

5.2 Physical metallurgy and manufactured materials

A. Context

Optimizing materials for targeted applications is the main aim of materials science and engineering. Materials can of course be optimized through the design of new alloy compositions, but also by **designing processing routes that lead to optimal microstructures** to enhance the properties of interest. CEMEF is a research center of MINES ParisTech located in Sophia Antipolis and dedicated to materials processing. About 150 persons including more than 60 PhD students are working at CEMEF on essential topics for the understanding of forming processes for all types of materials, and in close collaboration with industrial partners. CEMEF has notably a worldwide recognized expertise in manufactured metallic materials and related processes.

The works devoted to the **evolution of metallic microstructures upon thermomechanical processing** are based on the **combination of advanced experimental assessment of physical phenomena and advanced numerical models** of those phenomena. Combining high-level expertise on both experimental and numerical approaches makes a specificity of the works carried out in this field; Figure 1 is a synoptic scheme of the overall approach.

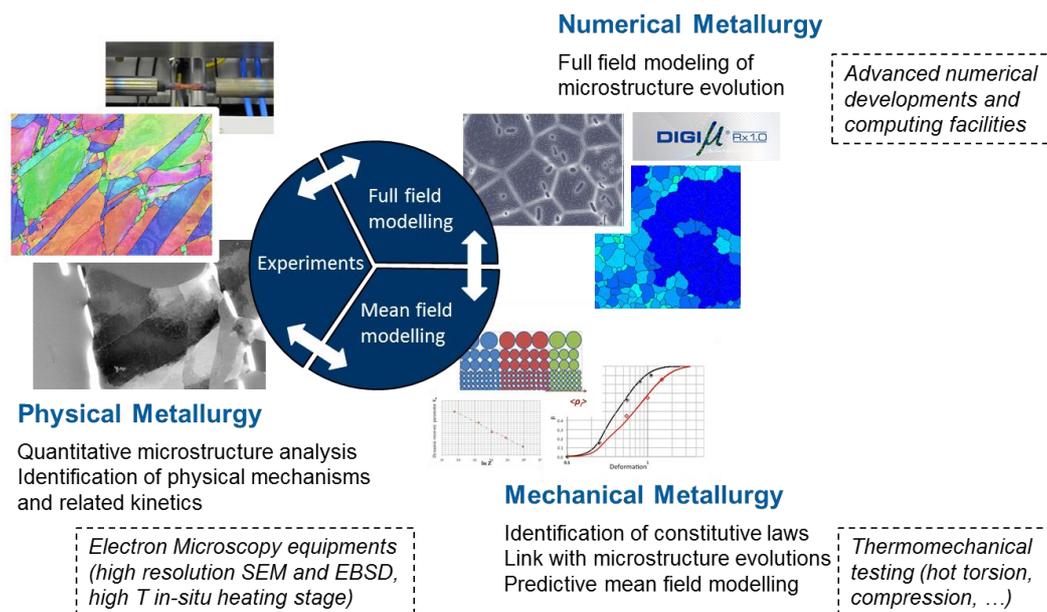


Figure 1. Synoptic scheme of the experimental and modeling approaches for studying microstructure evolution during thermomechanical processing of metallic materials

Most of the work is made to solve microstructure control issues raised by industrial partners. The temperature, strain and strain rate conditions of the industrial process are reproduced at the lab scale using thermomechanical testing devices (torsion, compression at room temperature or high temperature, up to typically 1200°C, thermal treatments up to 1500°C under vacuum or inert atmosphere). The lab tests are interrupted at different strain levels, so that the evolution of microstructure can be analyzed step-by-step. **Advanced microscopy and analysis techniques are necessary to fully reveal the microstructure complexity of engineering materials (cf Theme 12.3)**. In addition, specific equipment and analytical methods are developed when necessary. A heating stage has been designed to perform high speed and high temperature annealing inside the scanning electron microscope (SEM) chamber [1]. CEMEF is among the only few labs in the world to possess such a facility. Quantitative methods have also been settled to perform for example texture analysis from EBSD data [2] or phase analysis by combining two different

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SEM-related techniques [3]. The expertise of CEMEF in **quantitative microstructure analysis** is well established and is part of the research activity in physical metallurgy by itself.

Based on fine and quantitative microstructure analyses, the physical mechanisms responsible for the metallurgical evolutions and their kinetics are identified. The main involved mechanisms in this field are : plasticity, recovery, recrystallization, grain growth, precipitation and phase transformations, and coupling between those phenomena. **The identified mechanisms and kinetics are then modelled** and implemented in numerical simulation codes. Two types of numerical models can be defined:

- the so-called **mean-field models** [4], that statistically describe the microstructure evolution in terms of phase volume fractions, grains size distribution, dislocation densities, recrystallized fraction and so on, but with no topological description

- the so-called **full-field models**, that explicitly represent the (2D or 3D) polycrystalline microstructure and make a representative volume element evolve according to physical laws. Full-field models require the **development of advanced, suitable, efficient, and robust numerical methods** (cf Theme 5.3) and high performance computing resources (cf Theme 12.2), while mean field models are better suited for use in an industrial context. Nevertheless, full-field models are absolutely necessary when dealing with local microstructure instabilities that can hardly be described on a statistical basis. To date, CEMEF has one of the most developed set of models for describing (static and dynamic) recrystallization phenomena in the world and a very good command of the underlying physics. It is worth mentioning that those models are not simply phenomenological but physically based, so that are adaptable to other materials and processes.

[1] Bozzolo, N., Jacomet, S., Logé, R.E. Fast in-situ annealing stage coupled with EBSD: A suitable tool to observe quick recrystallization mechanisms, *Mat. Charact.* **70**, 28-32 (2012)

[2] Bozzolo, N., Gerspach, F., Sawina, G., Wagner, F., Accuracy of orientation distribution function determination based on EBSD data – Case of a recrystallized low alloyed Zr sheet", *J.Microsc.* **227**, 275-283 (2007)

[3] Charpagne, M.-A., Vennéguès, P., Billot, T., Franchet, J.M., Bozzolo, N., Evidence of multimetric coherent γ' precipitates in a hot-forged γ - γ' nickel-based superalloy, *J. Microsc.* (2016) in press

[4] Zouari, M., Bozzolo N., Logé, R.E., Mean field modelling of dynamic and post-dynamic recrystallization during hot deformation of Inconel 718 in the absence of δ phase particles, *Mat. Sc. Eng. A* **655** 408-424 (2016)

[5] Scholtes, B., Shakoor, M., Settefrati, A., Bouchard, P.O., Bozzolo, N., Bernacki, M., New finite element developments for the full field modeling of microstructural evolutions using the Level-Set method, *Comp. Mat. Sc.* **109**, 388-398 (2015)

B. Current activity



A great part of the current activity on microstructure of manufactured metallic materials concerns **nickel base superalloys**, owing to the existence of the OPALE industrial chair [6], co-funded by the French research funding agency ANR and the industrial group Safran. OPALE aims at optimizing properties of nickel base superalloys by controlling the microstructures obtained after forging. Superalloys are used for manufacturing pieces of turbojet engines owing to their mechanical strength at high temperature. Improving the performance of those materials will allow higher operating temperatures and therefore a better efficiency of the engines. This chair is based on a need expressed by the Safran group and brings together the expertise of CEMEF (MINES ParisTech, UMR CNRS) on the impact of processing on microstructure and the expertise of the Institute P' (UPR CNRS ISAE / ENSMA) on the impact of microstructure mechanical properties and in-service behavior. This ambitious project therefore covers a wide research field: from the fine material analysis and his mechanical behavior, to the modeling of the process-microstructure and microstructure-properties relationships. Fundamental research works (experimental and numerical developments, analysis of metallurgical mechanisms) will feed studies derived from industrial issues (support for the development of processing routes or new processes). Nathalie Bozzolo, professor in Physical Metallurgy at CEMEF, is the chair holder.

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Apart from nickel base superalloys, the combined experimental/numerical methodology described in section A is applied to many **other metallic materials and processes**, e.g. steels, titanium alloys, TiAl intermetallics, pure tantalum.

The numerical models for simulating recrystallization phenomena and predicting the microstructure obtained after a given thermomechanical path are in addition implemented into **software packages that are made available to the industrial partners** : through the full field simulation package DIGIMU® [7], and the DynamiX+ tool that can be used in combination with the Forge® commercial software. Digimu is being developed by Marc Bernacki at CEMEF in cooperation with a large industrial consortium :



[6] <http://chaire-opale.cemef.mines-paristech.fr/>

[7] <https://www.transvalor.com/fr/cmspages/digimu.86.html>

C. Future steps

Up to now, our work has been mainly focused on recrystallization phenomena. In order to better describe the behavior of a number of industrial alloys (steels, titanium alloys), studying the **coupling of phase transformations with recrystallization phenomena** became a necessity. This is one planned development. Another one concerns the nature of the investigated materials. The knowledge acquired on existing industrial materials can **help developing new materials and new processing routes**.

For instance, a French-German project has been submitted this year with Pr. G. Dehm (Max Plack Institut für Eisenforschung MPIE, Düsseldorf) to investigate the stability of **High Entropy Alloys**. HEAs are a new class of materials, which can realize exceptional combinations of mechanical, electrical and thermal properties unachievable by conventional alloys. They contain about equal amounts of at least five major elements, and can surprisingly crystallize as single solid solution face-centred cubic (fcc) or body-centred cubic (bcc) phases. A better knowledge of the metallurgical and physical behavior of those materials is a prerequisite for any future application.

Another example is also a French-German project submitted together with MPIE, but another team (Microstructure and Microscopy group of Dr. S. Zaefferer), that is devoted to development of processing routes to achieve the so-called **Grain Boundary Engineering** principle. The aim is then to control the density of grain boundaries and to optimise their morphological, crystallographic and chemical features, in order to improve the material resistance to intragranular damage (crack propagation and corrosion resistance).

Such a microstructure design strategy requires an integrated approach where both the physical principles of microstructure evolution during processing and the microstructure-property relations must be studied in detail, often at a very fine scale. Furthermore, the accurate description of the physical phenomena by which microstructure evolves more and more often requires experimental data and models accounting for the real 3D topology. **Developing the necessary tools to go to 3D descriptions at a fine scale** is thus also part of our plans for the near future.

It is worth mentioning here also promising collaborations with Pr. Guy Libourel (OCA) for the **characterization of meteoritic materials with the aim of better understanding the mechanisms by which they have formed**. For us : another type of material and a long-term evolution through a natural process, that pushes us to revisit our research strategy. As compared to conventional physical metallurgy studies where as much material as necessary is available, and almost as many experiments as necessary can be done to test hypothetical mechanisms, working on rare samples with no way of interacting with the formation process is a great intellectual experience. This collaboration has led to a joint research proposal that is being reviewed by the ANR (cf **Theme 5.4**).

D. International collaborations

Our main academic collaborations abroad are briefly listed below with names and topics :

- Pr A.D. Rollett and Pr G.S. Rohrer, Carnegie Mellon University, Pittsburgh, USA
International ANR-NSF Materials World Network project (annealing formation mechanisms and grain boundary engineering) ; Characterization of grain boundary character distributions
- Dr S. Zaeferrer, Max Plank Institut für Eisenforschung, Düsseldorf, Germany
Advanced electron microscopy techniques, Grain Boundary Engineering
- Pr G. Dehm and Pr. C. Scheu, Max Plank Institut für Eisenforschung, Düsseldorf, Germany
Interface physics and high entropy alloys
- Pr R.E. Logé, Ecole Polytechnique Fédérale de Lausanne, Neuchatel, Suisse
Thermomechanical testing, Mean field modelling of recrystallization phenomena
- Pr J. Signorelli, CONICET, Argentina
Development of advanced methods for quantitative analysis of EBSD data

E. List of people involved in the project

Permanent :

- Nathalie BOZZOLO (Pr, Physical Metallurgy)
- Marc BERNACKI (Pr, Numerical Metallurgy)
- Charbel MOUSSA (Assistant Pr., Mechanical Metallurgy)

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Engineers :

- Suzanne JACOMET (Electron microscopy)
- Gilbert FIORUCCI (Thermomechanical testing)
- Cyrille COLLIN (Metallography)

Post-doc :

- Dmitrii Ilin (Full field modelling of crystal plasticity)

PhD :

- Benjamin Scholtes (Developpement of the DIGIMU software)
- Marie-Agathe Charpagne (Microstructure of the forged René65 superalloy)
- Danai Polychronopoulou (Globularisation of lamellar titanium alloys)
- Romain Boulais-Sinou (Full field modelling of dynamic recrystallization)
- Anthony Seret (Influence of dislocation density on precipitation kinetics in Inconel 625 superalloy)
- Suzanne Vernier (Microstructure of forged the AD730 superalloy)
- Ludovic Maire (Coupling of mean field and full field models)

F. Most significant publications of the team

Jin, Y, Lin, B, Bernacki, M, Rohrer, GS, Rollett, AD, Bozzolo, N, Annealing twin development during recrystallization and grain growth in pure nickel. *Mat. Sc. Eng. A* **597**, 295-303 (2014).

Agnoli, A, Bozzolo, N, Logé R, Franchet, JM, Laigo, J, Bernacki, M, Development of a level set methodology to simulate grain growth in the presence of real secondary phase particles and stored energy - Application to a nickel-base superalloy. *Comp. Mat Sc.* **89**, 233-241 (2014).

Bozzolo, N., Souai, N., Loge, R. E., Evolution of microstructure and twin density during thermomechanical processing in a gamma-gamma ' nickel-based superalloy. *Acta Mat.* **60**:13-14, 5056-5066 (2012)

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Bozzolo, N, Chan, L., Rollett, A D., Misorientations induced by deformation twinning in titanium. *J. Appl. Cryst.* **43**, 596-602 (2010)

Bernacki, M; Chastel, Y, Coupez, T, Loge, RE, Level set framework for the numerical modelling of primary recrystallization in polycrystalline materials. *Scripta Mat.* **58:12**, 1129-1132 (2009).

Short CV of participants

N Bozzolo, Professor in Physical Metallurgy at CEMEF MINES-ParisTech. Expert in quantitative analysis of microstructures and textures, identification of metallurgical mechanisms and recrystallization phenomena. Holder of the OPALE ANR-Safran industrial chair. Member of the national board of the French Society for Metallurgy and Materials (SF2M). Head of research group "Metallurgy, Structure, Rheology"

M. Bernacki, Professor in Numerical Metallurgy at CEMEF MINES-ParisTech. Expert in numerical developments for the simulation of microstructure evolution and damage phenomena. Applicant for an ERC grant in 2016. Head of the "Numerical Materials" committee of the SF2M. Head of research group "MultiScale Modelling".

C. Moussa, Assistant Professor in Mechanical Metallurgy at CEMEF MINES-ParisTech. Expert in mechanical testing and mean field modelling of recrystallization phenomena in relation to the constitutive behavior. Member of research group "Metallurgy, Structure, Rheology".

S. Jacomet, Engineer in charge of Electron Microscopy and related techniques (EDS, EBSD, in-situ annealing) at CEMEF MINES ParisTech.

G. Fiorucci, Engineer in charge of the thermomechanical testing equipments and thermal treatment facilities at CEMEF MINES ParisTech.