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# KINEMATIC MEASUREMENTS OF 316L(N) POLYCRYSTALS TO IDENTIFY PARAMETERS OF CRYSTAL PLASTICITY LAW

A. Guery<sup>1,2</sup>, F. Hild<sup>1</sup>, F. Latourte<sup>2</sup> and S. Roux<sup>1</sup>

<sup>1</sup>LMT-Cachan, ENS Cachan / CNRS / UPMC / PRES UniverSud Paris, 61 avenue du Président Wilson, 94235 Cachan Cedex, France

<sup>2</sup>EDF R&D, Site des Renardières, avenue des Renardières - Ecuelles, 77818 Moret-sur-Loing, France  
[adrien.guery,francois.hild,stephane.roux]@lmt.ens-cachan.fr, felix.latourte@edf.fr

**ABSTRACT:** An identification procedure of parameters of a crystal plasticity law based on kinematic measurements is presented. First, the surface deposition of a random pattern to conduct Scanning Electron Microscope (SEM) imaging is performed. A Digital Image Correlation (DIC) method is then used to measure displacement fields that are compatible with the polycrystal geometry during an *in-situ* tensile test. These measurements are finally combined with a finite element simulation of the corresponding experiment that allows the identification of a set of parameters of a crystal plasticity law to be determined.

## 1. INTRODUCTION

Significant advances have been made in SEM imaging as well as in the modeling of materials at the microstructural scales during the last decades. As a consequence, small scale measurements can now be used to validate models describing the behavior of polycrystals. Crystal plasticity laws have been introduced to account for the physical features of plastic deformation at the grain scale by describing the activity of slip systems. The present study objective is to identify a set of parameters of a chosen law already validated for austenitic materials, proposed by Méric *et al.* [1]. The material chosen to validate this identification procedure is the 316L(N) austenitic stainless steel used in Pressurized Water Reactors (PWR) internals.

Kinematic fields measured via SEM imaging of a polycrystal surface during an *in-situ* tensile test provide a spatially dense experimental information for a given microstructure, which can be compared to a simulated response in order to identify or validate the model parameters. The measurements have to be accurate enough to capture strain localization patterns, despite the noise and artefacts of SEM acquisitions. DIC, which is commonly used at macroscopic scales, appears to be well-suited for these particular measurements. As DIC algorithms rely on local grayscale variations, a surface texture is needed. Thus, a speckle deposition technique was specifically developed in this work.

The aforementioned comparison between experimental measurements and finite element calculations requires a realistic information of the experimental microstructure. Electron BackScattered Diffraction (EBSD) acquisitions are used to characterize the microstructure at the surface of the sample, in terms of grain segmentation and orientations. These acquisitions are an additional information used to perform the identification step of the analysis.

## 2. DIGITAL IMAGE CORRELATION

A sequence of SEM images is acquired during an *in-situ* tensile test. The displacement fields are measured between two consecutive images with a finite element based DIC [2,3]. The displacement discretization is performed using an unstructured mesh, built from 3-noded triangular elements (about 20 pixel / 5  $\mu\text{m}$  sides), and created as conforming to the grain or twin boundaries as they are detected from EBSD (Figure 1). This property requires an EBSD acquisition of the surface of interest prior to the loading, and the determination of the transformation between EBSD and SEM coordinate systems. To treat the whole sequence of images, an incremental and updated scheme is adopted, namely, at the end of each iteration the nodal positions are updated with the measured displacement increment, and the deformed image becomes the reference image for the next step. The adopted DIC technique leads to a displacement uncertainty, as a root mean square (RMS) value, of 0.040 pixels (5.2 nm).

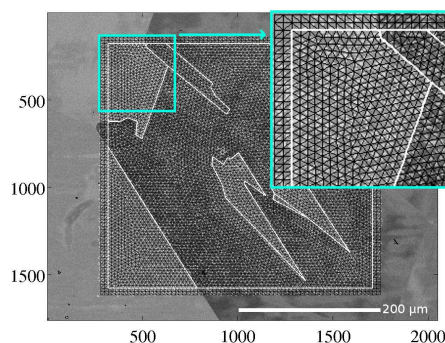
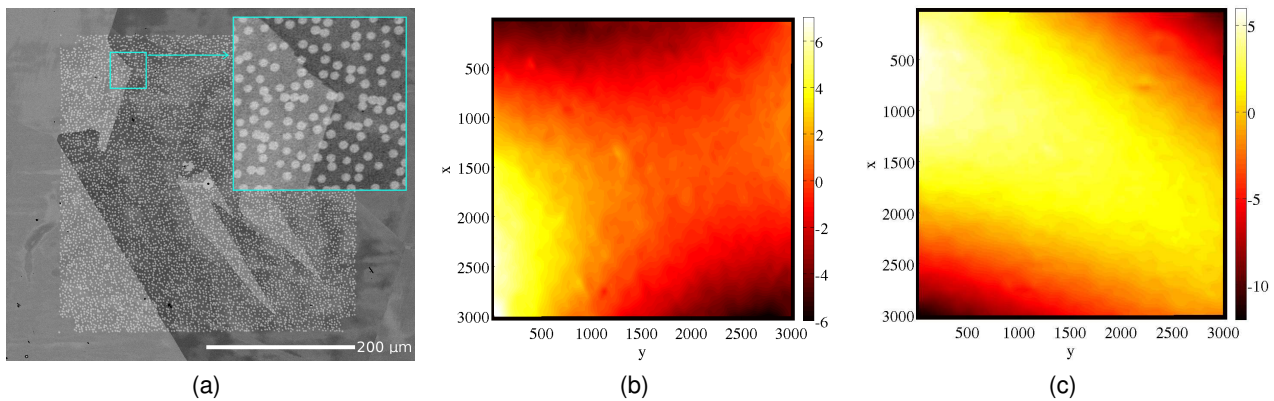


Figure 1 - Unstructured mesh compatible with the underlying microstructure for DIC analyses of the tensile test

### 3. COMPUTERIZED RANDOM SPECKLE FOR SEM IMAGING

Kinematic measurements with DIC require a grey level texture whose dynamic range is as large as possible with local contrast variations. If the natural texture of the material does not provide such contrast, different marking techniques can produce the suited artificial texture onto the surface of interest. In the present case, the raw surface imaged with secondary electron or backscattered electron detectors of a 316L(N) steel does not provide a satisfactory texture. In the context of SEM imaging, the deposition of microgrids by lithography is a viable technique [4]. An alternative solution is preferred in this study, which is based on a random pattern.

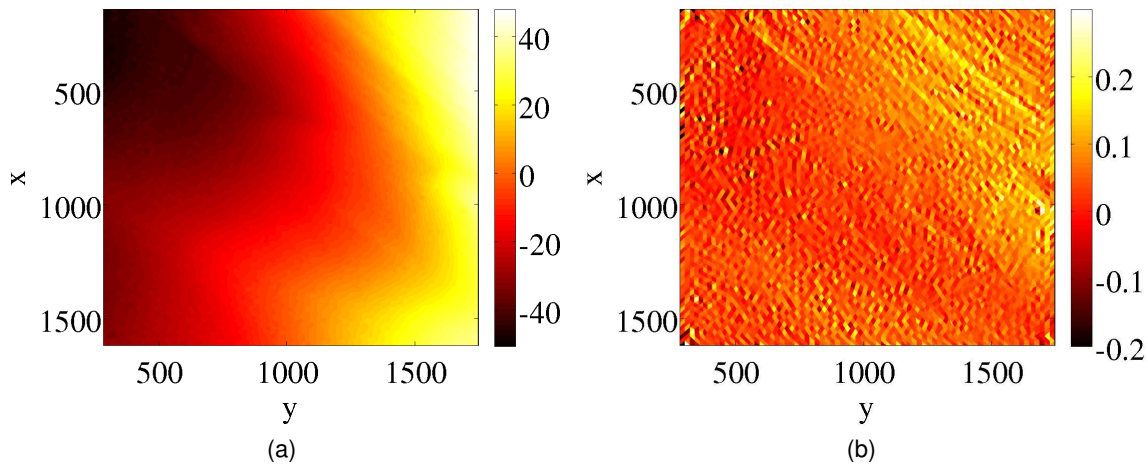
The pattern is generated from an known distribution of spots of random radius and position. This distribution is directly used to monitor the lithography process. One advantage of this technique is that the SEM image of the obtained speckle (Figure 2(a)) can be compared with the theoretical one, in order to measure the bias resulting both from lithography and image acquisition with the SEM (Figure 2(b) and 2(c)). This difference is quantified by a displacement field measured by DIC between the computer generated ideal image (as a Gaussian-shaped spots distribution), and the actual SEM image of the speckle. In this figure, the mean rigid body displacement has been removed to only consider the distortions field. One can observe that the bias is greater in the corners of the speckle, probably due to the electron beam deviation from the center of the speckle during the lithography or imaging process. The projection of this displacement field on a basis of shape functions taking into account some modelled distortions (radial, prismatic and decentering) leads to a residual displacement field whose RMS is 0.25 pixels (33 nm).



**Figure 2 - Backscattered Electron (BSE) image of the deposited speckle (a). Displacement field in pixels along the  $x$ -direction (b) and the  $y$ -direction (c) resulting from both lithography and SEM imaging. The physical size of one pixel is 0.13  $\mu m$**

### 4. RESULTS OF DISPLACEMENT FIELDS MEASUREMENTS

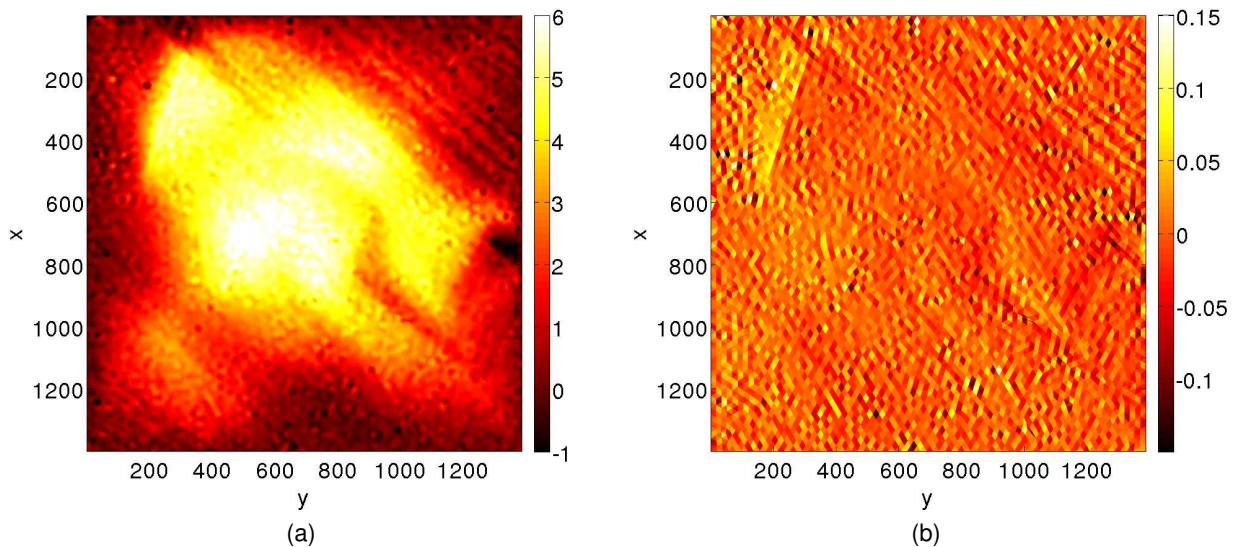
In Figure 3(a) and 3(b), one can see respectively the longitudinal displacement field measured for a macroscopic applied strain of 5%, and the corresponding longitudinal strain field. The DIC procedure is able to capture strain localization whose local level reaches about 20%, four times greater than the macroscopic applied strain.



**Figure 3 - Longitudinal displacement field in pixels (a) and longitudinal strain field (b) measured for macroscopic strain of 5%. The physical size of one pixel is 0.26  $\mu m$**

## 5. IDENTIFICATION OF PARAMETERS OF A CRYSTAL PLASTICITY MODEL

Simulations of the experimental tensile test are performed using the finite element package Code\_Aster and using a mesh built from that initially used in DIC analyses. The resulting mesh is a 6-noded elements extruded with one element through the thickness for a 3D calculation. This direct link between DIC and simulations allows the experimentally measured boundary conditions to be prescribed without interpolation. The crystal plasticity law [1] involves the plastic activity of single crystals slip systems in a viscoplastic constitutive equation. A total of 7 parameters, associated to the plastic flow and hardening, is chosen to be identified. The comparison of displacement fields measured by DIC and obtained by simulation leads to the identification of a set of parameters, using an inverse method adopted by Meuwissen *et al.* [5]. Each nodal displacement component at different time steps is taken into account in a least squares criterion. A Levenberg-Marquardt algorithm will be used to solve iteratively the minimization of this criterion. In Figure 4(a), one can see the difference between longitudinal displacement field obtained by finite element simulation with an initial set of parameters, and longitudinal displacement field measured by DIC, both for a macroscopic applied strain of 3% during the tensile test. In Figure 4(b), the difference between corresponding longitudinal strain fields is presented. The RMS of the longitudinal displacement fields difference is only 1.8 pixels ( $0.47 \mu\text{m}$ ) prior to identification. The implementation of the procedure is currently being worked upon.



**Figure 4 - Difference between fields measured by DIC and fields simulated by finite element with an initial value of the parameters, of longitudinal displacement in pixels (a) and longitudinal strain (b), for a macroscopic applied strain of 3%**

## 6. REFERENCES

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