Crystal deformation and rotation measurements in bainitic-ferritic steel

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Abstract:

An in-situ tensile test has been performed in an SEM on a microsample of 16MND5 steel, which is a bainiticferritic fine steel used in reactor pressure vessels. Platinum speckles with proper size have been deposited on the sample surface to allow for the measurement of surface kinematic fields via Digital Image Correlation (DIC). The association of EBSD and BSE images provides a microstructure free of tilt correction errors. The localization of crystal rotation and plastic strain are found to be clearly related in this experiment. **Key words:** crystal plasticity, rotation, strain localization, DIC

1. Introduction

The mechanical property of bainitic-ferritic steel with fine microstructure [1], which is used in reactor pressure vessels, is a key point for the safety of nuclear power plants, especially in accidental conditions. An in-situ tensile test is performed to study the crystal plasticity behavior of 16MND5.

2. Experiment

2.1 Experimental setup

A sample made of 16MND5 has been machined to a dog-bone shape, mechanically polished and finished with 50 nm colloidal silica suspension. A random speckle made of platinum disks is deposited on the region of interest [2]. The size of the patterns, 300 nm in diameter and 75 nm in height, has been chosen to both leave a trace of speckles on image quality (IQ) fields of EBSD analyses and not to perturb the indexing of crystallographic orientations of the underlying grains. A load is applied to the sample to reach a macroscopic strain of 6 %, and a series of backscattered electron (BSE) images are acquired. EBSD acquisitions are conducted for the initial and final states.

2.2 Analysis of experimental data

The sample is tilted to 70° for EBSD acquisitions and a tilt correction is necessary to adjust the shape of the grains. However, the automatic tilt correction of SEM revealed not precise enough [3] and a special effort has been dedicated to register BSE images, which are acquired in the horizontal position with little image distortion, with EBSD pictures.

Each speckle pattern is visible in IQ images of EBSD analyses. Yet it is doubled to two spots. One spot corresponds to the real position and the other one is related to its cast shadow. In order to register BSE and EBSD images, 'shadow spots' in EBSD images need to be erased or added in BSE images (Figure 1). Then standard DIC can be applied to correlate the two images. Consequently, a non-distorted microstructure of the ROI is obtained by combining the non-distorted coordinates provided by SEM images and the crystallographic orientations given by EBSD analyses. Figure 2 shows the grain boundaries extracted from EBSD pictures overlaid on BSE images. It is concluded that the grain boundaries coincide well with grain contrast in BSE images.



Figure 1. Speckle shown in IQ images (left) after tilt correction, raw BSE (middle) and transformed BSE

image where the shadow of each Pt spot has been added artificially (right)



Figure 2. Grain boundaries shown on BSE image, by association of BSE image and EBSD image.

The crystal rotation field between reference and deformed states is obtained by quaternion correlation on EBSD indexing results [4]. Displacement fields, and thus in-plane strain fields, are measured via DIC on BSE images. Since EBSD and BSE images are already registered, the rotation and the strain fields can be compared in the same coordinate system, as shown in Figure 3.



Figure 3. Crystal rotation (left) and strain along the tensile direction (right) between initial and final states. Grain boundaries are shown as green lines in (right). Significant and similar concentration is visible in both images.

3. Conclusion

The presented in-situ tensile test shows that BSE (or SE) images can be registered with EBSD images to obtain a non-distorted microstructure on a sample surface with proper platinum speckles. It has been demonstrated that a significant concentration of crystal rotation and plastic strain often develops in the same regions.

References

[1] B. Diawara, Caractérisation quantitative de la microstructure de l'acier 16MND5 des cuves de réacteurs nucléaires à eau pressurisée, PhD thesis, Grenoble INP, 2011.

[2] A. Guery, F. Latourte, F. Hild, and S. Roux. Characterization of SEM speckle pattern marking and imaging distortion by digital image correlation. Measurement Science and Technology, 25, 015401, 2013.

[3] G.Nolze, Image distortions in SEM and their influences on EBSD measurements. Ultramicroscopy, 107(2):172-183, 2007

[4] Q. Shi, F. Latourte, F. Hild, and S. Roux. Quaternion correlation for tracking crystal motions. Measurement Science and Technology, 27(9):095006, 2016.