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# A MESOSCALE EXPERIMENTAL APPROACH OF PLASTICITY AND DAMAGE

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## Abstract

In order to observe the phenomena occurring during deformation, mechanical test stages working inside a scanning electron microscope (SEM) chamber have been developed; they allow us to perform tests step by step, without unloading, with a fixed tensile axis, and to make observations at a constant magnification. Two stages are available: a tensile stage with a load cell in the range 0-500 daN and a micro furnace to heat the specimen up to 800 °C, a biaxial tension/torsion out-of-phase fatigue stage with a tension compression load up to  $\pm 1000$  daN and a torque of  $\pm 50$  Nm.

This system is completed by an image analyzer used to quantify microstructure morphologies and an electron backscattering diffraction device (EBSD) that allows us to determine the local crystalline orientation and to follow its evolution under deformation. A fiducial grid technique has been developed to measure some components of the local strain tensor. The microgrids are deposited on the surface of the sample by an electrolithographic technique; their step ranges from 1 to 10  $\mu\text{m}$  or more; they provide a good contrast for SEM and the underlying microstructure remains visible. From a qualitative point of view, the deformation of the grid gives indications on the local scale mechanisms that occur during the deformation of the sample. Image analysis techniques allow us to measure local displacements in order to quantify the local strain heterogeneities; strain maps can also be plotted over domains representative of the microstructure.

## Keywords

Scanning electron microscope, *In situ* mechanical tests, Electron backscattering diffraction, Fiducial microgrids, Local strain measurement, Deformation maps, Image analysis.

## 1. INTRODUCTION

Material Mechanics has a two-fold scope:

- to give a prediction of the overall mechanical behaviour of multiphase materials that can be integrated in structural calculations to predict the behavior of structures in service,
- to give rules for optimizing the mechanical properties of materials through the optimization of their microstructure.

Both of these scopes rely on the production of microstructure-based models.

The estimation of the overall elastoplastic behaviour of inhomogeneous materials from the behaviour of their constituents, is a classical but still unsolved problem. All the existing models make very different assumptions on the local interactions, which induce very different levels of stress and strain heterogeneities. The main microstructural characteristics of a considered material allow one to choose the pertinent type of model; as a matter of fact, the overall behaviour can be strongly influenced by very local events such as damage nucleation, initiation and propagation of micro-cracks or shear bands, recrystallization processes, that depend mainly on the mechanical state at the local scale. Then, the pertinence of a model has of course to be tested on its ability to give a good estimation of the global (macroscopic) behavior; but it is also of great interest to check the model hypotheses and estimations at the local (microscopic) scale.

## **2. REQUIREMENTS OF A MICRO-MACRO EXPERIMENTAL APPROACH**

Generally, the physical mechanisms of deformation are known in much detail, but are often difficult to introduce directly in scale transition models based on continuum mechanics; in order to do so, the mechanisms need to be transcribed in terms of stress and strain fields. Nevertheless in most of the cases that transcription is not enough because the results on the mechanisms are obtained in homogeneous and monotonous conditions that are not relevant to represent the mechanical state inside an heterogeneous material even if it is loaded under macroscopic uniform conditions. Of course the microscopic mechanisms are still valid but their interaction under complex loading (multiaxial, non-radial, cyclic...) have to be studied; the chronology and location of their occurrence have to be specified with respect to the microstructure.

All this leads to the development of specific experimental techniques, often at an intermediate scale, and devoted to the determination of the "missing links" concerning the active mechanisms and the morphological description of the initial and induced microstructure.

These experiments have two main objectives:

- provide the basic elements to implement a scale transition model,
- validate the estimations of the model at the pertinent scales.

Then one must be able to:

- observe and characterize the initial state and the evolution of the microstructure by means of the identification of phases, of their morphology, their space distribution, and their crystalline orientation,
- identify and characterize the active deformation mechanisms (crystallographic glide, twinning, cavitation, decohesion, phase rupture, grain boundary sliding,...), precise their role and influence for the overall behavior and measure their kinetics and space distribution,
- measure at the pertinent local scale(s) and at the global scale the parameters defining the mechanical state (stress and strain fields).

Thus, *in situ* mechanical test devices are needed to be able to observe, at the different pertinent scales, the deformation mechanisms activated. Moreover, techniques allowing one to perform mechanical measurements at these scales and to follow the evolution of morphological and crystallographic parameters are essential.

## **3. DESCRIPTION OF THE EQUIPMENT**

The block diagram on figure 1 schematizes the equipment used for the local scale approach of material mechanics.

On the basis of a Scanning Electron Microscope (SEM) several complementary accessories give access to the essential parameters of the material and allow us to follow their evolution with deformation.

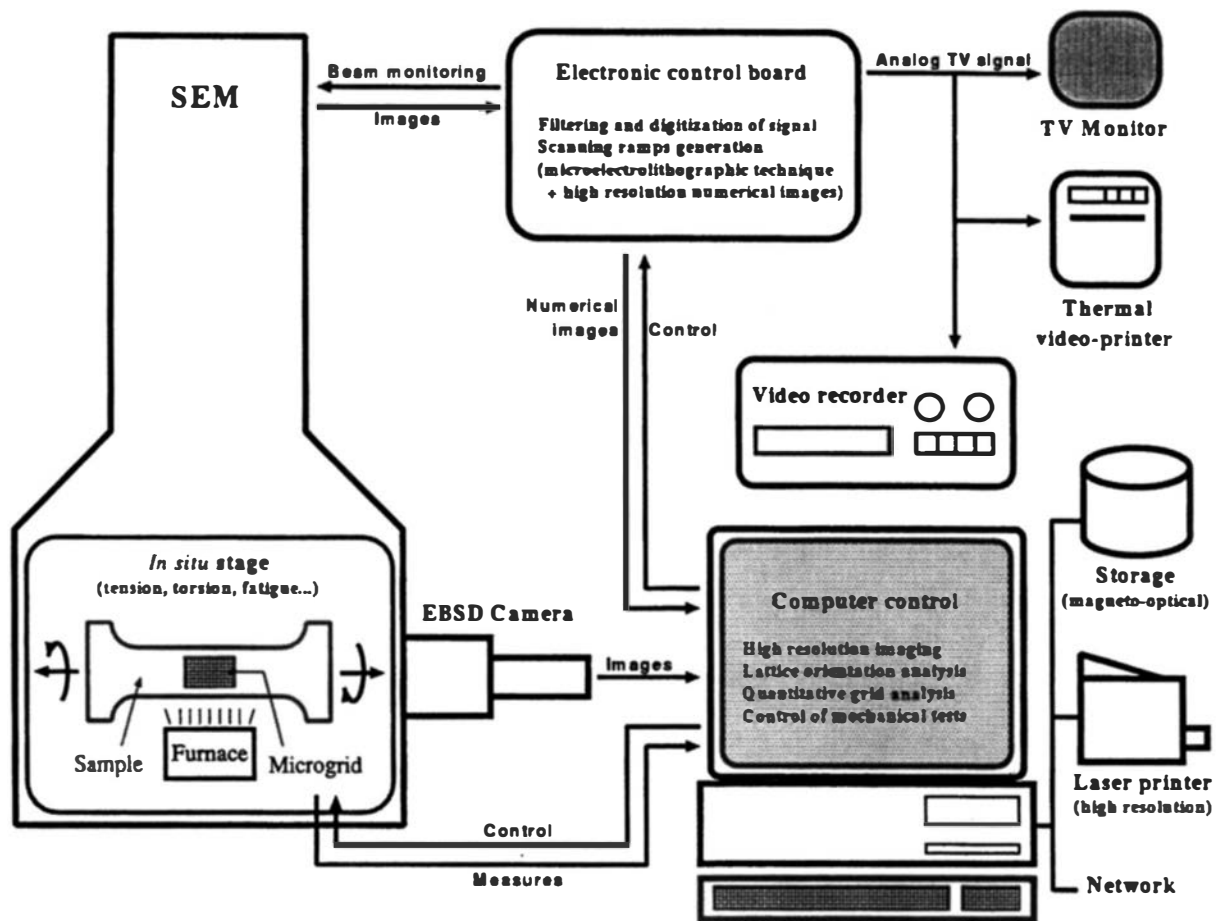


Fig. 1: Block diagram of the mesoscale micromechanical analysis equipment.

### 3.1 Scanning Electron Microscope

It allows the observation of surfaces under magnifications from  $\times 5$  to  $\times 100\,000$ ; the different contrast sources allow us to enhance either the topography induced by plastic deformation or the heterogeneities in chemical composition or in crystallographic orientation. A SEM with a large chamber is essential to the installation of *in situ* mechanical test stages suitable to house specimens much larger than the constitutive heterogeneities.

### 3.2 Tensile Stage

The use of an *in situ* tensile stage allows us to perform mechanical tests and to record the events induced by deformation, with or without unloading; it can also be proceeded step by step. The flat specimen, typically with a  $6 \times 2 \text{ mm}^2$  cross section and a 30 mm gauge length, is fixed on the tensile stage inside the SEM chamber. Under load, it can be rotated  $360^\circ$  around the Y tensile axis and translated in X and Y directions to allow the observation of a surface area exceeding  $2 \text{ cm}^2$ . The tensile tests are performed under crosshead displacement speed control in the range  $0.1 - 60 \mu\text{m/s}$ ; this leads to a strain rate range from  $10^{-5}$  to  $10^{-2} \text{ s}^{-1}$ , for usual sample gauge lengths, which is a classical range for quasi-static macro-tests; the tensile force is given by a load cell in the range  $0 - 500 \text{ daN}$ . A micro furnace can be fixed under the specimen to heat it up to  $800^\circ\text{C}$ . Different accessories allow us to test metallic, polymeric, ceramic or composite materials in tension, compression or flexion,.

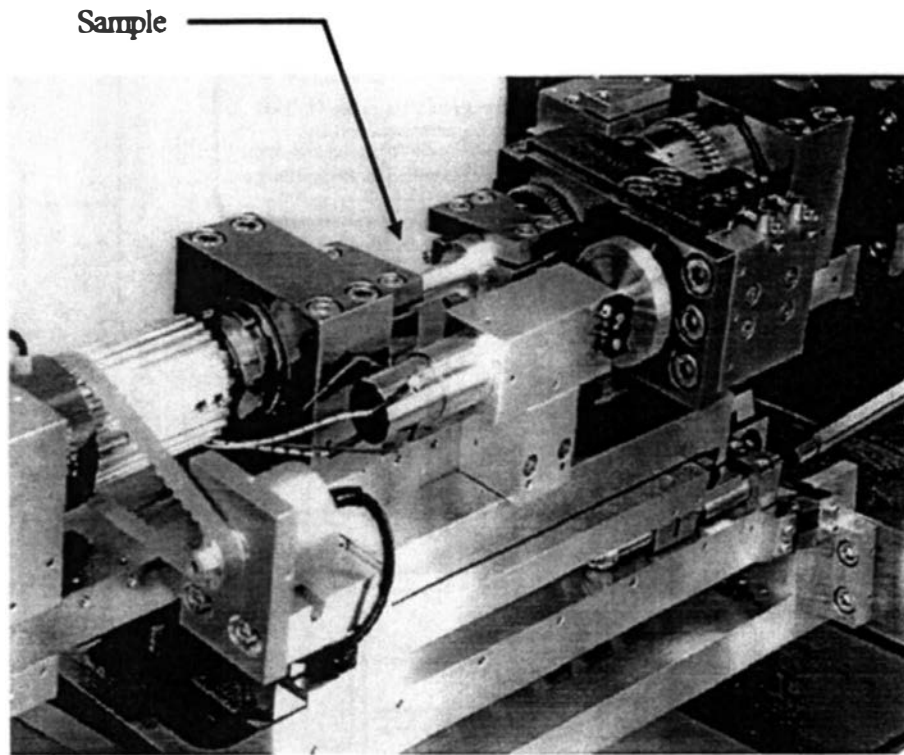


Fig. 2: Tension-torsion fatigue stage

accessories allow us to test metallic, polymeric, ceramic or composite materials in tension, compression or flexion,.

### 3.3 Tension-Torsion Fatigue Stage

A biaxial fatigue (eventually out-of-phase) stage gives access to fatigue and/or non-proportional loadings (figure 2). Thus, more realistic tests, closer to the service conditions, can be performed. Various specimens can be used depending on the type of test: crack propagation in mode I, II or in mixed mode can be performed on CT type specimens, tension/compression fatigue tests can be performed on cylindrical or parallelepipedic specimens, and torsion or tension/torsion tests require tubular specimens. The specimen can be rotated 360° around the tension/torsion axis and translated in X and Y directions. The tension/compression force is given by a load cell in the range  $\pm 1000$  daN; the torque can be applied in the range  $\pm 50$  Nm; in fatigue mode the frequency is limited to 0.1 Hz. The tests can be performed under various modes controlled by a micro-computer [2].

### 3.4 Electron BackScattering Diffraction (EBSD)

An EBSD camera, fitted to the SEM chamber, allows us to determine very quickly the crystallographic orientation of areas as small as a few  $\mu\text{m}^2$ , giving access to the non-ambiguous characterization of deformation mechanisms (slip systems, twins,...) and to the measurement of the local crystallographic rotations [3].

### 3.5 High Resolution Image Acquisition, Storage and Treatment

An electronic device linked to the SEM controls the electron beam scanning rate and operates the video signal digitization; it allows us to improve the image quality by filtering and summation. Moreover, the system can record very large size images ( $> 4000 \times 4000$  pixels) in order to ensure, in a single shot, resolution compatible with a proper characterization of the local micro-mechanical events whilst embracing a sufficiently large domain in order to get pertinent information on the statistics of these events. These images can be treated to extract, by means of image analysis techniques, statistical parameters characterizing the microstructure (e.g. covariograms)[1].

## 4. LOCAL SCALE MECHANICAL MEASUREMENTS

In order to check whether a model takes account of the inhomogeneities at the local scale (the microstructure scale), measurements of mechanical parameters (stress and strain) must be performed at that very scale.

### 4.1 The Local Scale Mechanical Parameters

The mechanical transformation at the local scale in a heterogeneous material subjected to plastic deformation can be decomposed into elastic strain ( $\epsilon^e$ ), plastic strain ( $\epsilon^p$ ), elastic rotation ( $\omega^e$ ), rigid body rotation of the unloaded lattice ( $\omega^d$ ) and plastic rotation ( $\omega^p$ ):

- $\epsilon^e$  is the deformation of the crystal lattice
- the total strain  $\epsilon = \epsilon^p + \epsilon^e$  is the deformation of a basis attached to the material
- $\omega^d + \omega^e$  is the relative rotation of the crystal lattice vs. an external basis
- $\omega^p$  is the relative rotation of a basis attached to the material vs. the crystal lattice
- the total rotation  $\omega = \omega^e + \omega^d + \omega^p$  is the relative rotation of a basis attached to the material vs. an external basis.

Several consequences can be drawn from these definitions:

- $\epsilon^e$  is the origin of the local stresses; diffraction techniques (X-rays, electrons, neutrons) allow one to measure it but they provide only average global scale values, the local values being up to now out of reach. Then  $\epsilon^e$  neither  $\epsilon^p$  can be measured at the local scale.

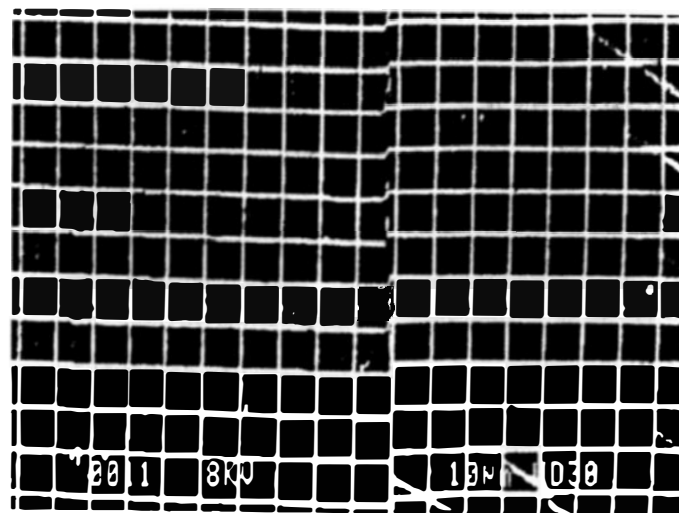


Fig. 3: Grain boundary sliding  
(the step of the grids is approximately  $5\mu\text{m}$ )

- $\omega^d + \omega^e$  can be measured by EBSD.

The determination of the local total strain  $\epsilon$  and rotation  $\omega$  requires to record the relative position of two bases:

- a basis attached to the material is essential; it is provided by the deposition on the samples of fiducial microgrids.

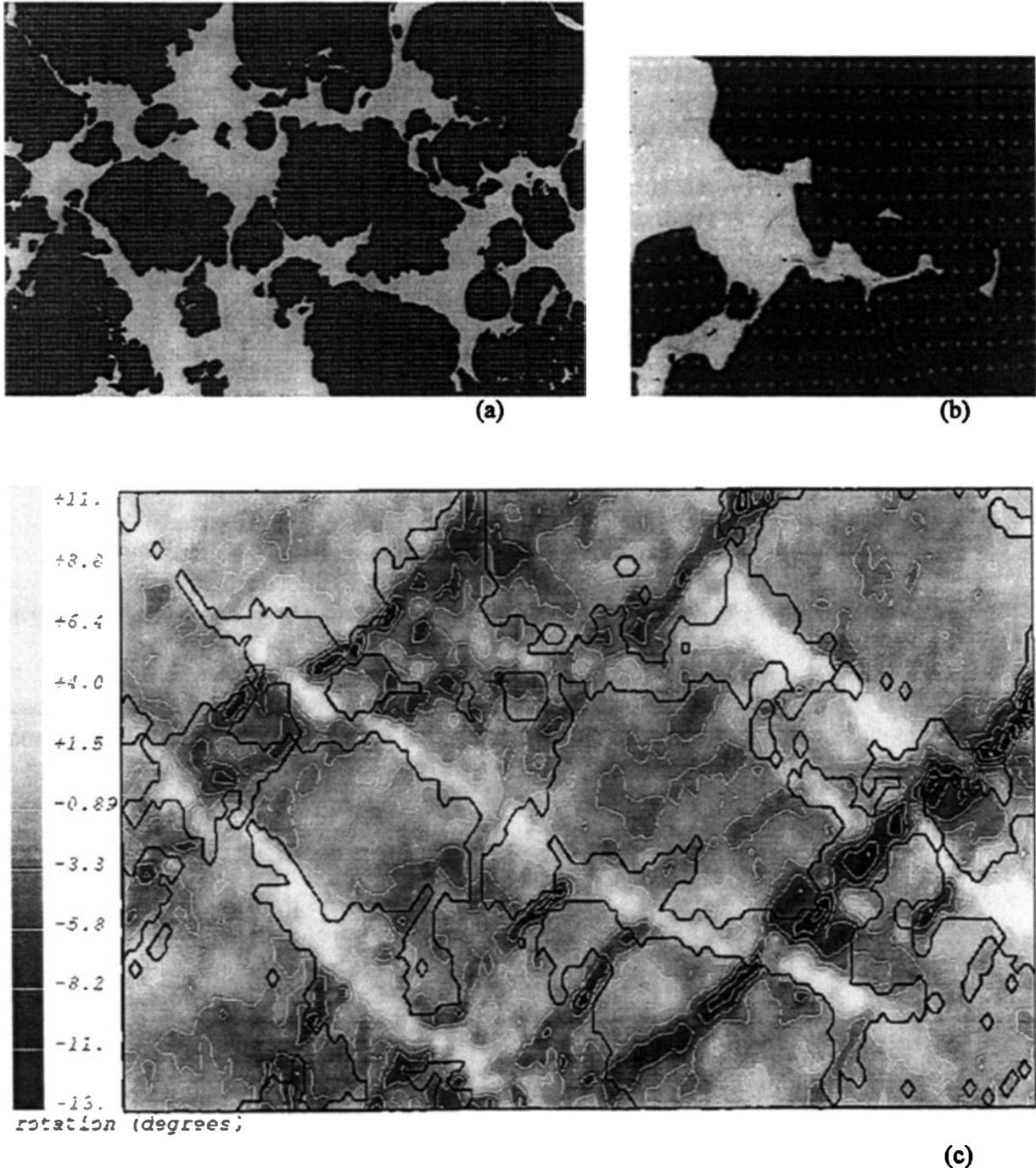


Fig. 4: Quantitative grid analysis technique. (a) Representative domain of an iron/silver two phase material before deformation. (b) Magnification of a subdomain after approximately 10% global strain; the distance between two grid dots is close to  $5\mu\text{m}$ . (c) Map of the in-plane rigid-body rotation angle: the strain localizes into shear bands. The displacements of about 10000 dots have been recorded.

- a fixed external basis is needed; the loading axis of the *in situ* deformation stages linked to the SEM chamber gives such a fixed basis.

## 4.2 The Microextensometry Technique

Prior to any mechanical test, the specimen is covered with a square microgrid made of gold lines or dots by means of a micro-electrolithographic technique that has been described elsewhere in detail [1]. The pitch of the grid has to be chosen so that it remains small in comparison to the smallest wavelength of the expected strain field; it may easily vary in the range 1 - 15  $\mu\text{m}$ . The grid covers an area close to  $2 \times 2 \text{ mm}^2$ .

The grids have different uses:

- they facilitate the identification during the *in situ* tests of the areas of interest;
- they make it possible to detect unambiguously the inception of micro-events induced by deformation [3,4];
- observed with an appropriate scanning mode in the SEM they form moiré patterns that give an enhanced view of weak strain heterogeneities, enabling the early detection of some events [3];
- they enable the qualitative and quantitative study of grain boundary sliding (Fig. 3);
- in association with efficient image processing techniques they give the in-plane components of the local displacement field over large domains representative of the microstructure [1]. Their derivation leads to specific components of the local total strain  $\epsilon$  and total rotation  $\omega$  tensors. The local strain heterogeneities can then be characterized by the average of these components over each phase as well as by distribution function. Different averages per phase or distribution functions can be calculated in order to; it is also possible to obtain contour plots of these components over the considered domain; the obtained maps give a powerful qualitative information on the actual strain localization modes during deformation (Fig. 4).

## 5. CONCLUSION

In order to feed and test physically based models for the elastoplastic behaviour of inhomogeneous materials, an experimental equipment has been developed. On the basis of a Scanning Electron Microscope, several complementary accessories give access to the essential parameters of the material and allow to follow their evolution with deformation.

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