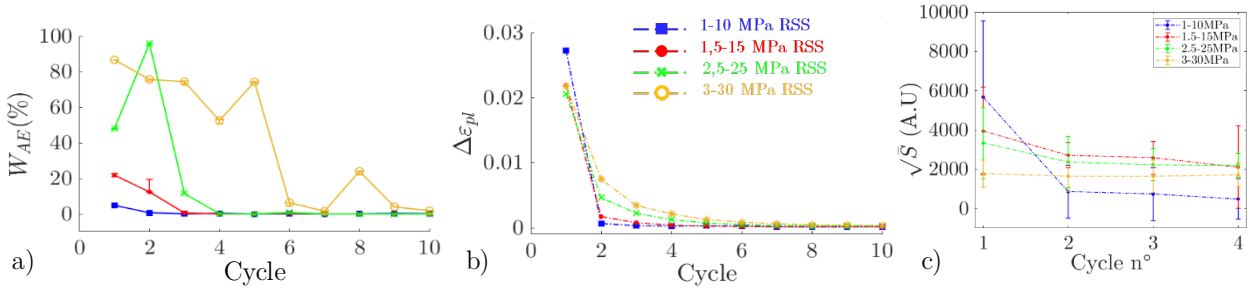


FIGURE 2.11 – Evolution of W_{AE} (%), $\Delta\varepsilon_{pl}$ and $\sqrt{S}$ during the first cycles of each cyclic step (on a unique pure Cu sample, $R_\sigma = 0.1$, at 4 different imposed stress amplitudes)

Otherwise, the peak of W_{AE} during the softening stage (B) of a non-annealed Al (figure 2.9(a)) suggests that a re-organization of the transient initial cell structure (verified by TEM observations [Videm & Ryum 1996]) occurs partly through (relatively) large dynamical instabilities. On the contrary, in the absence of a softening stage (case of annealed Al under strain-imposed fatigue tests, figure 2.9(b)), no peak of W_{AE} is observed, suggesting a more progressive evolution towards a well-defined wall pattern, without a need of complete re-organization of an initial, transient cell structure. The absence of W_{AE} peak after the hardening stage is also observed in the case of cyclic tests on Cu at a given amplitude where there is no softening stage, the dislocation structure accommodates the imposed stress amplitude.

Thereby, we can draw the conclusion that during the initial stages of cyclic deformation, a link is revealed on one side between strong dislocation patterning, hardening and mildness, and on the other side a link between microstructural re-organization, softening and wildness. With accumulating cyclic deformation, the total dislocation density increases and a dislocation pattern emerges. This leads to strain hardening and the reduction of the internal length scale l , which causes, in turn, a decrease of the degree of wildness of the plastic flow. The strong difference

in the degree of initial wildness between non-annealed (higher ρ_i) and annealed Al samples (lower ρ_i), (and pure Cu single crystals with very low ρ_i) as well as the fact that the severe microstructural re-organization (strong variations of ρ) accompanying softening (when present) is associated with a transient increase of wildness, are also consistent with this conceptual framework.

At the end of the day, we (partially) sweep the wild-to-mild transition in pure fcc metals within a given test by strain-hardening the material during cyclic loading. In this sense, the results reveal the sensitivity of the degree of intermittency of plastic flow (wildness) to the (emergent) internal length scale l .

Furthermore, we observed, during the initial stages of cyclic deformation, that the decrease of wildness with cycling is accompanied by an increase of the scaling exponent of the cumulative probability distributions for AE burst energies τ_E , meaning that a milder plasticity can be associated with larger values of τ_E . This anti-correlation between W_{AE} and τ_E is consistent with what I presented earlier and theoretical developments [Weiss *et al.* 2015, Zhang *et al.* 2017].

2.3.2 Reorganizations upon rising stress levels

To go forward in the study of the correlation between microstructural evolutions and dislocation patterning under cyclic loading on the one hand, and the nature of collective dislocation dynamics on the other hand, cyclic tests with different imposed stress amplitudes were performed, during Gabriel's PhD thesis. Stress-imposed cyclic loadings on the very same copper single crystal specimen (oriented in [110] direction) were performed with increasing stress amplitudes (figure 2.13) -each step lasting 5000 cycles at a given imposed RSS⁶ amplitudes, see appendix A-. The development of dislocation structures is characterized thanks to EBSD maps and ECCI images performed between each cyclic stress step.

Each stress amplitude increment leads to the formation of a new dislocation structure. The initial cell structure gives way to the formation of two coexisting structures : one made of dense walls (inside disorientation bands (DBs)) and one consisting of dense cells (figure 2.13(b), see appendix A for more characterizations). Upon increasing stress level, dislocation cells size and distance between walls gradually reduce while the dislocation density⁷ increases and the dislocation structure become denser.

Note that the scale of the dislocation structure can be characterized using three different methods : the quantity $\sqrt{S}$, obtained from a relation between the acoustic power and the plastic strain rate (equation 2.3⁸), the dislocation mean free path λ_{av} estimated from microstructural investigations [L'Hôte *et al.* 2019a] (an average mean

6. on 111 glide planes

7. The dislocation density is estimated from ECCI images using the same methodology as the one proposed in Ham works [55].

8. See continuous AE model in section 2.2.2.2

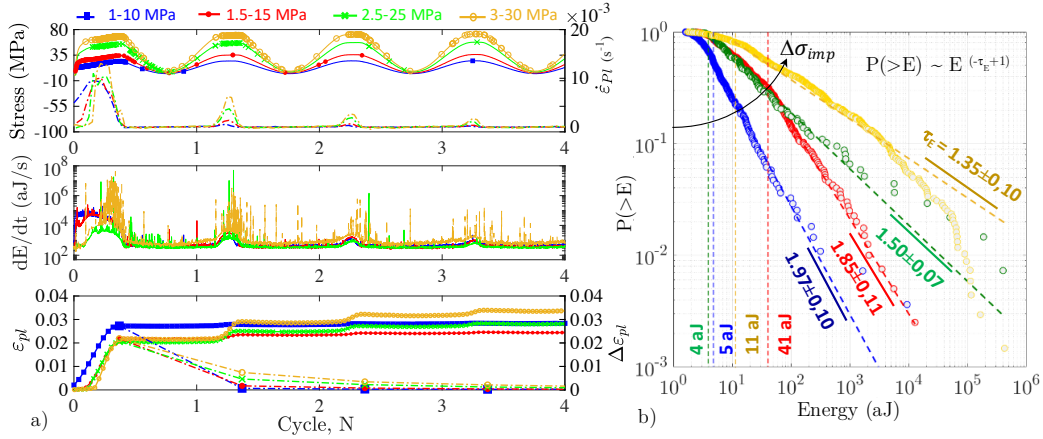


FIGURE 2.12 – a) Mechanical and AE response to imposed rising alternating stress levels on a sole Cu specimen. Over 4 cycles. (Top panel : the colored dots indicate the stress levels at which AE bursts were detected. b) Cumulative PDF of discrete AE energy (gathering all the bursts detected during all the cycles of each cyclic step). Vertical dashed lines represent E_{cutoff} for each step. The blue, red, green, and yellow colors refer to the 1-10, 1.5-15, 2.5-25, and 3-30 MPa RSS steps, respectively.

free path λ_{av} is manually measured from ECCImages) and $1/\sqrt{\rho}$. Indeed, a similar evolution of $\sqrt{S}$ and λ_{av} can be observed as a function of the dislocation density, from figure 2.13(c), which validates the continuous AE source model. Actually, we chose to compare $\sqrt{S}$, generated during the first cycle of each stress step (cycle where the main plastic phenomena occur), to the dislocation structure previously created. Since $\sqrt{S}$ is theoretically related to the characteristic sizes of the dislocation structure, it must be constrained by the dislocation structure obtained at the end of the previous stress step. And, the correlation between $\sqrt{S}$ and λ_{av} shows that, during the first fatigue cycle, the continuous part of the acoustic power (mild plasticity) keeps the memory of the dislocation mean free path of the previous microstructural state.

Nevertheless, these microstructural evolutions show that, as soon as the stress is increased, the former dislocation structure is no longer stable and undergoes major rearrangements, implying a rapid vanishing of this memory. Those reorganizations occurred at the beginning of the cyclic steps and took place, at least partly, through dislocation avalanches.

We have seen in the previous section that through cycling within a given stress step, the mean free path decreases (decrease of $\sqrt{S}$) as a new dislocation structure emerges. Simultaneously, the dislocation avalanche activity decreases, as illustrated by a decrease in the number of avalanches and of the wildness with increasing number of cycles (figure 2.11). However, we note that the larger the imposed stress amplitude, the more energetic the dislocation avalanches and the greater the wildness (increase of W_{AE} , figure 2.11). That is, the increase of wildness is accompanied by a decrease

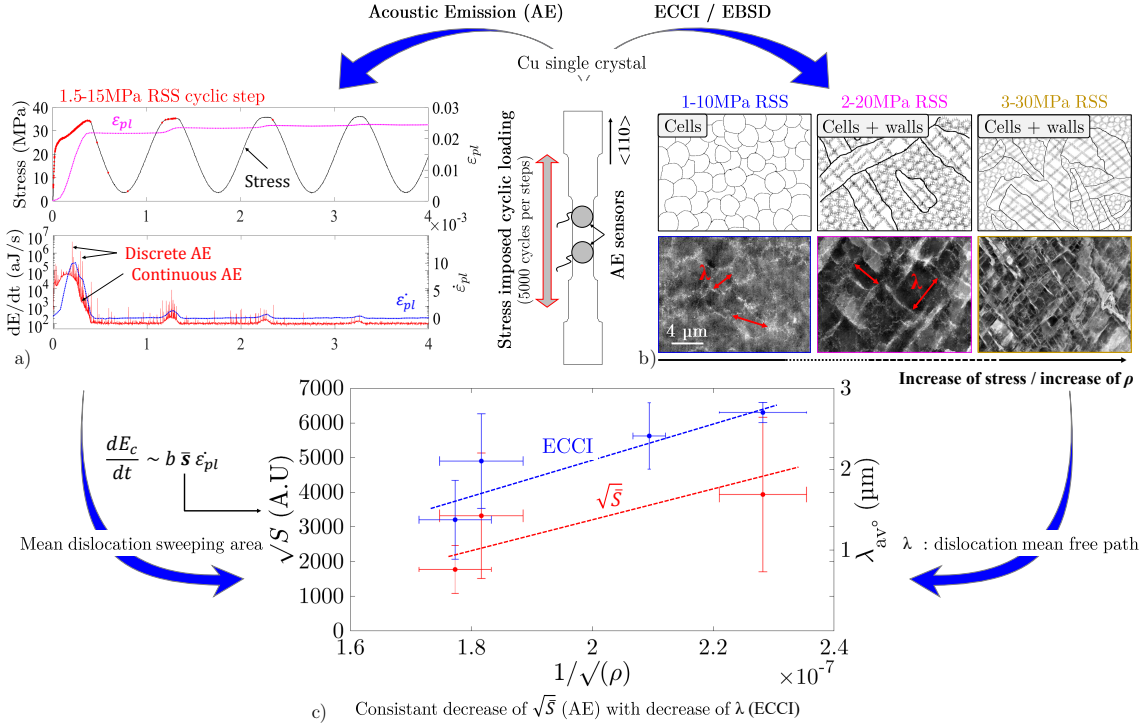


FIGURE 2.13 – Correlation between microstructural evolutions and dislocation patterning under cyclic loading

of the critical exponent value τ_E of the power law distributions (figure 2.12), which is consistent with what I presented in section 2.2.1.

In the case of rising stress, the wildness, this time, increases with the dislocation density and the dislocation mean free path decreases which may seem in opposition to what I presented before through cycling in a given stress step. However, this could result from the combination of both an increase of elastic energy brought to the crystal upon increasing stress, and the break-up of walls or cells containing a larger dislocation density (figure 2.11).

About 50 and 30 years ago, P. Neumann [Neumann 1969, Neumann & Neumann 1970] and C. Laird [Lorenzo & Laird 1982] observed strain bursts during imposed-stress cyclic loading of fcc metallic single crystals, as the consequence of a global collapse of the dislocation structure, in the form of dislocation avalanches gradually spreading throughout the crystal. In our study, avalanches are contained within the cycles and we show that they coexist with a mild plasticity, which is not discussed in the work of Neumann and Laird.

In other words, the reorganization of a denser dislocation structure occurs through more energetic dynamical events. Upon reloading at a larger stress amplitude, the dislocation structure is reorganized and dislocation avalanches can travel over distances much larger than the former mean free path. If the concept of dislocation mean free path seems adequate to explain the mild plasticity, questions can be

raised about its pertinence and meaning in the case of dislocation avalanches. This illustrates the complex interplay between dislocation avalanches and the emergence of a stable dislocation structure.

2.4 Influence of load history and activated slip systems on dislocation dynamics

We would now like to explore the influence of activated slip systems and of the imposed path or loading ratio on the dislocation (i) microstructure and (ii) dynamics. For this purpose, we performed, during Gabriel's PhD thesis, similar cyclic tests to those previously presented (section 2.3.1) on a pure copper single crystal whose crystallographic orientation favors the sliding of a greater number of systems. For orientation [110], 4 sliding systems are activated simultaneously compared to 6 for orientation [111]. In addition, several Cu samples, with [110] orientation, were subjected to different loading paths until the same specific stresses were reached : either through significant increases in the stress amplitude, as of the first cycle, or through the application of a progressive stress ramp, $R_\sigma = 0.1$. And similar tests as those analyzed earlier (section 2.3.1, loading in appendix A) were performed with a negative ratio $R_\sigma = -1$.

2.4.1 Influence of cristallographic orientation

Crystallographic orientation influences the dislocation pattern and acoustic activity. A double system of dislocation walls is observed homogeneously in the sample [111] and gradually forms as the stress amplitude increases. The plasticity processes are exclusively concentrated in the first cycle of each fatigue stage for [111]. The dislocation mean free path decreases as the stress increases and walls are formed. Just as for the [110] oriented specimen, W_{AE} increases with stress, especially from the moment the first dislocation walls appear (figure 2.14). This implies that these avalanches are partly the result of the collapse of the dislocation walls during loading. As a result, in the first cycle, the microstructure is (i) increasingly swept by avalanches as the stress increases and (ii) reorganizes immediately afterwards. Overall, the avalanche activity is lower in [111] than in [110]. This seems to be due to the higher number of possible avalanche sources in [110] due to the heterogeneity of the structure (disorientation bands, cells, walls, etc.) and to the higher number of sliding systems in the [(111)] oriented sample : the interaction between dislocations and strain-hardening is faster.

2.4.2 Influence of loading path and ratio

The loading path (either large increases in stress amplitude from the first cycle or progressive stress ramp) does not affect the arrangement of dislocations in the case of imposed-stress tests ($R_\sigma = 0.1$). However, for a progressive stress ramp, even if acoustic activity is present at each cycle as the material deforms,

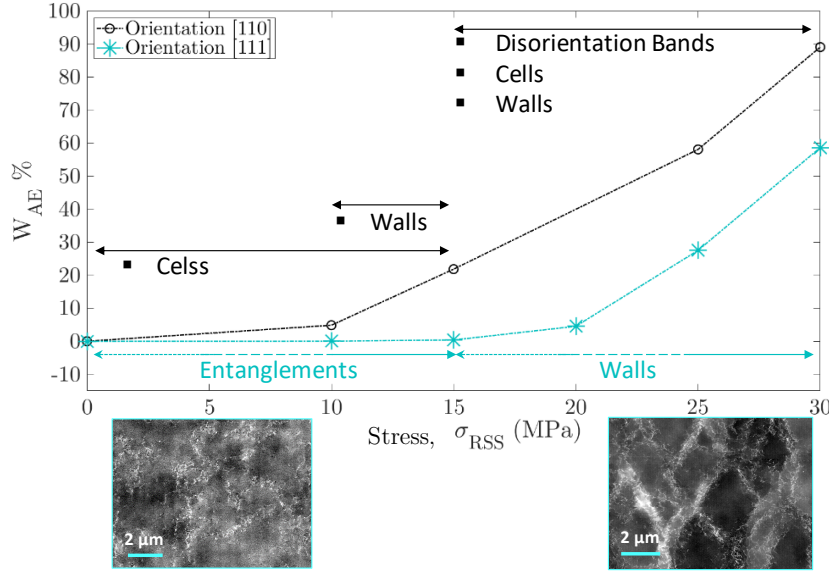


FIGURE 2.14 – Wildness of plasticity W_{AE} with applied stress amplitude for a Cu single crystal with [110] orientation (black) and [111] orientation (blue).

W_{AE} remains low, suggesting that the very gradual increase in stress amplitude does not allow dislocation avalanches to be strongly generated and that they are likely to be restricted in their propagation. Dislocation avalanches are, however, generated when a dense structure of dislocations is formed (DBs, cells and walls). The mean free path (measured from $\sqrt{S}$) increases during the first cycles, showing that no dislocation structure is yet formed, and then decreases, symbolizing the interactions between dislocations and the gradual emergence of a dislocation structure.

The loading ratio ($R_\sigma = 0.1$ vs. $R_\sigma = -1$), on the other hand, has a great influence on the implementation of the dislocation structure and on the dislocation dynamics. Unsurprisingly, the dislocation structure is always modified with increasing stress. The DBs containing walls and dislocation cells observed at $R_\sigma = 0.1$ (see section 2.3.1) give way, in the negative ratio test, to the first development of a cell structure, which, with increasing stress, will gradually convert into dislocation veins, then into PSBs and then into dense dislocation cells (figure 2.15(a,b)).

As we have seen for aluminum, the acoustic power increases during the tensile and compressive phases (figure 2.15(c)), illustrating the back-and-forth effect of unpinned dislocations.

This time, the continuous component of the AE power, dE_c/dt , decreases slowly while the wildness of plasticity, W_{AE} , still decreases rapidly over the first cycles (figure 2.15(e)). This suggests that (i) the present dislocation structure is partially reorganized during the first cycles, (ii) the emergence of a new avalanche-blocking structure is rapid, and (iii) its consolidation requires a large number of cycles. For

larger stress amplitudes, the PSB-type structure forms. The plastic deformation increasing at each cycle (figure 2.15(d)) indicates that a stable microstructure configuration is not achieved and reflects a gradual transformation of the structure from matrix to PSBs. The presence of dislocation avalanches and W_{AE} peaks after a large number of cycles (figure 2.15(c,e)) suggests that the structure is regularly rearranged : collapse of the matrix structure allowing the formation of new PSBs or collapse of some PSBs leading to the formation of dense dislocation cells.

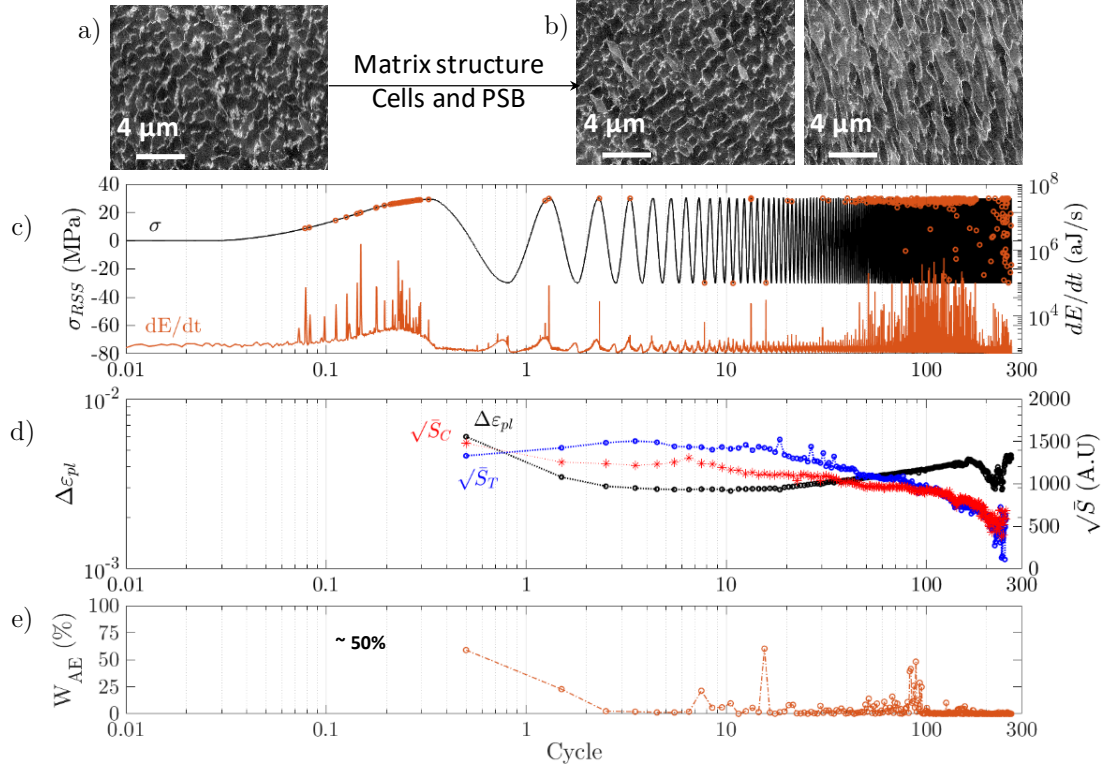


FIGURE 2.15 – a) Dislocation structure after 5000 cycles at $-25/25\text{MPa}$ RSS. (b) Dislocation structure after 5000 cycles at $-30/30\text{MPa}$ RSS. (c) Stress (black) and dE/dt (orange) along cycles. (d) $\Delta\epsilon_{pl}$ (black) and $\sqrt{S}$. (e) W_{AE}

Damage

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After discussing the initiation of plasticity in semi-cristalline polymers and dislocation mediated plasticity in **fcc** metallic materials, I will move forward in the process of damage from micro-crack initiation to global failure.

3.1 Micro-crack initiation

As stated in chapter 2, dislocation patterning under cyclic loading of **fcc** metals gives rise eventually to **PSBs** formation [Laird *et al.* 1986], which is known to be the phenomenon at the origin of fatigue crack initiation.

As I already mentioned, Laird and co-workers reported, more than 30 years ago, directly from high-speed recording of the stress-strain hysteresis loops, the existence of transient mechanical instabilities during the cyclic loading of pure **fcc** metallic single crystals that they called "strain avalanches" [Yan & Laird 1985, Buchinger *et al.* 1985, Lorenzo & Laird 1982]. These authors interpreted these events as collective and coordinated dislocation motions within **PSBs** and argued that such plastic instabilities might be associated with fatigue crack initiation. However, it was later understood that these instabilities could be largely machine-induced, providing only a highly distorted view of true plastic avalanches . As a result, this topic was largely abandoned.

Today, with the various studies we have carried out, we investigate whether there is indeed a link between plastic avalanches, **PSBs**, and fatigue crack initiation.

In section 2.3.1, only the hardening and softening stages (A and B) of strain-controlled fatigue tests on aluminum were investigated. I will now focus on the

secondary hardening stage (C), associated with the development of a well-defined structure and the emergence of PSBs. The AE data can presently be linked to dislocation rearrangements and microcrack nucleation along primary slip planes on stage I of the fatigue life of a sample. We have indeed observed at this stage the first signs of slip irreversibility and crack initiation along PSBs appearing at the surface (figure 1.11 (c)).

Just after reaching a minimum at the beginning of this stage (C), the wildness measure W_{AE} increases again of about one order of magnitude (figure 2.9). Hence, the unequivocal relationship between mildness and hardening, discussed in chapter 2, breaks down here, suggesting another origin of acoustically detectable avalanches. Instead of microstructural rearrangements within the wall-and-ladder matrix structure, the recorded AE bursts may be the signature of collective and coordinated dislocation motions along PSBs leading to the formation of incipient microcracks. This hypothesis is supported by the evolution of AE data during this stage.

If we look at the occurrence of the discrete AE activity within the loading cycle (figure 3.1(a)), the burst activity is roughly symmetric in tension and compression in the early stages, concentrated at plastic yield and, to a lesser extent, during the plastic plateau. In fact, no bursts are triggered during unloading phases, which is in agreement with their plastic origin. But, during the secondary hardening stage, a possible emergent asymmetry (more bursts triggered under tension) might be consistent with incipient microcracking, although this point has to be taken with caution owing to limited statistics.

The proposition is above all endorsed by the increase of the burst rate dn/dN at this stage (figure 2.9) and the evolution of the cumulative distribution of AE burst energies along the fatigue stages (figure 3.1(b)).

As already mentioned, the initial hardening stage is characterized by a power law cumulative distribution of AE energies. Afterwards, the cumulative distribution drifts away from a power law to a log-normal-like distribution for small size events with several outliers during the primary softening stage (B). Actually, this distribution emphasizes the possible coexistence of mild and wild fluctuations in crystal plasticity and it corroborates the relation between strong dislocation patterning, hardening, and mildness on one side and the link between microstructural reorganization, softening, and wildness on the other side, that I presented earlier. Finally, a power-law tail appear to be emerging again in the energy distribution as the system reaches the onset of stage II fatigue crack growth : plastic avalanches do exist during this stage (C). Therefore, this would be the signature of plastic instabilities associated with microcrack nucleation along primary slip planes, reviving and supporting the initial proposal of Laird and coworkers [Yan & Laird 1985, Lorenzo & Laird 1982].

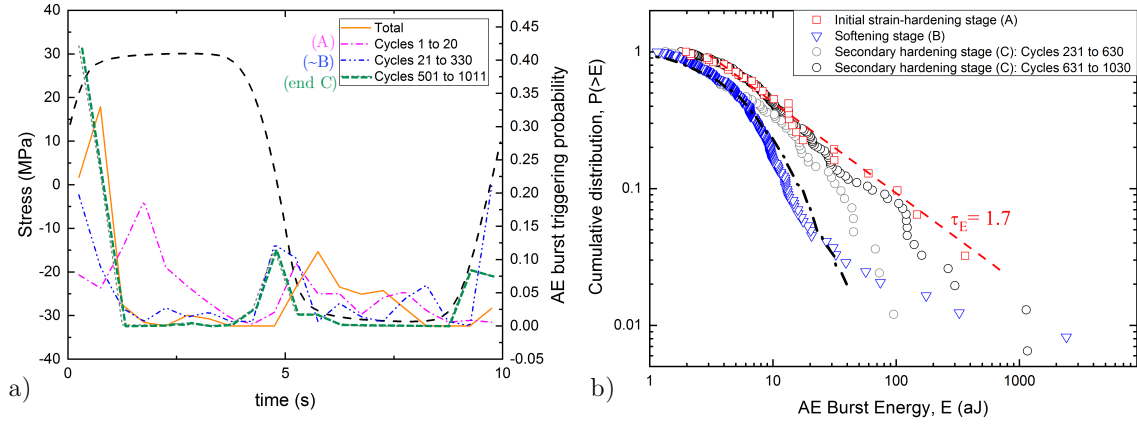


FIGURE 3.1 – a) Occurrence of the discrete AE activity within the loading cycle. All the bursts recorded during the different stages of deformation have been gathered together to build these histograms. b) Cumulative probability distributions for AE burst energies recorded at different stages of cyclic deformation. The black dash-dotted line represents a corresponding log-normal distribution for the softening stage, while the red dashed line exemplifies the power law tail associated with the initial strain-hardening stage. (For strain-controlled tension-compression with $R_\epsilon = -1$; $\Delta\epsilon = 0.95\%$ tests on Al.)

3.2 Fatigue cracks

Earlier, I promised to outline the final stages of fatigue. Now is the time, and more than that, I'm going to focus on the fatigue cracks.

In metals, steels and engineering alloys, the mechanisms of fatigue crack nucleation and propagation, precursors of terminal failure, are well identified. We have seen that under cyclic loading, plastic deformation localizes along **PSBs**. As first postulated by Wood [Wood 1958], this irreversibility and the associated surface roughness generate stress concentrations, hence sites for crack nucleation [Sangid 2013].

Unlike generalized damage evenly spread within a structure, the presence of one or few fatigue cracks does not modify significantly the global mechanical properties of the material such as elastic stiffness. As stage II propagation often occurs within the bulk, their detection and monitoring from visual inspection is also problematic, whereas X-ray tomography (e.g. [Buffiere *et al.* 2006, Dezecot *et al.* 2016]), although a very powerful technique, in particular as it allows in situ observations of short fatigue crack growth [King *et al.* 2011, Buffiere 2017], can be hardly applied to structural components in service. Propagating slowly, without clear signature in terms of macroscopic mechanical behavior, the fatigue crack eventually attains a critical length such that the stress intensity factor at crack tip reaches the fracture toughness during the loading cycle, triggering an unstable propagation and global failure (stage III).

Even if lifetime and safety prediction methodologies based on the non-destructive monitoring of mechanical properties [Constantinescu *et al.* 2004] have been proposed, the early detection and monitoring of fatigue crack growth (FCG) remains a crucial challenge which motivated numerous studies tempting to detect a modification of material properties by means of electrical resistivity, ultrasonic energy, electromagnetic impedance or electrochemical properties. A common limitation of these non-destructive methods is that they detect a modification of a physical property averaged at the scale of the structure or the component, hence are hardly sensitive to the effect of a single (or few) crack in its early stages of propagation, and faced with classical signal/noise issues. This can lead to too late alarms.

AE has therefore been proposed as a monitoring tool of fatigue for a long time [Harris & Dunegan 1974]. However, most of these early works as well as more recent ones [Roberts & Talebzadeh 2003, Berkovits & Fang 1995] recorded the global AE activity, such as the number of AE counts, or detected bursts per cycle, or AE entropy [Chai *et al.* 2018, Sauerbrunn *et al.* 2017] and shared limitations with the other non-destructive methods listed above : tracking the slow crack growth from a global measure is difficult and highly sensible to the SNR. This is reinforced by the non-specific nature of these AE measurements, as different mechanisms can generate AE during fatigue, such as dislocation avalanches (as I presented before), phase (e.g. martensitic) transformations [Shaira *et al.* 2008], twinning [Shaira *et al.* 2008], or fracture processes [Davidsen *et al.* 2007], not to mention environmental/mechanical noise.

Several approaches were developed to discriminate the AE signatures of different source mechanisms : Shaira *et al.* [Shaira *et al.* 2008] used e.g. the k-means approach for clustering AE signals into different classes (representing mechanisms) in 304L stainless steels. Several other approaches in AE pattern recognition and signal source mechanism identification were developed on other materials [Godin *et al.* 2004, Maillet & Morscher 2015, Moevus *et al.* 2008, Sibil *et al.* 2012] but their in-service feasibility is yet to be demonstrated.

More recently, Aggelis *et al.* showed that certain AE characteristics undergo clearly measurable changes much earlier than final fracture [Aggelis *et al.* 2011] and used both infrared lock-in thermography and AE to determine fatigue limit [Kordatos *et al.* 2012, Kordatos *et al.* 2013]. Also, they used the RA value (ratio of rise time to amplitude of the WFs) which quantifies the shape of the first part of the AE signals for characterization of the cracking mode. Chai *et al.*, as for them, suggested that the AE technique is sensitive to the identification of the presences of stage IIa, stage IIb in the Paris region and the shear crack growth during FCG (change of slope in AE cumulative count and cumulative energy vs. ΔK) [Chai *et al.* 2017].

3.2.1 Precursors to rupture in fatigue : acoustic mutliplets

I present here a way to detect fatigue cracks, in metallic materials, much earlier than final failure, before any macroscopic sign of material damage.

We have revealed, for the first time, in different metallic materials, the detection of AE bursts specific of fatigue crack growth during cyclic loading [Deschanel & Weiss 2018a, Deschanel & Weiss 2018b, Deschanel *et al.* 2017]. These so-called acoustic multiplets are characterized by highly correlated WFs, signature of a unique source. They are repeatedly triggered over many successive loading cycles at nearby stress levels and originate from a single location (using cross-correlations, the relative source locations of the similar AE signals can be determined with improved accuracy, see following section 3.2.3). These results are drawn from uniaxial strain-controlled cyclic fatigue tests performed on pure aluminum and several other fcc metals (304L austenitic stainless steel, polycrystalline electrolytic copper and Copper-Cobalt-Beryllium alloy), mainly performed on the tailor-made fatigue machine with optimized SNR.

Figure 3.2 shows the occurrence of AE signals during the loading (number of cycles, N), at specific level of stress σ within the cycle. We notice the remarkable feature : the presence of clusters consisting of bursts triggered every cycle at almost the same, or at a slowly evolving, stress. These sequences can last hundreds of cycles. The bursts of a given cluster are characterized by very similar WFs, with cross-correlation coefficients always larger than 0.8, generally greater than 0.99¹ (figure 3.3(a) and (b)).

AE multiplets have been identified during the monotonic compressive failure of a salt sample [Moriya *et al.* 2006] or during tension of CFRP composites [Mills-Dadson *et al.* 2017], but this is the first time that they are reported during fatigue.

Owing to their specificity, they can be easily discriminated from other AE sources or environmental noise, i.e. are less subjected to noise/signal problems, and can be detected much before any modification of macroscopic properties (the first multiplets were generally detected before 2/3 of fatigue life).

These repeating AE bursts are reminiscent of repeating earthquakes, or multiplets, first identified in the 80's [Geller & Mueller 1980]. In seismology, these multiplets are interpreted as repeated stress releases at a same asperity along the fault. In this case, the repeatability is not related to a cyclic loading but to intermittent instabilities (stick-slip) under a slow far-field driving. Indeed, multiplets have been used to evaluate repeating earthquakes and more specifically estimate the slip rate and the relationships between slip behavior and earthquakes [Matsuzawa *et al.* 2004]. In our case, we interpret these multiplets as the specific signature of the slow, incremental propagation of stage II fatigue crack at each cycle (leaving striations on crack surfaces, figure 1.16) or the rubbing along its faces (fretting). They are not experimental artifacts, as (i) they are localized inside the samples, (ii) we recorded

1. A cross-correlation study is conducted on the WFs (within the multiplets, or all over the test) : a measure of similarity between two signals as a function of a time lag.

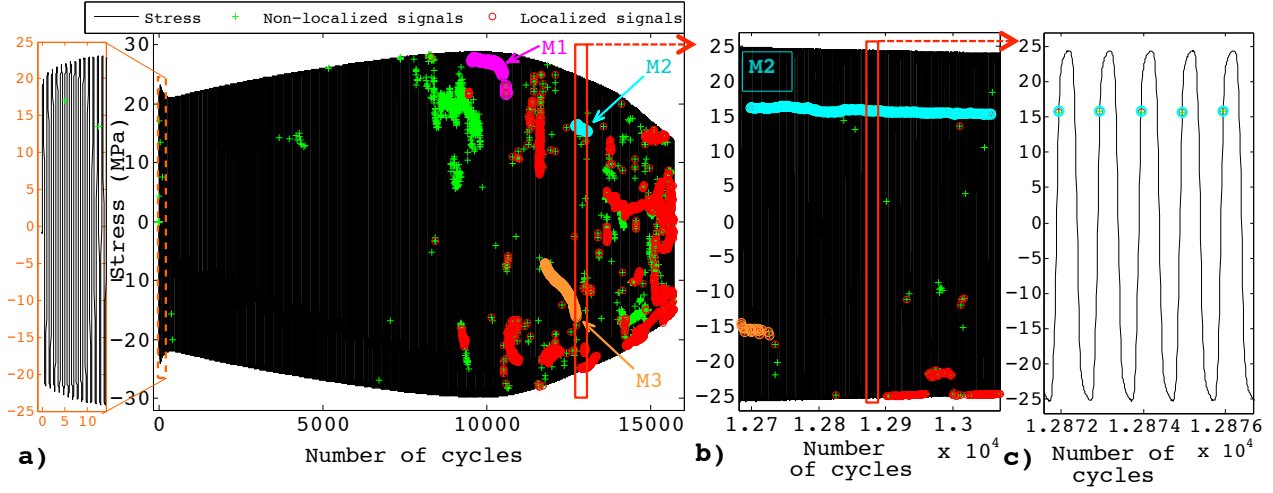


FIGURE 3.2 – Acoustic activity during a fatigue test ($\Delta\varepsilon = 0.5\%$ on aluminum at $0.1Hz$) : stress vs cycles with non localized AE signals (green crosses) and localized signals (red circles). Magenta, cyan and orange circles correspond to multiplets M1, M2 and M3 respectively. b) Enlargement on M2 and c) on some loading cycles.

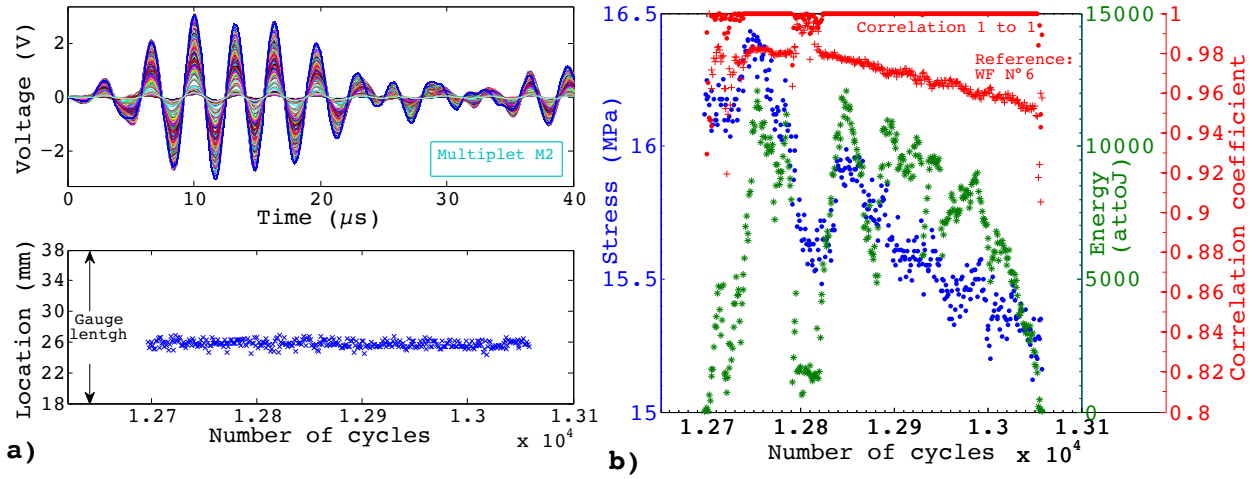


FIGURE 3.3 – WF composing multiplet M2 from figure 3.2 and location of the localized signals (AE signals occurring every cycle at $\sigma \approx 16MPa$ during ~ 360 cycles). b) Corresponding triggering stress (blue) and energy (green) of the WF over the cycles, as well as correlation coefficients of one WF of M2 as a function of the following one in the multiplet (closed red circles) and correlation coefficients between the 6th WF and all the others (red crosses).

them during tests performed with two different loading machines (iii) but not during the cyclic loading of samples below the elastic limit, or during the early stages of fatigue. If multiplets can be observed either in tension or compression, they are never symmetric, and not triggered at plastic yield. Under conditions for which the plastic zone ahead of crack tip is larger than the grain size, a situation encountered in our tests on ductile materials where macroscopic plastic yield is crossed over at each cycle (figure 3.2(c)), stage I crack growth is very short [Suresh 2001], hence hardly compatible with the repeatability of multiplets over hundreds of cycles.

During the first 60% of fatigue life, multiplets were mainly detected under tension (for instance multiplets M1 and M2), hence related to crack growth (see figure 3.5(a) for detection of multiplets within the cycle). Considering a crack of length a extending uniformly by an increment da along the crack front of length l , the AE wave energy scales as $E_{AE} \sim K_I l (da)^{(3/2)}$, where $K_I \sim \sigma \sqrt{a}$ is the SIF at crack tip [Evans 1979]. Although fatigue crack growth is triggered for K_I -values much below K_{Ic} , and the conditions for Linear Elastic Fracture Mechanics (LEFM) are not obeyed in ductile materials, one can reasonably assume incremental growth to be, in average, triggered at lower tensile stresses when the crack length increases. This trend was generally observed, although sometimes accompanied by shorter term fluctuations (figure 3.3(b)).

In the later stages of fatigue, we also observed AE multiplets under decreasing and, to a lesser extent, increasing compression (figure 3.5(a)) (e.g. multiplet M3). The unlocking of a frictional asperity during either crack closure or re-opening, i.e. rubbing of crack faces, is the most plausible source mechanism in this case.

3.2.2 Coda Analysis

Although multiplets can be tracked over hundreds of cycles, they generally fade out before final failure (figure 3.2). This does not necessarily mean that the associated crack stopped growing. In these ductile Al samples, multiple cracks are activated and the final cyclic softening stage is characterized by extensive damage.

As mentioned in Chapter 1, section 1.2.1.2, we have to bear in mind that the waveform $W(f)$ results from the convolution of the source signal $S(f)$ and of transfer functions in the frequency domain depending on the acquisition chain : $W(f) = S(f) \times G(f) \times D(f)$. Assuming that the medium $G(f)$ changes little over a short interval during the test, two similar AE signals received by one sensor $W_1(f) \approx W_2(f)$, in a small period of time, imply two similar sources $S_1(f) \approx S_2(f)$ as $D(f)$ is unchanged.

The initial, most energetic part of the signal (0-40 μ s in our analyses), mainly controlled by the source characteristics $S(f)$, remains highly correlated over most of the multiplet lifetime, then decorrelates suddenly (figure 3.3(b)). This final decorrelation may indicate a previously isolated crack starting interacting and coalescing with others, or, under compression, a modification of the characteristics of the asperity, hence changing significantly the emitted WFs. On the other hand, the limited but progressive decorrelation of the WF during the multiplet lifetime might

result from slowly evolving source characteristics, and/or from a modification of the traversed medium $G(f)$ as the result of increasing damage. This last mechanism is better characterized by the evolution of the diffusive coda (sampled in the interval 60-100 μs on figure 3.4(a) for multiplet M3 of figure 3.2). This coda, resulting from multiple scattering of the emitted elastic wave on internal defects, samples the damage spread within the material [Schurr *et al.* 2011]. Its decorrelation rate during the multiplet lifetime is much larger than that of the more energetic part of the WF (figure 3.4(b)), signing a progressive damaging during fatigue cycling of this Al sample. This is also emphasized with the evolution of the power spectral density during the multiplet (not shown here, see [Deschanel *et al.* 2017]). Similar results were obtained for the other samples and other materials. Therefore, using the repetitive AE at each loading cycle as a "natural" generator of a nearly identical source, such analysis of the diffusive coda could be used to track the average evolution of damage.

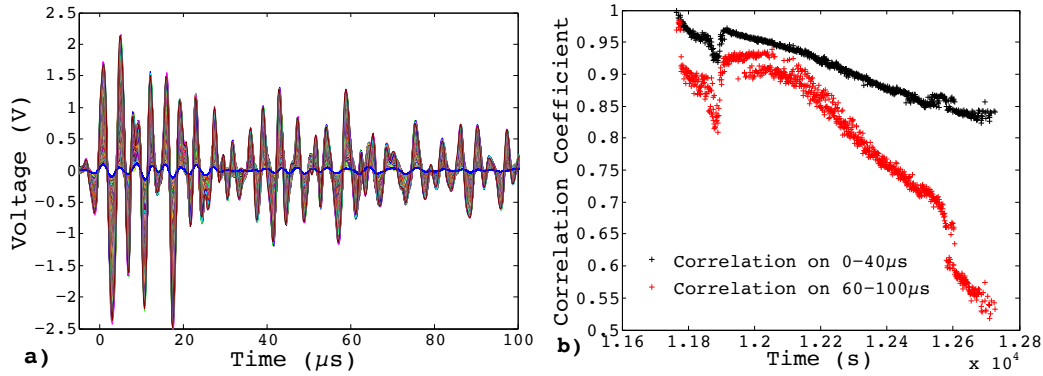


FIGURE 3.4 – (a) 933 WFs of multiplet M3 of figure 3.2's fatigue test. (b) Correlation coefficients between the first WF and all the others, when the correlation is performed on the first 40 μs (black dots) and on the interval 60-100 μs (red crosses) of the WFs corresponding to the coda waves.

3.2.3 A new way to localize

Classical procedure

As presented in section 1.2.1, with two sensors, a 1D-localization along the specimen length is possible from the time difference of arrival Δt of each WF at the two sensors (arrival times, given by the data acquisition system, being determined from first threshold crossings). I already presented the limitations of this method in section 1.2.1.7 (Δt strongly dependent on the chosen threshold, erroneous arrival times, etc.).

The classical procedure described in section 1.2.1.7 gives systematically dubious shifts in the absolute position of the source within the multiplet (figure 3.5(b), black squares) that would be in contradiction with a common origin.

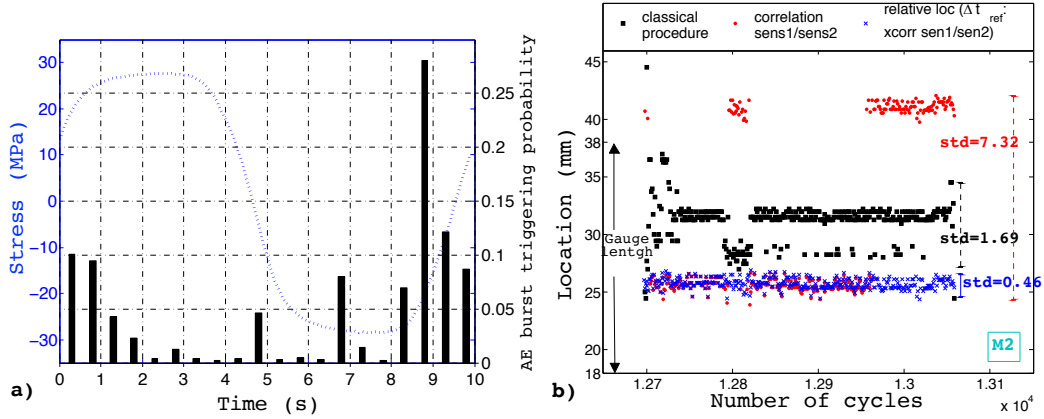


FIGURE 3.5 – Fatigue tests on Al at 0.1Hz, $R_\epsilon = -1$. a) Detection of multiplets within the cycle (for $\Delta\epsilon = 0.5\%$ and 0.95%). The dotted blue line represents a typical loading cycle for such strain-imposed fatigue tests. (b) Localization of the AE signals of multiplet M2 of figure 3.2, using the classical procedure (black $\square$), the Grabec’s methodology (red $\circ$) and our method : the correlation of WFs within a multiplet (blue $\times$).

Localization using correlation between waveforms

To circumvent these limitations, I presented in section 1.2.1.7 alternative methods to determine the location of AE sources and measure a reliable wave arrival time. Among other procedures, to improve the accuracy on Δt , Grabec proposed to cross-correlate the signals of a same event received by different transducers, with Δt maximizing this cross-correlation [Grabec 1978]. However, this methodology leads to unphysical shifts in position within a given multiplet (figure 3.5(b), red dots), as in most cases, except for early stages of damaging, the cross-correlations between WFs recorded at sensors 1 and 2 are non-significant (in the range 0.3-0.8, this remaining correlation resulting from the resonant character of the AE transducers). This illustrates the shortcomings of this method when the medium is too damaged and therefore the condition $G_{sen1}(f) \approx G_{sen2}(f)$ (linked to the medium) no longer fulfilled.

On our side, we are taking advantage of the correlated WFs within the multiplets, recorded at the same sensor, to improve the location of the AE signals and to present a new way to map the sources. In this case, $D(f)$ (characteristic response of the sensor) is unchanged by definition, whereas $G(f)$ remains almost unchanged as long as we consider only the initial impulsive part (0-40 μs) of the WFs (section 3.2.2 above). Inspired by studies of seismic multiplets (or repeating earthquakes) (e.g. [Poupinet *et al.* 1984, Rubin *et al.* 1999, Lengliné & Marsan 2009]), we can perform a relative localization of the different WFs in the multiplet using the time delay δt found between two WFs in a multiplet².

2. I explain the procedure in details in appendix B

We show in figure 3.5(b) (blue crosses) that taking advantage of the correlated WFs within a multiplet allows suppressing the spurious shifts in position obtained with the classical or the Grabec's procedures. This strongly argues for a unique source.

Thereby, we have revealed for the first time the presence of repeating nearly identical AEs during fatigue testing of metals. Originating from a unique source and triggered at each loading cycle at close stress levels, these acoustic multiplets are a specific signature of incremental fatigue cracking. Their detection thus open the way towards a new possible reliable monitoring of the onset of fatigue cracking during mechanical tests, or within structures in service, as the first multiplets were generally detected before 2/3 of fatigue life, before any macroscopic sign of material damage such as cyclic softening.

3.3 Fractoluminescence

If I pursue my idea of following the life course of a material along this manuscript, it seems that we are approaching the end, that is to say the rupture of the material. I will not venture here into the broad field of fracture dynamics, but I will present acoustic and light measurements as a crack grows in an inorganic crystal scintillator until its global rupture. And even if it raised many more questions than answers, this study could shed some light (!) on the fracture mechanisms.

Our study of fractoluminescence³, has actually its origins in astroparticle physics. Rare-event searches in particle physics, like those looking for particle dark matter (that may make up the bulk of the matter in the Universe), employ draconian measures to reduce all forms of background that could hide their signal. And, less than 10 years ago, several dark matter searches have observed intriguing events incompatible with known backgrounds [Angloher *et al.* 2012]. That is why my colleague at Queen's University, Philippe Di Stefano decided to study fracture as a potential background in rare-event searches, in particular as a source of light in the experiments deploying scintillating detectors. And, on the material science and fracture physics side, we (along with my colleagues from ILM, Institut Lumière Matière, and the Physics Laboratory at ENS-Lyon) believe that combined light and acoustic measurements might offer new ways to explore fracture processes.

3.3.1 Correlation between light and acoustic emission

For our purpose, after various tests, we converged on scintillator samples compressed in the DCDC geometry (see section 1.2.2.4) in order to perform controlled crack growth experiments. Our preliminary study [Tantot *et al.* 2013] demonstrated

3. Fractoluminescence, emission of light from fracture, is presented in section 1.2.2.4

that in certain scintillating materials used for particle detection, fracture was accompanied by emission of light with the same spectrum as the visible scintillating light, and that fractures could therefore contribute to backgrounds in rare-event searches. We have evidenced a strong correlation between mechanical-stress-induced emission of photons and of acoustic phonons, at room temperature and in a normal atmosphere (figure 3.6(a)). Besides, at least for BGO, event amplitudes for all channels are power-law distributed (figure 3.6(b)). For both AE channels, the exponents are 1.7 ± 0.2 (errors are one standard deviation). Assuming the AE energy is proportional to the amplitude squared, the power-law distribution of the energy would have as an exponent 1.4 ± 0.1 . This is compatible with the exponent obtained for the light channel 1.2 ± 0.2 , for which amplitude is proportional to energy. A more precise measurement might check if the absolute value of the AE exponent is in fact larger than that of the light channel, an indication of dissipation in the acoustic channel. The waiting time distribution of the acoustic and light events are also power-law distributed with compatible scaling exponents. This indicates that both types of emissions have a common origin.

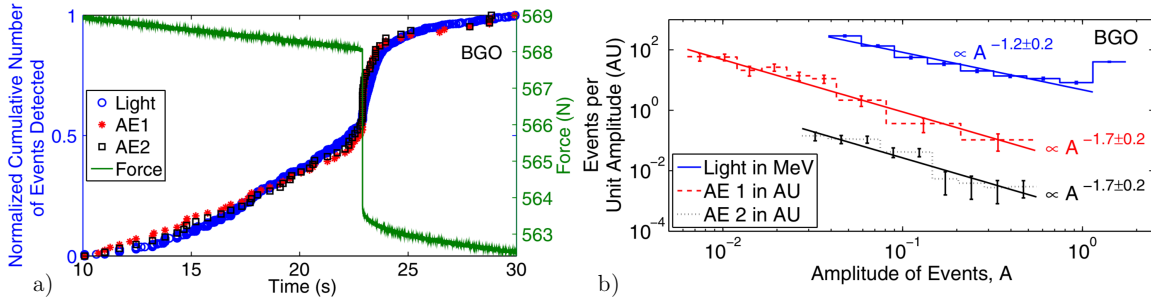


FIGURE 3.6 – (a) Normalized cumulative number of events on AE and light channels around the main fracture, of a compressed DCDC BGO sample. The drop in force signaling the main fracture corresponds to a sharp increase in the event rates for photons and AE. (b) Amplitude distributions for the light and AE. Amplitude of the light channel is proportional to energy. All distributions are fitted by power laws (up to saturation, for light).

A finer analysis of the light emission down to the nanosecond level pointed out that the photons emitted during the fracture were grouped in clusters. These photon clusters are the small units which are actually studied when building the energy and waiting time distributions at the large scale. Thus, the distribution of these clusters are directly related to the crack propagation. We defined two types of photon clusters : the short cluster (1 ms) and the large cluster (1 s). The short clusters have a duration comparable with the acoustic events. The photon distribution within these types of cluster remain to be investigated in further experiments. Preliminary results show that the temporal statistic is different in each type of clusters (they exhibit different power law exponents).

We also argued that light emission could give new insight on the rupture dy-

namics and provide quantitative information about the energy fracture budget. At least 3.10^{-5} of the elastic energy released by fracture would be converted into light energy but this result should be taken with caution. A fuller understanding of the energy budget requires knowledge of the energy transformed into phonons, or heat.

3.3.2 Effect of atmosphere

3.3.2.1 Ambient atmosphere

To fully benefit from the excellent resolution of the photomultiplier (PMT) (single photons with nanosecond timing), to be able to follow the actual crack propagation, and to control the atmosphere around the sample, we have developed an enhanced multi-channel setup, described in section 1.2.2.4. It allows us to measure accurately light emissions, acoustic emissions, crack velocity, compression and loading force during stable and fast crack propagation in controlled atmosphere.

The applied compression stress allows to control the velocity of both cracks up to a critical length, at which point the specimen abruptly breaks into two parts (fast fracture) [Pallares *et al.* 2009]. The slow crack propagation below the critical length is not only determined by the applied stress but also by subcritical rupture processes - i.e. a thermally activated energy path at the crack tip which ease bond breaking. The surrounding gas is a well-known agent that may reduce the critical energy barrier required for thermally activated fracture [Rice 1978].

A typical fracture test at ambient pressure with a BGO sample is presented in figure 3.7 (after a ramp of force, the force is kept constant). Other tests with increasing constant force segments have also been performed. Three regimes may be observed from the propagation of two opposite cracks : (1) the reopening of the previously initiated crack, (2) a slow crack growth up to a critical length and (3) the fast fracture, cleaving the material in two. AE is correlated with the whole crack propagation (figure 3.7 (c)) and the slow crack propagation is linearly correlated with AE. In these conditions, light emissions are prominently observed at the moment of fast fracture.

There are at least two possible mechanisms for fractoluminescence in scintillators. First of all, the generation and separation of the two crack surfaces causes a large charge separation. Over the small gap sizes, it is possible that there is arcing through the atmosphere, generating flashes of light. It is also possible that the UV arcing is interacting with the scintillator, which in turn fluoresces. Charged particles released from the fracture may also contribute to the scintillation light produced. The interaction of the UV arcing and other charged particles may be another possible mechanism of the light produced during fractoluminescence.

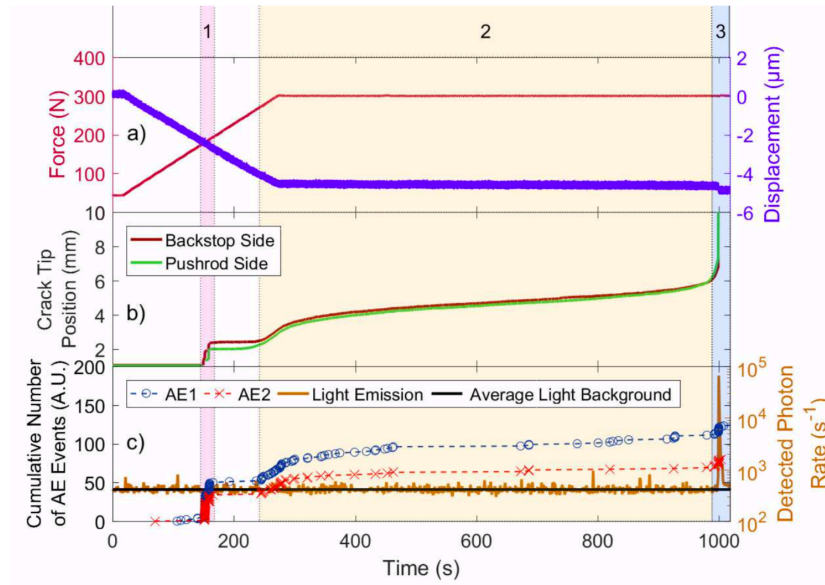


FIGURE 3.7 – DCDC crack growth in a BGO sample under a normal atmosphere. Notable regimes are 1) reopening of previously initiated crack, 2) slow crack growth, 3) fast fracture.

3.3.2.2 Controlling for humidity

In all cases, light emissions have been observed but they strongly depend on the environment and on the loading conditions. In addition to ambient environment, fracture experiments have been performed under vacuum, dry and humid nitrogen atmospheres to evaluate their impact.

Tests under vacuum showed that the ratio of energy released converted into light is lower during slow fracture compared to fast fracture. The difference between slow and fast cracks seems to indicate that other mechanisms may produce light during the fast rupture regime compared to the subcritical one. This difference could also suggest that some charge neutralization processes, dominant during the subcritical regime, do not have time to take place for fast crack, so that the electrical field stays high. A closer look at the amount of light energy emitted during slow crack in vacuum, shows that the light energy is proportional to the created surfaces. However, the scaling prefactor between the light energy and the created surfaces strongly fluctuates between specimens. This prefactor may depend on the acceleration and the pressure of the surrounding gases (differences are observed between experiments at ambient pressure and in vacuum). Fracture could create charged surfaces inducing a gap independent electric field. The surface charge density can either be canceled by migration of the charges through the crystal or the water meniscus, or by the bombardment of the accelerated charges produced by the crack propagation.

Otherwise, it was observed that the forces required to fracture the crystals is

dependent on the water vapor pressure (figure 3.8(a)). Fractures occurring in dry nitrogen and vacuum atmospheres (low vapor pressure) required approximately 50% greater applied force than fractures occurring in ambient or humid nitrogen conditions. It is consistent with the fact that water vapor in the atmosphere has been shown to cause microcracks degrading materials. The atmosphere seems to have also an effect on the energy dissipated in fracture, and by extension, the fracture emissions. Fractures in ambient conditions dissipated less energy than fractures in vacuum and dry nitrogen atmospheres. This result mirrors the results we obtained examining the force required to fracture the crystals, suggesting a similar mechanism.

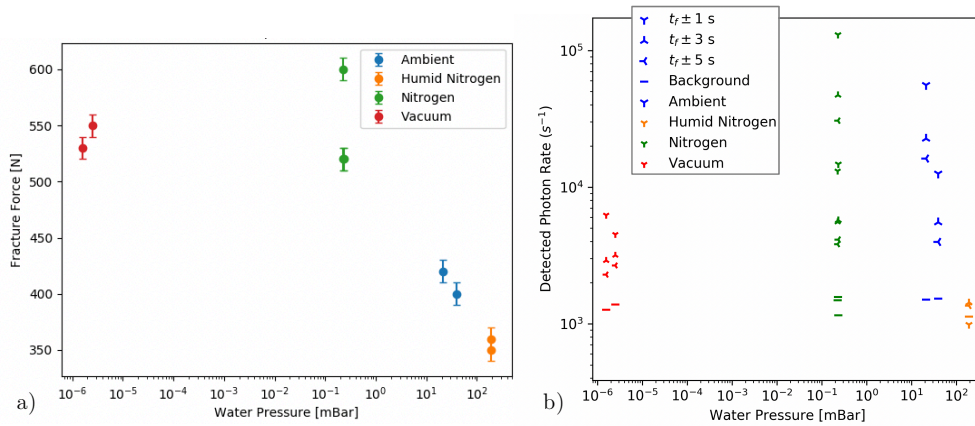


FIGURE 3.8 – Changing the water vapor pressure by controlling humidity and the vessel pressure impact (a) the force needed to initiate fast fracture and (b) the detected photon rate during fast fracture. (The shapes indicates the time windows of integration around fast fracture, and the color indicates the atmosphere in which the experiment was performed.)

Humidity was also observed to affect the light emitted from the sample during fast fracture (figure 3.8(b)). We observe very little emission of light at low and high water vapor pressure levels, and higher detected photon rates when the water vapor pressure is at an intermediate level. This result is consistent with Paschen's law if we hypothesize that light production is in part due to arcing across the new crack surfaces. Paschen's Law states that the voltage required to generate arcing has a minimum value for a given vapor pressure and gap distance, below and above which the breakdown voltage increases.

To conclude, this exploratory study opens up a number of questions. The quantification of the amount of elastic energy converted into light is really just a first attempt and we should take advantage of the excellent resolution of the light channel to precisely calibrate the energy scale and this, also for AE : to quantify the fraction of energy going into acoustic phonons and calibrate the energy scale of acoustic phonons.

Besides, is there effectively a proportional factor between the emitted light po-

wer, acoustic energy and the crack velocity? Our preliminary results incline to prove so but further experiments with different crack velocities are necessary to validate this correlation. Also, a further analysis of the photon clusters could help understand their origin (crack growth, avalanche of dislocations, etc). Finally, additional measurements of crack profiles, amount of charges on the crack surfaces in different atmospheres, radiofrequency emissions (signature of electrical breakdowns) and measurement of temperature at the crack tip could give valuable information on the light emission mechanisms.

Outcome and research project

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All materials are different and what I have learned from one material is not necessarily valid for another. We all wish to find universal laws but they play at hide and seek.

I decided to go with the course of material damage along the sections of this manuscript. Of course, that doesn't apply to all materials.

Plastic deformation is observed in most solid materials, one can verify that the material undergoes a non-reversible change of shape in response to applied force for instance, but the physical mechanisms behind can vary widely from one material to another. I have of course looked at a very small part of plasticity, and I focused on some crystalline materials only (in the Plasticity chapter, mainly on dislocation-mediated plastic deformation). Even when I studied polymers, they were semi-crystalline polymers! One can obviously say "of course, that's just because it's the crystalline areas talking!". Well, one should not forget that we suggested that deformation of cavitation can be associated with the release of a relative high energy.

Either way, it is not for their screaming strength that I chose to study this or that material for such and such research, but I must admit that I am very happy that I managed to decipher a little bit of their language!

I guess I should say the same for damage. I concentrated on a tiny part of the field and on few materials. We all see what damage can do to a specimen, to a material but the physical sources can be quite different for different materials and conditions. It applies equally to the origin of cracks in materials. For instance, I have presented incremental, repetitive crack propagation in [fcc](#) metallic materials;

well, such growth is also observed in polymers or metallic glasses, but the nature of stress concentrators for crack nucleation is different.

Anyhow, during these past 10 years, I have attempted to gain an understanding of the phenomena of fractures, damage, plasticity; especially in the case of fatigue (but remember, historically, the notion of damage was introduced during the study of material fatigue!); always trying to apprehend the physical mechanisms at the source. We are far from having it all figured out!

At the end of the day, I can say shamelessly that the AE technique offers advanced possibilities to comprehend the different phenomena in a non-destructive way and therefore enables to anticipate the rupture of materials and structures in an early way. It is therefore a logical continuation of the quest to understand phenomena to wish to use one's knowledge to predict behaviors, particularly macroscopic ones, in the light of knowledge on a microscopic or mesoscopic scale. And it is only natural that some of the research projects I will be carrying out in the coming years are related to the prediction of fatigue failure, in light of the discoveries we have been able to achieve. These new projects combine the AE technique with AI.

Some other research directions are also more linked to further fundamental questions. A never ending story!

4.1 Early warnings of crack propagation

4.1.1 Proof of concept

I have reported in section 3.2.1 our discovery of AE specific to fatigue crack growth : acoustic multiplets characterized by nearly identical waveforms, repeatedly triggered over many successive loading cycles at the same stress level and originating from a single localized source. They mark the slow, incremental propagation of a fatigue crack at each cycle, or the rubbing along its faces. Being specific to fatigue cracking, they can be used as early warnings of crack propagation, which will ultimately lead to structural failure.

Yet, the tests we performed are preliminary and all the analyses have been carried out post mortem, not *in-operando*, and in optimized (low SNR) environments. Therefore, a broader project has emerged to extend this seminal work to *in-operando* detection and monitoring under industrial environments with the ambition to certify the physical origin of the AE-induced underlying mechanism.

Besides, isolating the AE bursts associated with single crack propagation events during controlled and in-situ monitored lab-experiments would be a good way for analyzing AE in an ideal situation. As it happens, it has been reported in the literature that Poly(methyl methacrylate) (PMMA) may experience intermittent failure under some well-defined loading [Guerra *et al.* 2012]. And, actually, these experiments allow for isolating a single crack propagation event. Such results have been reproduced at GeM laboratory using Tapered Double Cantilever

Beam (TDCB) specimens. Varying the macroscopic loading rate from 0.5 mm/s (figure 4.2(a)) to 5 mm/s (figures 4.1, 4.2(b)), there is a clear transition from a continuous cracking at low velocity ($\sim 50\text{mm/s}$) to intermittent dynamic crack propagation at higher velocity ($\sim 100\text{mm/s}$). Further, a closer inspection of the crack faces reveals that, even in the "static" phase, small burst of $\sim 50\mu\text{m}$ crack increments are observed, which makes possible isolating crack propagation events operating at different space and time-scales.

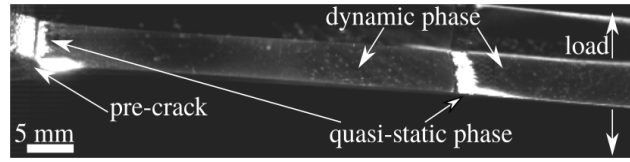


FIGURE 4.1 – Quasi-static phases vs. dynamic crack propagation in PMMA.

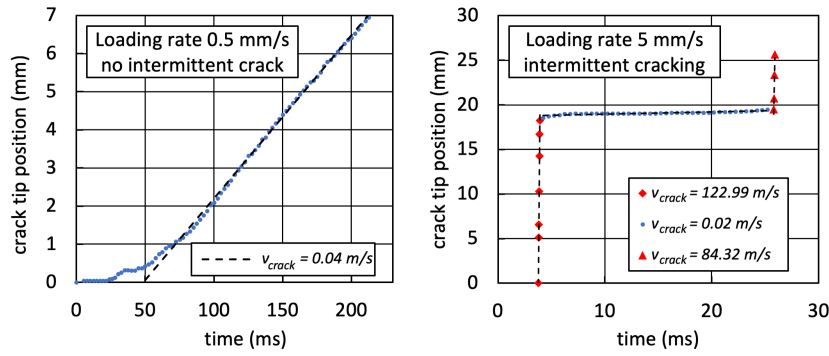


FIGURE 4.2 – From high-speed images (40 kfps), analysis of crack length evolution for two loading rate (0.5mm/s and 5 mm/s).

AE-based crack growth models and some attempts for quantification from in situ AE monitoring have actually been proposed to improve structural integrity assessment. For instance, Rabiei and Modarres proposed [Rabiei & Modarres 2013] (i) an AE-based instantaneous damage severity index, and (i) an AE-based estimate of the crack size distribution at a given point in time, assuming a known initial crack size distribution.

Nevertheless, the quantitative analysis of crack tip related phenomena using only AE has not yet been finalized. There is thus a need for AE analysis to be improved to better extract and quantify the signal features associated with crack propagation. We also suggest that isolating the typical AE associated with crack advance experimentally would benefit for a better understanding of AE signal and their relation to mechanisms. Our hypothesis is that if intermittent crack propagation (as reported in [Guerra *et al.* 2012]) can be reproduced, crack advance related AE could be analyzed independently from other mechanisms. If the wave formations from the crack tip to the AE sensor can be tracked by any experimental

mean, then a deeper understanding of the AE content can be achieved. Modeling these successive events numerically would allow proposing AE source models, useful for a better analysis of AE signals and their intrinsic relation with mechanical events.

4.1.2 From physical modeling to industrial applications

This seminal study naturally leads us towards a project on early detection of fatigue and intermittent failure using full-field approved AE multiplet, that we want to tackle in the near future. And actually, this project, which we name "e-WARNINGS", has been selected for funding, following the AAPG2019 of the ANR. I am the coordinator of this project which includes five partners : fours laboratories (MatéIS, GeM Nantes, ISTerre UGA-Grenoble and LaMCoS INSA Lyon) and one industrial partner (Mistras Group, SAS). We identified the following objectives :

- (i) **to understand the origin of these multiplets, identify and model the physical processes involved, for different materials, under different loading conditions.**

We will discriminate, characterize and identify the different source mechanisms appearing during fatigue crack propagation (crack growth, crack closure, fretting), using pattern recognition analysis. Indeed, being able to distinguish crack propagation events from friction between the crack faces is a key issue. We will thus perform experiments in which crack propagation can easily be isolated from other crack related events. *In fine*, we will provide an experimental AE database to test and optimize the detection algorithms that will be developed to detect *in-operando* the propagation of cracks.

- (ii) **to develop new AE source models by analyzing the detected signals in connection with mechanical events. Ultra-high speed DIC and advanced numerical fracture simulations will be used to finally improve the AE signal analysis.**

Associated with full displacement field measurements by DIC, we should be able to follow and analyze, from the crack tip to the AE sensor, the wave formation arising from a single crack propagation event. Acquiring high-resolution experimental data in space and time will allow to develop and validate an AE source model using numerical simulations. One of the challenges is to perform the "deconvolution" of the acquired signal to retrieve the origin of the AE, i.e. the source. Once the numerical simulations reproducing the wave propagation observed experimentally are validated, it will be possible to compute numerically the response of the AE sensor from any crack tip related event. Then, the crack tip events being parameterized, a source model will be elaborated, allowing for a robust estimate of the crack tip event from the AE signals. The model will be used to analyze fatigue crack propagation experiments and propose a relationship between crack advance and AE signals characteristics.

Furthermore, the numerical simulations will be used to improve the resolution of AE in terms of crack localization or crack growth.

and

- (iii) **to propose a new, reliable non-destructive on-line monitoring of the onset of fatigue cracking during mechanical tests or within structures in-service.**

We want to develop machine learning algorithms for automatic identification of these acoustic multiplets allowing an early detection of fatigue cracking during loading, and thus, ultimately, triggering a preventive stop. We will perform though experiments an evaluation of the computational efficiency of the algorithms in an operational mode. Ultimately, these detection algorithms will be extended to greater structures/equipment and noisy environments, towards an industrial application. Hence, the proposed method will detect the unambiguous presence of stage II fatigue cracks providing early warnings of failure, and a critical criterion will be used to make a preventive stop of the equipment and anticipate serious damage. Moreover, if the damage concept tolerance [Jones 2014] is to be used to operate the structure with active cracks, the method will have to be supplemented by an independent estimate of the incremental advance of such cracks, which will be obtained thanks to the source models developed in the project.

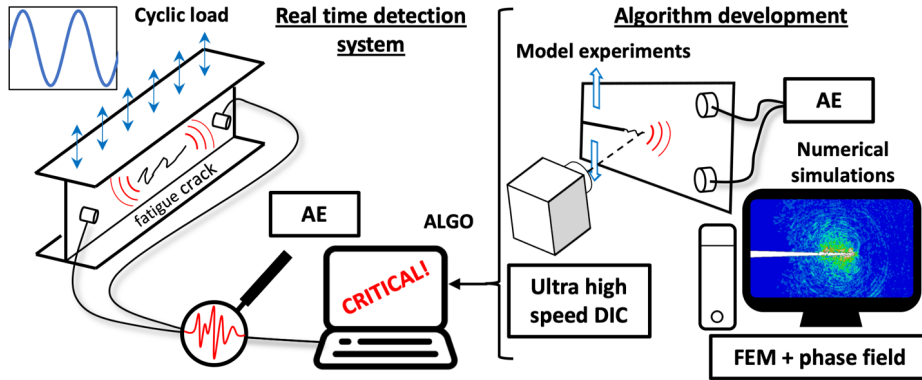


FIGURE 4.3 – Global picture. Left hand side : industrial application by measuring the AE multiplets in a fatigue crack propagation scenario. Right hand side : development of the real-time detection algorithm.

Overall, the global picture of this project can be illustrated as in figure 4.3. The specific detection of the acoustic multiplets in materials, together with state-of-the-art technologies enabling full-field measurements as well as the development of new AE source models, will allow us to obtain a new way to detect, monitor and measure fatigue crack extension (as well as surface asperity and average slip) *in-situ* and *in-operando* with a non-destructive technique. We will also be able to

experimentally validate, through novel ultra-high speed full field measurements, the source models on which the interpretation of the AE signals are based. The new monitoring method and the dedicated detection/monitoring algorithm we propose will enable the identification of cracks and underlying failure risks with unprecedented sensitivity and specificity, well before any unstable propagation occurs.

The methodology developed in this project combining AE and AI could be used in other cases for pattern recognition and fundamental comprehension of phenomena at the source of the AE signals.

4.2 Fatigue and AE towards lifetime prediction

Given that I have now worked on a few projects related to fatigue of metallic materials, why stop here? I wish to continue these studies to move towards lifetime prediction. And a new project, focused on fatigue on corroded aluminum samples, monitored by AE, towards lifetime prediction, has started last November. It is part of a European project, entitled U-CROSS : "Early detection and progress monitoring and prediction of corrosion in aeronautic Al alloys through calibrated Ultrasonic CorROsion Sensors application". This larger project is an Horizon 2020 / Clean Sky project.

The need of reducing aircraft weight, in order to decrease fuel consumption, pushes the aerospace industry to incorporate light materials, such as aluminum alloys, in many parts of the aircraft such as fuselage, engines and systems. One of the main drawbacks of Al alloys is that they are sensitive to microstructural localized corrosion, generating corrosion events such as pits. Corrosion is a dynamic process in which propagation rate remains difficult to predict and varies depending on the type of corrosion occurring (pitting, intergranular, exfoliation, filiform, corrosion-fatigue and SCC (Stress Corrosion Cracking)). Currently, the presence of corrosion damages may be detected by means of NDT. However, corrosion initiation and mechanisms behind the defects cannot be distinguished by currently used methods. As a consequence, the aircraft industry needs to develop methods in order to detect corrosion, mechanical damage and predict the durability of aluminum alloys. In this context, the AE technique is appropriate. A common approach in such a case is to identify the nature and the origin of degradation or damage phenomena from the analysis of AE signals by classifying the signals into classes using pattern recognition technique (k-means, neural networks, machine learning,...).

One of the main objectives of U-CROSS is to develop and validate the application of AE, for corrosion and mechanical damage monitoring. An important point is to be able to detect in real time the early stages of localized corrosion and to monitor the progress of damage with time. The contribution to U-CROSS at Mateis is conducted through a PhD study that started this November 2019 with PhD student Théophile Vié, whom I supervise with Nathalie Godin and Bernard Normand (Mateis). The

objective is to predict, via AE, the lifetime of corroded samples under fatigue loading. Cyclic fatigue tests will be performed on aluminum alloys with different coatings and different corrosion initiation methods. The main steps of the project are :

- (i) to experimentally investigate the effect of corrosion on the lifetime of Al alloys samples during fatigue tests.
- (ii) to analyze the waveform in order to identify the AE signature of each damage mechanism - comparison between localized corrosion and fatigue corrosion.
- (iii) to build a new model to estimate the remaining useful lifetime [RUL](#) of the corroded samples or to predict the number of fatigue cycles before corrosion is initiated.

Those two projects will also allow us to move towards broader studies in the field of Structural Health Monitoring ([SHM](#)), an area that the laboratory wishes to develop. [SHM](#) aims to give, at every moment during the life of a structure, a diagnosis of the state of the constituent materials, of the different parts, and of the full assembly of these parts constituting the structure as a whole. [SHM](#) is not only a new and improved way to make a non-destructive evaluation as it involves the integration of systems into a metallic structure by means of additive manufacturing [[Strantza et al. 2015](#)], of sensors, possibly smart materials, data transmission, and processing ability inside the structure.

4.3 Diverse future perspectives

Each project opens up new questions and possible paths for future developments and new projects. I will present some of them in the following section.

4.3.1 In the context of dislocation dynamics

Once these two more applied projects presented, I have to go back to my basics and pursue more fundamental projects.

The strength of AE in the analysis of the collective phenomena in plasticity is to provide the opportunity to scan in 3D and in situ the dislocation dynamics and therefore to indirectly track the structuration in situ. Moreover, the possibility to perform microstructural characterizations along the cycles was a real asset. Nevertheless, the various steps of mounting and dismounting, and the successive polishing are steps prone to damage the samples. And, in our case, it has not been possible to follow the evolution of the dislocation structure on the exact same area. A solution would therefore be to implement a fully in-situ set-up in a [SEM](#), with specimens being deformed directly in the [SEM](#), the strain being monitored using a suitable DIC method and the dislocation dynamics monitored by AE. Associated with in-situ stages, it would allow direct observation of the dislocation structure evolution, under a limited number of cycles at imposed stress. We must be vigilant though that the use of an in-situ stage is not currently compatible with [R-ECCI](#), since the

sample has to be rotated. R-ECCI should therefore be developed on the basis of beam rotation rather than specimen rotation.

An AE/tomography combination could also be considered, even if it seems complicated, since the involved processes are fast and small in size. The dislocation dynamics would thus have to be monitored under synchrotron radiation.

Otherwise, the influence of crystallographic orientation could be further investigated, by studying oriented single crystals for single or double slip, in addition to multiple slip, in order to examine how junctions between dislocations influence collective dynamics (are strong junctions obstacles to avalanches?). We may proceed with strain-imposed tests, in which the gradual transformation of the dislocation structure over the cycles can be monitored by AE, and may provide further information on the role of dislocation avalanches.

In addition, we should study a wider range of crystallographic structures (from bcc, other fcc to hcp) and compare the extreme behaviors on both side of the isotropy, scaling from a plastically isotropic material to a model hexagonal material with extreme anisotropy (ice). This study could be done in collaboration with Maurine Montagnat (Centre d'Etude de la Neige) and, cryo-EBSD could be used to characterize dislocation substructures of ice single crystals by means of a Nye tensor type of approach (relating local misorientation to dislocation densities). This would provide an overall picture of the influence of dislocation interactions, of the role of dislocation structures and of the loading mode on dislocation dynamics and on the origin of dislocation avalanches.

Furthermore, we could benefit from using sensors with better sensitivity and wider bandwidth (reaching out the 20 MHz limit), specifically for the analysis of dislocation movements within a fcc-type dislocation substructure. Even more, it would be interesting to design dedicated sensors with the best possible sensitivity-bandwidth ratio, possibly in collaboration with LVA (Laboratoire Vibrations Acoustique). And frequency analysis would also enhance the quality of the analyses.

4.3.2 In the context of fractoluminescence

Meanwhile, an important question that lingers in my head is related to the calibration of an energy scale. The projects mentioned above will certainly provide some answers to this query. Moreover, as stated in our study on fractoluminescence, light emission allows to quantify the amount of elastic energy converted into light and offers a possible calibrated energy scale. Nevertheless, a fuller understanding of the energy budget requires knowledge of the energy transformed into phonons, or heat. Studies quantifying the fraction of energy going into acoustic phonons or calibrating the energy scale of acoustic phonons are very patchy.

In addition to acoustic phonons measurement with AE, at room-temperature, a harder-to-implement technique of cryogenic calorimetry would allow measurement

of all the phonons and would therefore provide more insight into the energy budget. Actually, we have designed at Queen's university, during Alexis's PhD thesis, a cryostat with the company ColdEdge Technologies. This system, just begging to be used, will allow to break DCDC specimen at different temperatures down to $6^\circ K$. And this will also be a step toward a better fundamental understanding of fracture mechanisms. And, for the record, the setups we designed are not restricted to scintillators, but could be used for other materials such as PMMA and glass.

Otherwise, the discussions on the light emission mechanisms are still relevant, therefore we have to pursue the test under different environments and for different crack velocities to get a clearer picture. For instance, the measurement of the amount of charge on the crack surfaces in different atmospheres, and a direct measurement of the crack profile could give valuable information. It could also help confirm the difference between slow and fast crack growth. Moreover, the measurement of the radiofrequency emissions, used as the signature of electrical breakdowns, would also allow to test the hypothesis of an electrical breakdown between the crack surfaces. Additionally, the measurement of the temperature at the crack tip would demonstrate whether or not photons can be emitted by thermoluminescence (or black-body radiation) and it would help the quantification of energy release.

Also, I would like to collaborate with Tanguy Rouxel (Institut de Physique de Rennes), whose work has included the development of mechanoluminescent vitreous materials, emitting light under mechanical stress [Dubernet *et al.* 2015], for a better understanding of the mechanoluminescence mechanisms.

4.3.3 In the context of additive manufacturing

Additive manufacturing (AM) techniques have been on the upswing for a few years now, allowing the realization of on-demand geometries that could not be manufactured with conventional techniques, also developed at Mateis (several PhD thesis supervised by Jean-Yves Buffière [Persenot *et al.* 2019] and Xavier Boulnat [Villaret *et al.* 2019]). In particular, many studies nowadays make use of samples produced by AM, such as for the AE monitoring of crack growth behavior of additively manufactured titanium components by [Strantza *et al.* 2017].

However, these techniques still present some limitations such as the need for support structures in specific geometrical cases (bridges) or the apparition of unwanted cracks due to thermic residual stresses. It would therefore be of great interest to understand the damage mechanisms occurring during the additive manufacturing process [Wu *et al.* 2019] in complex geometries and more specifically the crack initiation and propagation due to residual stresses. Thus, an idea could be to model and simulate cracking in these structures and identify microscopic damage behaviors, combining AE, in-situ testing and advanced multiscale numerical simulations.

Another promising approach would be to control the damage of architectural components, in connection with Xavier Boulnat's project on robocasting of archi-

tectural structures. One of the benefits of AM is to aim at structures that "tolerate" damage, by modifying the architecture of the components : crack deviation, interface creation, etc. If the contribution of AE is well explored on bulk materials, it is currently limited on architectural materials (lattice structures, porosity gradient structures, etc.). The contribution of AE, combined with tomography/Digital Volume Correlation (DVC) could provide valuable information for damage localization on complex structures.

4.3.4 A little further

Collaborating with simulation researchers could be beneficial for the understanding of physical phenomena at the source. As mentioned in section 4.1, the idea of combining the EA technique with numerical simulation on model experiments is to find the origin of AE, i.e. the source. It would also be beneficial to collaborate with researchers developing models for plastic flows in crystal as for instance O.U Salman and L. Truskinovsky that showed that nonlinear continuum elasticity can be effective [Baggio *et al.* 2019] or researchers developing dislocation dynamics simulations as B. Devincre and S. Kubin [Devincre *et al.* 2008, Kubin & Sauzay 2016] or J. Amodeao at Mateis [AMO 2018] or D. Rodney and M. Fievel [Nogaret *et al.* 2008], all demonstrating the interest of mesoscale simulations for addressing in a predictive manner the complex problems of dislocation dynamics (e.g. dislocation patterning and strain hardening in crystals). Thus, the simulation of specific events, crack propagation, dislocation emission from the crack-tip, dislocation interactions etc. coupled with AE analysis could give us valuable information to understand the origin of the phenomena. Eventually, these simulations could provide an AI algorithm with the basic elements to interpret the genuine signal, for example from an ideal single crystal experiment.

Afterword

While the research I've been conducting is very experimental, it is nonetheless mostly fundamental. This is actually the prime reason I chose to turn to research. I don't mean to disappoint you, but no, I was not necessarily eager to torture the materials on a daily basis at work! On the other hand, I clearly longed to understand what was going on within their matter to a scale invisible to our eyes. Already, in the days of my engineering school, I was looking for the senses that could help me understand the very small movement in matter. Since then, I have developed a taste for science, attempting to have a keen nose, and I have always attached great importance to observation and experimentation by constantly looking, touching whenever possible, and I eventually ended up listening to materials, always!

The beauty of science is serendipity, such as improving a technique by discovering a correlation by chance. Somewhat like developing a predictive algorithm through the unexpected discovery of acoustic multiplets¹.

Among the (too) many questions we face in research, we may ask ourselves, why are we doing research? Historically, this has been possible because there was no longer the need for subsistence, no longer the need to go hunting or gather food. The nobles in particular were the ones who had the luxury of doing research. Today, society questions the purpose of funding research. Is this still a luxury? All the more since part of this very same society is suspicious about the production of the said research; controversies about Covid-19 cure, questions about genetically modified organisms (GMO), etc. have been raised. Science, more than ever, must be flawless when it is addressed to a wide audience.

Certainly, this is a healthy question, but to what extent should it be applied? Especially when public policies for science (and for the health care system, as seen during the Covid-19 crisis, which took place while I was writing this HDR) use evaluation and management systems from the private sector.

I, for one, am wondering about my research : what is my driving force? The understanding of fundamental physical phenomena²? The need to feel useful, to contribute to people's living and safety? I would definitely say both!

In the end, I strongly focused on the quest for precursors, be it to fracture of materials, fatigue or plasticity. I am trying to understand how these major events are initiated at small scale and provide us with valuable insights regarding the underlying phenomena. It is only natural that by investigating these precursors, I will leverage this knowledge to propose innovative methods for the early prediction of catastrophic events, such as the structures collapse.

1. For the record, we initially thought that these were artifacts in the final stages of fatigue tests!

2. One can say to unlock the secrets of matter, but this is too cliché!

Curriculum vitae

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5.1 Personal details

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5.2 Professional experience

Research Assistant - PhD thesis in Materials Science and Engineering 2002-2005

Groupe d'Études de Métallurgie Physique et de Physique des Matériaux (GEMPPM, now MATEIS), INSA Lyon and Physics' Laboratory, Ecole Normale Supérieure of Lyon.

Research directors : G. Vigier (INSA Lyon) and S. Ciliberto (ENS Lyon).

«Dynamics of fracture precursors in heterogeneous materials : application to vitreous polymer foams»

- Description of materials fracture using acoustic emission (AE).
- Statistical analyses of fracture precursors.
- Synthesis of polyurethane foams and porous epoxy resins.
- Microstructural characterization by scanning electron microscopy and X-ray micro tomography.

- Mechanical properties in linear and non-linear domains.

2006

Postdoctoral Research Fellow -Assistant researcher

Department of Mechanical Engineering, MIT -Massachusetts Institute of Technology- Cambridge (U.S.A), under the supervision of M.C Boyce and R.E. Cohen, in collaboration with Dupont USA.

« *High Rate Behavior of Polymeric Nanocomposites : Microstructure, Mechanisms, and Mechanical Behavior* »

- Design and development of polymers and polymer nanocomposites with tailored microstructure for high rate mechanical performance.
- Understanding of the mechanisms of deformation and energy absorption : study of large strain deformation behaviors over nearly eight orders of magnitude in strain rate, from $10^{-4}/s$ to almost $10^4/s$.

01/2007-present

Associate professor (section 28) MATEIS laboratory : Materials : EngIneering and Science ; INSA Lyon, UMR 5510,

01/2007 - 09/2012 ENDV team (Evaluation Non Destructive et Durée de Vie)

09/2012 - present METAL team (METals and ALloys)

- ↪ Development of acoustic emission (AE) data analysis to study the damage of heterogeneous materials up to global rupture and the dynamics of fracture precursors (power law statistics)
 - Interface between mechanics and physics : innovative description of materials fracture as a critical phenomenon. Influence of material properties on scaling laws using specifically developed materials.
 - Source mechanisms identification : relationship between microstructure/ AE and macroscopic properties (simultaneous in situ tensile tests with AE and X-ray tomography).
- ↪ Study of cyclic fatigue of metallic materials via AE signal analysis to apprehend the evolution of criticality as an indicator of fatigue.
 - Link between the evolution in acoustic signal structure and the changes in the collective behavior of defects.
 - Study of AE multiplets (signature of crack growth/ friction)
- ↪ Characterization of plasticity and damage in semi-crystalline polymers with AE and ultrasonic active techniques.

2013-2015

Visiting researcher (January-August 2013 and September 2014-March 2015)

Department of Physics, Queen's University, Canada with P. Di Stefano

Study of fractoluminescence, by coupling scintillation particle detection techniques to AE : correlation of acoustic and light emission during fracture of materials.

5.3 Teaching activities

My teaching activities take place 100% at INSA Lyon undergraduate department (Formation Initiale aux Métiers d'Ingénieurs, FIMI), in the SCAN (Science and English) and in the classical studies (PCC), except for the supervision of final year projects (FYP) in departments (SGM, GM).

SCANFirst (Science and English, 1st year), L1 level, 2007-2018 :

- Responsible for the Electromagnetism course module and head of physics SCAN1st for the second semester In charge of the electromagnetism lectures, tutorials and practical for SCANfirst groups (12h lecture, 41.5h tutorial + practical). All lectures, tutorials and practical are taught in English. My investment in this new section opened in 2007 when I arrived, and transformed and enlarged in 2013, was rendered from a pedagogical point of view by the creation of a course entirely in English, including an electronic support, the development of a tutorial booklet in English (60 pages), the preparation of a lecture booklet and the translation of some educational documents. I have also implemented teaching methods to integrate through specific educational facilities a student population from various backgrounds in terms of culture, language and scientific achievements.
- Electrokinetics tutorial+ practical (45.5h)

SCANSecond (Science and English, 2nd year), L2 level, 2007-2013 :

- Electricity / Magnetism practical (28h).

Classical section (PCC, 1st year), L1 level, 2013-present : - Electrokinetics (45.5h TD + TP), Mechanics (48h TD + TP), Electromagnetism (41.5h TD +TP)
 - 2013 : participation in the creation of new tutorial/practical in electricity for the first year INSA - In charge of the « Filters » practical during the first semester, since 2017. - From 2018 : head of Mechanics' practical for the second semester.

Continuing Education (Centre d'Actualisation Scientifique et Technique, CAST), Insavalor :

- Acoustic Emission tutorial/practical (4h) (intervention on an ad-hoc basis depending on the year)

Departments Science and Engineering of Materials (SGM) and Mechanical Engineering (GM) : Supervision of 15 final year projects (FYP, M2 level), see below.

5.4 Doctoral and scientific supervision

I had the opportunity to work with 5 PhD students (plus one who started in November 2019) and a number of master's students and final year engineering students. I list below the students with whom I have collaborated.

Thesis

- ↪ Florent Philip : (2008-11) Modeling and lifetime monitoring of filament-wound composite structures in a wet environment. Application to hybrid tubes.. Funding IFP (CIFRE thesis). Joint supervision 50% with Nathalie Godin (MATEIS-INSA Lyon), (Dir. Joël Courbon, MATEIS-INSA Lyon).
- ↪ Wafa Ben-Rhouma : (2009-13) Acoustic emission study of plasticity and damage of aluminum in low-cycle fatigue. Funding ANR Blanc. Joint supervision 50% with Jérôme Weiss (LGGE-Grenoble).
- ↪ Nicolas Casiez : (2011-14) Characterization of the initiation of plastic deformation and damage of semi-crystalline polymers by ultrasonic methods. Funding ANR JCJC. Joint supervision 50% with Olivier Lame (MATEIS-INSA Lyon).
- ↪ Alexis Tantot : (2012- 2015). Acoustic and light emissions during fracture of inorganic scintillators. Funding : France Canada Research Fund (FCRF). Joint supervision 33% with Philippe Di Stefano 34% (Queen's University, Canada) and Loïc Vanel 33% (LPMCN, Lyon University).
- ↪ Gabriel L'Hôte : (2016-2019) Combined study of dislocation dynamics under cyclic loading of pure monocrystalline copper : acoustic emission and microstructural characterization. Funding : doctoral grant with doctoral school Materials. Joint supervision 50% with Sophie Cazotte (MATEIS-INSA Lyon); (Dir. Joël Courbon, MATEIS-INSA Lyon).
- ↪ Théophile Vié : (2019- ...) Fatigue and Acoustic Emission on corroded aluminum samples towards lifetime prediction. Funding European Project (CleanSky 2 : U-Cross). Joint supervision 50% with Nathalie Godin (MATEIS-INSA Lyon)

Master M2 Research internships

- Kevin deHaan : (Septembre 2014- Avril 2015) Design of a Breakable MEMS Beam. Co-encadrement avec 50 % Philippe Di Stefano, Queen's university, Canada.
- Gwladys Lefèvre : (Mars-Septembre 2016) Émissions lumineuses et acoustiques lors de la rupture de scintillateurs inorganiques. Co-encadrement 50% avec Philippe Di Stefano, Queen's university, Canada.
- Gabriel L'Hôte : (Mars-Septembre 2016) Étude de la dynamique des dislocations de monocristaux de cuivre sous chargement oligocyclique par émission acoustique. 100%
- Robert Squibb : (Septembre 2016- Janvier 2018) Quantum Mechanical Effects in Sub Critical Fracture. Co-encadrement 30% avec Philippe Di Stefano, Queen's university, Canada (40%) et Loïc Vanel INL, France (30%).
- Thabet Marwa : (Septembre 2017- Avril 2019) Investigation of fractures as a background in rare-event searches. Co-encadrement 35% avec Philippe Di Stefano, Queen's university, Canada 35% et Loïc Vanel INL, France (30%).

- Hamza Charra : (Mars 2019- Septembre 2019) Physique de la rupture de câbles textiles technologiques : détermination et modélisation de la cinétique de rupture en utilisant la technique d'Emission Acoustique. Collaboration Michelin. 100%

Final year projects, M2 level

- Benoit Maitrinal et Luis Alberto Lopez Palacios : (2007) Etude de l'endommagement d'un matériau composite à matrice polymère (Couplage de techniques tomographie aux rayons X et Emission Acoustique en traction). Collaboration avec Schneider Electric.

- Emile Maras : (2007-2008) Etude des précurseurs à la rupture et prévision de la durée de vie des matériaux hétérogènes à partir de modèles statistiques.

- Jérémie Steyaert : (2008-2009) A l'écoute des polymères

- Raphael Michel : (2009-2010) Etude de l'initiation de la plasticité dans les polymères semi-cristallins par Emission Acoustique.

- Romain Pellet : (2010-2011) Détection de la déformation plastique de polymères semi-cristallins par Emission acoustique.

- Romain Ducros : (2011) Déconvolution de la signature de capteurs d'Emission Acoustique.

- Mathilde Gatepin : (2011-2012) Etude des précurseurs de la fatigue de l'acier 304L et du cuivre pur par émission acoustique.

- Mireia Alsina Ciladecans : (Sept 2013- Avril 2014) Recherche de multiplets en Émission Acoustique lors d'essais de fatigue d'aluminium pur.

- Rémi Chateau : (Février-Juin 2015) De la radio à l'audiovisuel : Instrumentation couplée d'émission acoustique et de corrélation d'images pour essais de fatigue.

-Théo Foschia : (Février-Juin 2016) Essais de fatigue cyclique sur acier 15-5PH pour suivi de fissures : Emission Acoustique et corrélation d'images. 100%

-Carolina Esparza-Contro : (Sept 2016- Mars 2017) Analyse couplée de données d'Emission Acoustique et de corrélation d'images pour le suivi de l'avancée de fissures de fatigue sur acier 15-5PH. 100%

-Emilie Robert : (Fev 2017- Juin 2017) Traitement de données issues de différents capteurs pour le suivi d'un procédé de grenaillage. Collaboration CREE Saint Gobain. Co-encadrement 50% avec Valérie Kaftandjian (IVA).

-Gurvan Hamon : (Sept 2017- Mars 2018) Etude de cinétique de rupture de câbles textiles hybrides par la méthode d'Emission Acoustique. Collaboration Michelin. 100%

-Demeng Fan : (Fev 2018- Juin 2018) Contrôle Qualité de joints PTFE OmniSeal Saint-Gobain. Collaboration CREE Saint Gobain. 100%

-Joel Zapatero Hernandez : (Sept 2018- Mars 2019) Étude de cinétique de rupture des renforts textiles des pneumatiques par la méthode d'Émission acoustique. Collaboration Michelin. 100%

Participations to theses committees

- Jury de thèse (examineur) Ammar Ould Amer, ENSTA ParisTech, 11 Décembre 2014 "Cinétiques d'endommagement à différentes échelles d'un acier inoxydable austénitique en fatigue à amplitude constante et variable"
- Jury de thèse (rapporteur - dérogation pour rapporteur de thèse non HdR validée par le Directeur de l'Ecole Doctorale "Sciences de la Matière" de Rennes, ED254) Marion Dubernet, Département Mécanique et Verres, Institut de Physique de Rennes, 06 Décembre 2016 "Mécanoluminescence de composites particuliers à matrice vitreuse"
- Jury de thèse (examineur) Chi-Cong Vu, Institut des Sciences de la Terre, Grenoble, 16 Octobre 2018 "Statistical size effects on strength : from Rocks to structural materials"
- Jury de thèse (examineur) Theany To, Département Mécanique et Verres, Institut de Physique de Rennes, 27 Février 2019 "Fracture toughness and fracture energy of inorganic and non-metallic glass"

5.5 Scientific responsibilities

Public contracts

ANR project : Coordinator + Responsible INSA :

Projet ANR PRCE (Projet de recherche collaborative / Entreprise) AAPG 2019.

"e-WARNINGS : Early detection of fatigue and intermittent failure using full-field approved Acoustic Emission multiplets"

Collaboration 4 laboratoires + 1 industriel : GeM Nantes, ISTerre Grenoble, LaMCoS INSA Lyon + Mistras Group SAS.

Total budget of 603 041.57 € / 48 mois. (Budget INSA 172 k€) [2 thèses + 2 post-docs + environnement / instrumentation...]

European project CleanSky2/ Horizon 2020 : responsible INSA

Call : H2020-CS2-CFP09-2018-02 (Clean Sky 2 Call for Proposals 09)

U-CROSS : "Early detection and progress monitoring and prediction of corrosion in aeronautic Al alloys through calibrated Ultrasonic CorROsion Sensors application"

En collaboration avec Fundacion CIDETEC (Espagne), UBFC (Université Bourgogne Franche-Comté), Titania Ensayos Proyectos Industriales SL (Espagne) et Mistras Group (France).

INSA : une thèse financée et environnée 2019-2022 + overheads.

Industrial contracts

-Responsable INSA :

-Contrat de collaboration Réf INSAVALOR : Michelin - MATEIS - 2019 - Contrat CO0023585 (25k€) Physique de la rupture de câbles textiles technologiques : détermination et modélisation de la cinétique de rupture en utilisant la technique d'Emission Acoustique Référence : 4090657536

- Contrat de collaboration Réf. Michelin (10k€) : Étude de cinétique de rupture des renforts textiles des pneumatiques par la méthode d'Émission acoustique.
- Contrat de collaboration Réf. Michelin Mateis 2017 (10k€) : Etude de cinétique de rupture de câbles textiles hybrides par la méthode d'Emission Acoustique.
- Contrat d'étude Réf INSAVALOR : SAINT GOBAIN - MATEIS - 2018 - PFE (10k€) : Contrôle Qualité de joints PTFE OmniSeal Saint-Gobain.
- Responsable Mateis :
- Contrat de collaboration Réf INSAVALOR : Saint Gobain -LVA- (10k€). Traitement de données issues de différents capteurs pour le suivi d'un procédé de greffage.

Organisation de conférences

Membre du comité d'organisation des Journées Scientifiques et Techniques de l'Association pour les Matériaux Composites (JST-AMAC) "Essais non destructifs et durée de vie des composites" ayant eu lieu les 27 et 28 Octobre 2011 à l'INSA de Lyon.

Membre d'instance nationale

Comité National de la recherche Scientifique

Élue en section 9 -Ingénierie des matériaux et des structures, mécanique des solides, biomécanique, acoustique -, siège de membre du collège B2, de Septembre 2012 à Septembre 2016.

Travaux de referee

- Évaluations de projets ANR, projets PEPS, CNRS.
- Comités d'évaluation HCERES : évaluation du laboratoire IPR (Institut de Physique de Rennes) 19/01/2016 - 21/01/2016.
- Travaux de referee dans des journaux internationaux :
- Physical Review Letters, Polymer, Smart Materials and Structures, International Journal of Fatigue, Journal of Statistical Mechanics : Theory and Experiment, Measurement Science and Technology, Engineering Research Express.

Activités d'intérêt collectif

- Membre élu du conseil de laboratoire MATEIS depuis septembre 2017.
- Responsable des séminaires du laboratoire MATEIS, INSA-Lyon, 2012-2020
- Commission de spécialiste : Élue (section 28) 2012-2013. Comité de sélection 2016.
- Responsable des systèmes d'Emission Acoustique du Groupe METAL, MATEIS

5.6 List of publications

International refereed journals

1. G. L'Hôte, S. Cazottes, J. Lachambre, M. Montagnat, P. Courtois, J. Weiss,

- & **S. Deschanel** Dislocation dynamics during cyclic loading in copper single crystal. *Materialia*, 2019. 8 : p. 100501.
2. J. Weiss, **S. Deschanel**, W. Ben Rhouma, L. Truskinovsky, Plastic intermittency during cyclic loading : From dislocation patterning to microcrack initiation. *Physical Review Materials*, 2019. 3(2) : p. 023603.
 3. G. L'hôte, C. Lafond, P. Steyer, **S. Deschanel**, T. Douillard, C. Langlois, S. Cazottes Rotational - Electron Channeling Contrast Imaging analysis of dislocation structure in fatigued copper single crystal, *Scripta Materialia*, 2019. 162 : p. 103-107.
 4. **S. Deschanel**, W. Ben Rhouma, J. Weiss, Acoustic emission multiplets as early warnings of fatigue failure in metallic materials. *Scientific Reports* 7, 13680, (2017).
 5. N. Casiez, **S. Deschanel**, T. Monnier, O. Lame, Ultrasonic in situ investigation of the initiation of Polyethylene's plastic deformation during tensile tests, *Polymer* 123(Supplement C) (2017) 258-266.
 6. A. Tantot, C. Bouard, R. Briche, G. Lefevre, B. Manier, N. Zaim, **S. Deschanel**, L. Vanel, P. Di Stefano, A multi-channel setup to study fractures in scintillators. *Measurement Science and Technology*, 2016. 27(12) : p. 125601.
 7. J. Weiss, W. Ben Rhouma, T. Richeton, **S. Deschanel**, F. Louchet, and L. Truskinovsky, From Mild to Wild Fluctuations in Crystal Plasticity, *Physical Review Letters*, 114 (2015) 105504.
 8. N. Casiez, **S. Deschanel**, T. Monnier, O. Lame, Acoustic emission from the initiation of plastic deformation of Polyethylenes during tensile tests. *Polymer*, 55 (2014) 6561-6568.
 9. **S. Deschanel**, L. Vanel, N. Godin, E. Maire, G. Vigier, and S. Ciliberto, Mechanical response and fracture dynamics of polymeric foams. *Journal of Physics D : Applied Physics*, 2009(21) : p. 214001.
 10. Godin, N., S. Chaki, J. Courbon, **S. Deschanel**, S. Gillet, and B. Gautier, Acoustic emission potentialities for characterization of Mullins effect in natural rubber materials filled with carbon black. *Polymer Testing*, 2009. 28(1) : p. 103-105.
 11. **S. Deschanel**, B.P. Grevskis, K. Bertoldi, S.S. Sarva, W. Chen, S.L. Samuels, R.E. Cohen, and M.C. Boyce, Rate dependent finite deformation stress-strain behavior of an ethylene methacrylic acid copolymer and an ethylene methacrylic acid butyl acrylate copolymer. *Polymer*, 2009. 50(1) : p. 227-235.
 12. **S. Deschanel**, L. Vanel, N. Godin, G. Vigier, and S. Ciliberto, Experimental study of crackling noise : conditions on power law scaling correlated with fracture precursors. *Journal of Statistical Mechanics : Theory and Experiment*, 2009(01) : p. P01018.
 13. Bertoldi, K., M.C. Boyce, **S. Deschanel**, S.M. Prange, and T. Mullin, Mechanics of deformation-triggered pattern transformations and superelastic beha-

- behavior in periodic elastomeric structures. *Journal of the Mechanics and Physics of Solids*, 2008. 56(8) : p. 2642-2668.
14. Boyce, M.C., S.M. Prange, K. Bertoldi, **S. Deschanel**, and T. Mullin. Mechanics of periodic elastomeric structures. in *Constitutive Models for Rubber V*. 2008. Paris - France : Taylor & Francis Ltd.
 15. Degischer, H.P., A. Kottar, B. Foroughi, E. Maire, N. Godin, V. Carmona, **S. Deschanel**, and F. Simancik. Tensile deformation and cracking sequence of foamed AlSi10 with skin. in *Porous Metals and Metallic Forms : MetFoam 2007*. 2008 : DEStech Publications, Inc.
 16. Mullin, T., **S. Deschanel**, K. Bertoldi, and M.C. Boyce, Pattern Transformation Triggered by Deformation. *Physical Review Letters*, 2007. 99(8) : p. 084301-4.
 17. Sarva, S.S., **S. Deschanel**, M.C. Boyce, and W. Chen, Stress-strain behavior of a polyurea and a polyurethane from low to high strain rates. *Polymer*, 2007. 48(8) : p. 2208-2213.
 18. Santucci, S., P. Cortet, **S. Deschanel**, L. Vanel, and S. Ciliberto, Subcritical crack growth in fibrous materials. *Europhysics Letters*, 2006. 74(4) : p. 595-601.
 19. **S. Deschanel**, L. Vanel, G. Vigier, N. Godin, and S. Ciliberto, Statistical properties of microcracking in polyurethane foams under tensile test, influence of temperature and density. *Int. J. Fracture*, 2006. 140 : p. 87-98.

National refereed journals

20. **S. Deschanel** & J. Weiss, Contrôle de la fatigue des matériaux par émission acoustique. *Les Techniques de l'Ingénieur*, n°IN226. A paraître Décembre 2018.
21. **S. Deschanel** & J. Weiss, Vers une détection précoce de la rupture par fatigue par émission acoustique. *Traitements & Matériaux* 450, 41-44 (Janvier-Février 2018).
22. Godin, N., **S. Deschanel**, M. R'Mili, P. Reynaud, J. Lamon, G. Fantozzi, Identification de la signature acoustique des différents mécanismes d'endommagement lors d'essais de fatigue sur CMC. Application de classificateurs supervisé et non supervisé. *Revue des composites et des matériaux avancés*, 2013. 23(1) : p.85-98. doi :10.3166/rcma.23.85-98.
23. Godin, N., **S. Deschanel**, and J. Courbon, Émission acoustique : identification de la signature acoustique des mécanismes d'endommagement et prévision de la durée de vie. *Mater. Tech.*, 2009. 97(1) : p. 35-42. Book chapter
24. **S. Deschanel**, J. Courbon, and G. Vigier, Durée de vie et potentiel restant des matériaux et des structures. *Les controverses technologiques de L'INSA de Lyon*, Juillet 2009 . Publié aux Presses polytechniques et universitaires romandes.

Book chapter

25. **S. Deschanel** , J. Courbon, and G. Vigier, Durée de vie et potentiel restant des matériaux et des structures. Les controverses technologiques de L'INSA de Lyon, Juillet 2009 . Publié aux Presses polytechniques et universitaires romandes.

Conference proceedings

26. **S. Deschanel**, A. Tantot, L. Vanel, P. Di Stefano, Light and Acoustic Emissions during fracture in inorganic scintillators. Proceedings, 14th International Conference on Fracture, ICF14. Rhodes (Greece), 2017.
27. P. C. F. Di Stefano, C. Bouard, S. Ciliberto, **S. Deschanel**, O. Ramos, S. Santucci, A. Tantot, L. Vanel and N. Zaim, From Dark Matter to Brittle Fracture. Fracture, Fatigue, Failure and Damage Evolution, vol. 8, 183-186 (2016)
28. **S. Deschanel**, W. Ben Rhouma, J. Weiss, Acoustic Emission multiplets during fatigue of aluminum. Proceedings, 13th International Conference on Fracture, ICF13. Beijing (China), 2013.
29. Ben Rhouma W., Deschanel S., and Weiss J., Collective Dislocation Dynamics and Avalanches during Fatigue of Aluminum. AIP Conf. Proc. 1389, pp. 1536-1539, doi :<http://dx.doi.org/10.1063/1.3637920> Numerical Analysis and Applied Mathematics ICNAAM 2011.
30. F. Philip, N. Godin, **S. Deschanel**, J.M. Leroy, F. Grosjean, J. Courbon Caractérisation des endommagements d'un composite Carbone/PA12 par émission acoustique. Comptes Rendus des JNC 17 - Poitiers 2011.
31. F. Philip, N. Godin, **S. Deschanel**, J.M. Leroy, F. Grosjean, R. Estevez, J. Courbon. Identification of damage sources in thermoplastic composites (Carbon/PA12) through acoustic emission analyses based on pattern recognition, ECCM14 Budapest, 2010.
32. **S. Deschanel**, L. Vanel, N. Godin, G. Vigier, S. Ciliberto, Statistical properties of microcracking in heterogeneous materials : conditions on power-law scaling correlated to fracture precursors. ICF-12, 6, p. 4634-4642. 12th international Conference on Fracture. Ottawa (Canada), 2009.
33. **S. Deschanel**, M.C Boyce, R.E Cohen, Rate dependent mechanical performance of Ethylene Methacrylic Acid (EMAA) copolymers and POSS-enhanced EMAA nanocomposites. American Chemical Society, Division of Polymeric Materials : Science and Engineering. PMSE Preprints Volume 96, Spring 2007. ACS National Meeting, Chicago, USA. March 25-29, 2007.
34. S.Ciliberto, **S. Deschanel**, A.Guarino, S.Santucci, R.Scorreti, L.Vanel Failure time, critical behavior and activation processes in crack formation - Proceedings of the 11th International Conference on Fracture, Turin, Italy (March 20-25, 2005)

Appendices

Microstructure characterization of pure Cu single crystal

This appendix presents the microstructural characterization of dislocation structures of a sole pure copper single crystal specimen ([110]), under imposed rising alternating stress levels. Five stress-imposed fatigue steps, lasting 5000 cycles each, of increasing stress amplitudes were performed : 1-10, 1.5-15, 2-20, 2.5-25, and 3-30 MPa RSS.

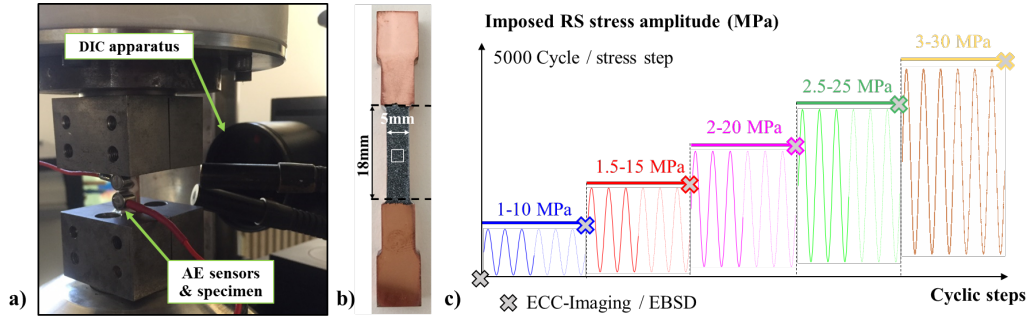


FIGURE A.1 – (a) Cyclic fatigue machine with DIC apparatus. (b) Photograph of the copper specimen used Gabriel L'Hôte PhD thesis with the typical speckle pattern on the gauge length. (c) Stress-imposed fatigue steps.

The figure A.2 presents EBSD map -texture component- (left-hand column) and R-ECCI images showing the evolution of the dislocation structure with imposed stress from the initial state to the 3-30 MPa RSS. The texture component gives information about the orientation difference with respect to a reference orientation (chosen in each image). Dislocations appear in white in the ECCI images. Dislocation mean free paths, which corresponds to the characteristic length of the dislocation structure (cell size, and distance between walls), were measured manually. The mean free path (λ_c and λ_w referred to the mean path of the cell and the wall structure, respectively) related to each dislocation structure is indicated on the right of the figure. Red arrows show how λ were measured.

The dislocation density ρ has been estimated using the same methodology as the one proposed in Ham works [Ham 1961].

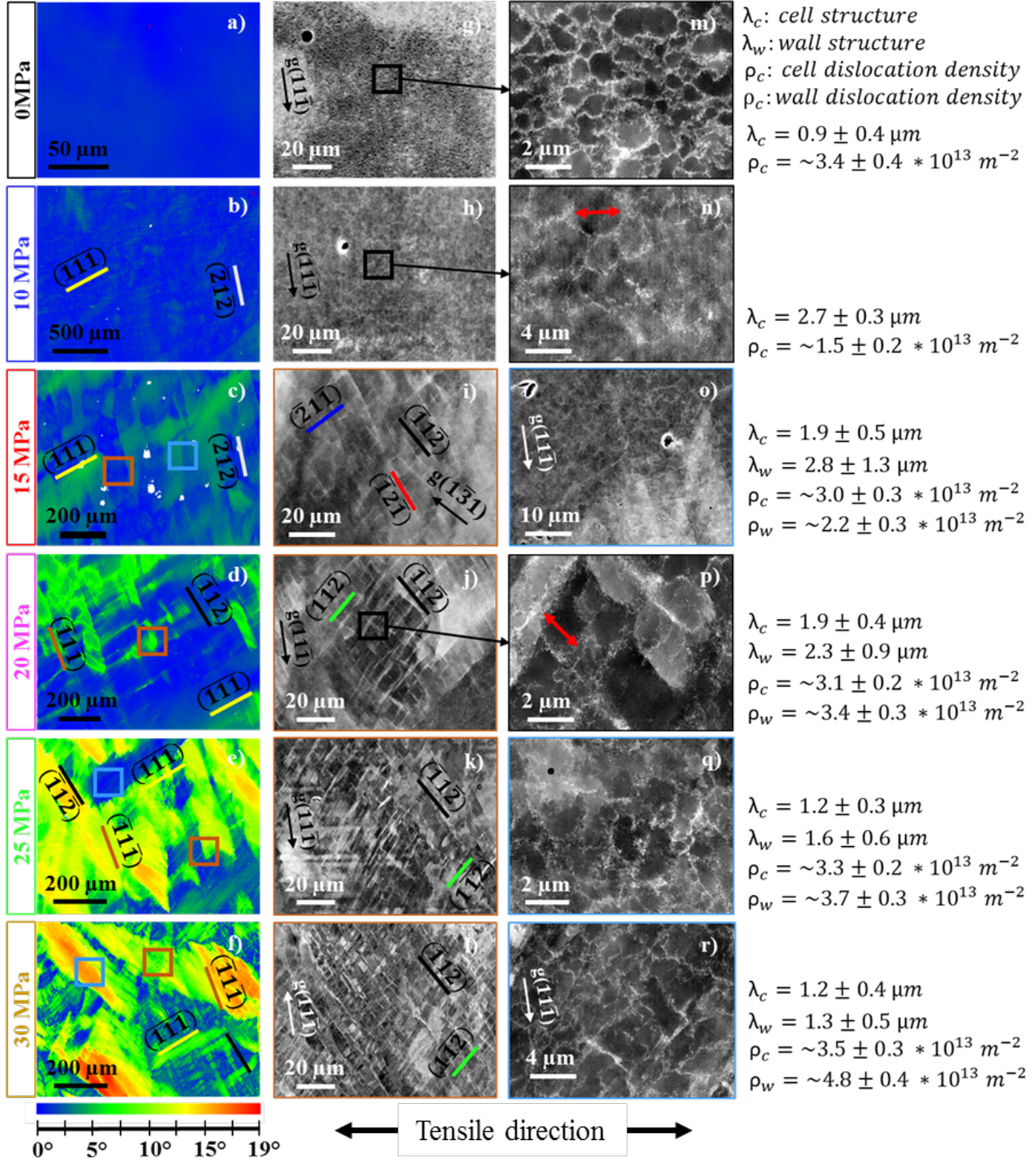


FIGURE A.2 – Microstructure characterization : EBSD maps and ECCImages.

Localization using correlation of WFs within the AE multiplets

This appendix presents in more details the methodology for the 1D-localization of the sources of the bursts included in a multiplet, using their cross-correlations. The localization along the sample and relatively to other members of the multiplet can thus be determined with improved accuracy.

Classical procedure

Let's note $t_{1,i}$ and $t_{2,i}$ the arrival times of the different WFs of a given multiplet, for sensor 1 and 2 respectively, given by the first threshold crossing. Using the classical localization procedure, the positions of the different members of the multiplet would be given by $p_i = (t_{1,i} - t_{2,i})\frac{v}{2} + \frac{d}{2}$. This methodology gives systematically dubious shifts in the absolute position of the source within the multiplet (black squares of figure 3.5(b)) that would be in contradiction with a common origin.

Localization using correlation between waveforms

To improve the accuracy on Δt (difference of arrival times between sensor 1 and sensor 2) and to circumvent the limitations of the time arrival estimation, Grabec proposed to cross-correlate the signals of a same event received by different transducers, with Δt maximizing this cross-correlation [Grabec 1978]. However, this methodology is only applicable if the characteristic responses of the two sensors are very similar (i.e. $D_{sen1}(f) \approx D_{sen2}(f)$) and if the medium crossed by the elastic waves is homogeneous, ensuring $G_{sen1}(f) \approx G_{sen2}(f)$. Both conditions are hardly fulfilled, especially in a damaged medium. We checked this methodology, and found that in few cases, for AE multiplets recorded during the early stages of damaging, i.e. when the material remains relatively homogeneous, the correlation coefficients between WFs of a same event recorded at the two sensors over the interval 0-40 μs were in the range 0.5 to 0.8 (figure B.1a), i.e. slightly larger than coefficients obtained between WFs randomly selected. Then, for WF i within such multiplet, the cross-correlation gives a time lag $\delta t_{sen1/sen2}^i$ that is used to correct $\Delta t_i = (t_{1,i} - t_{2,i}) + \delta t_{sen1/sen2}^i$ and consequently the associated location. In these cases, AE events belonging to a same multiplet are localized around a mean position, with a standard deviation of the order of 500 μm , without spurious shifts (figure B.1a). However, in most cases, the cross-correlations between WFs

recorded at sensors 1 and 2 are non-significant, i.e. of the order of correlations found for randomly selected WFs. In those cases, the Grabec's methodology leads to unphysical shifts in position within a given multiplet (figure B.1b, red dots), illustrating the shortcomings of this method when the medium is too damaged and therefore the condition $G_{sen1}(f) \approx G_{sen2}(f)$ no longer fulfilled.

Consequently, we are here taking advantage of the correlated WFs within the multiplets, recorded at the same sensor, to improve the location of the AE signals and to present a new way to map the sources. In this case, $D(f)$ is unchanged by definition, whereas $G(f)$ remains almost unchanged as long as we consider only the initial impulsive part (0-40 μ s) of the WFs. Inspired by studies of seismic multiplets (or repeating earthquakes), (e.g. [Poupinet *et al.* 1984, Rubin *et al.* 1999, Lengliné & Marsan 2009]), we can perform a relative localization of the different WFs in the multiplet using the time delay δt found between two WFs in a multiplet.

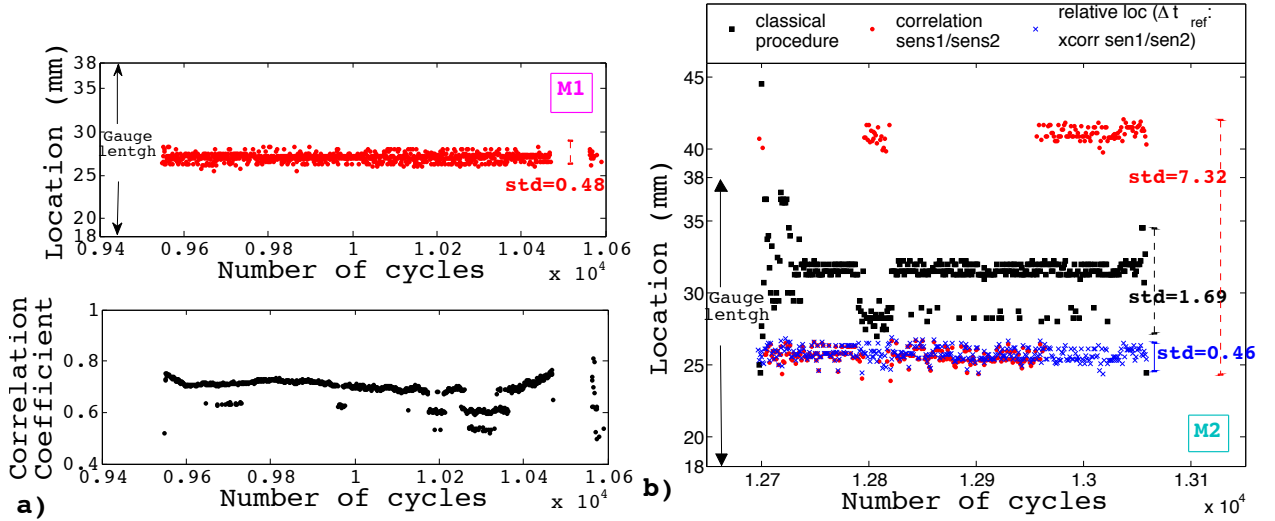


FIGURE B.1 – Fatigue test at $\Delta\varepsilon = 0.5\%$ on Al at 0.1Hz. (a) Localization of AE signals of multiplet M1 of figure 3.2, using cross correlations of the signals received by the two sensors and corresponding correlation coefficients. (b) Localization of AE signals of multiplet M2 of figure 3.2, using classical procedure (black $\square$), Grabec's methodology (red $\circ$) and our method : correlation of WFs within a multiplet (blue $\times$).

We choose an event as a reference in the multiplet, for which the difference of arrival times between sensor 1 and 2 sets the reference position. This can be done for instance using the first threshold crossing i.e. $\Delta t_{ref} = (t_{1,ref} - t_{2,ref})$, or alternatively from the Grabec's cross-correlation methodology mentioned above. These two estimations of the absolute position of the reference are significantly different, generally by more than few *mm*. This illustrates the intrinsic uncertainty

in absolute positioning of AE sources. Also, as the exact location is required for the reference event, we can perform a manual inspection of the two WFs to obtain the exact time of arrival from the time of the first peak.

For each sensor, we then compute the cross correlations of all the WFs in a multiplet compared with the reference : time delays between each WF and the reference, $\delta t_{1,i}$ and $\delta t_{2,i}$, are obtained. A new time of arrival can thus be determined for each wave in the multiplet : $t_{1,i}^{corr} = t_{1,i} + \delta t_{1,i}$ and $t_{2,i}^{corr} = t_{2,i} + \delta t_{2,i}$, giving a new time interval $t_{1,i}^{corr} - t_{2,i}^{corr}$. Finally, $\Delta t_{1,i}^{corr} = (t_{1,i}^{corr} - t_{2,i}^{corr}) + \delta t_{sen1/sen2}^{ref}$ allows to determine the position of the event i *relatively* to the reference : $p_i^* = \Delta t_{1,i}^{corr} \frac{v}{2} + \frac{d}{2}$. The irreducible limitation of this method is directly linked to the limitation of the system in terms of clock synchronization between the different sensors (accuracy¹). For velocities $3500 < v < 5000 \text{ms}^{-1}$, this translates into an uncertainty on the positions between 350 and 500 μm . We show in figure 3.5(b) (blue crosses) that taking advantage of the correlated WFs within a multiplet allows suppressing the spurious shifts in position obtained with the classical or the Grabec's procedures. Moreover, the standard deviation of the position (relatively to the reference) falls down to 460 μm , i.e. very close to the theoretical uncertainty of 500 μm for Al. This strongly argues for a unique source. On the other hand, the *absolute* position of this source remains uncertain to a larger extent (few mm).

1. The hardware clock of the system is set to an accuracy of $0.1\mu\text{s}$ which represents the irreducible synchronization accuracy between the clocks of the different transducers

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