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Phase transformation in AISI 304 stainless steel during in situ biaxial loading in SEM and with X-ray diffraction.

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Current global trend in various industrial fields is to reduce products dimensions. This miniaturization of systems is at the origin of new questioning about materials behavior at small scale. Actually the more the dimensions of an item are close from the order of magnitude of its microstructure, the more its response to some mechanical loading may differ from the macroscopic scale. The characterization of the effect of miniaturization is generally performed thanks to in situ uniaxial tensile tests [1]. However the probability for a material to undergo such a simple mechanical loading path during its service life is very low. It is then necessary to study materials response to different types of loadings more representative of reality.

The aim of this work is to perform in situ biaxial tensile tests on AISI 304 stainless steel in Scanning Electron Microscope (SEM) associated with X-Ray Diffraction (XRD) in order to study the material evolution (microstructure, plasticity, phase transformation, texture, damage) with a direct link to its mechanical properties during the whole loading.

This steel is austenitic at room temperature and shows a grain size of approximately 20µm. As there is currently no existing norm for biaxial testing, the first step of this study consisted in developing a specimen geometry permitting to obtain a real biaxial stress state at the centre of the specimen with incipient crack appearing at the center. Several specimen geometries were designed and modeled using finite element method. The most convenient geometries were machined in order to be tested on a macroscopic test bench fitted with a thermal imaging camera allowing to follow the strain evolution in the center of the sample during loading until crack appearance [2]. Two different specimen geometries showing a crack initiation at the center of the specimen were validated and machined at the microscopic scale in order to be used for in situ biaxial tests with a micromachine designed by MICROMECHA company.

The loading was operated in several steps; an EBSD cartography and SEM images were taken after each loading. Data obtained from these experiments permitted to study the evolution of crystallographic texture, grain morphology, grain rotation, intergranular misorientations, the appearance of shear bands as well as phase transformation.

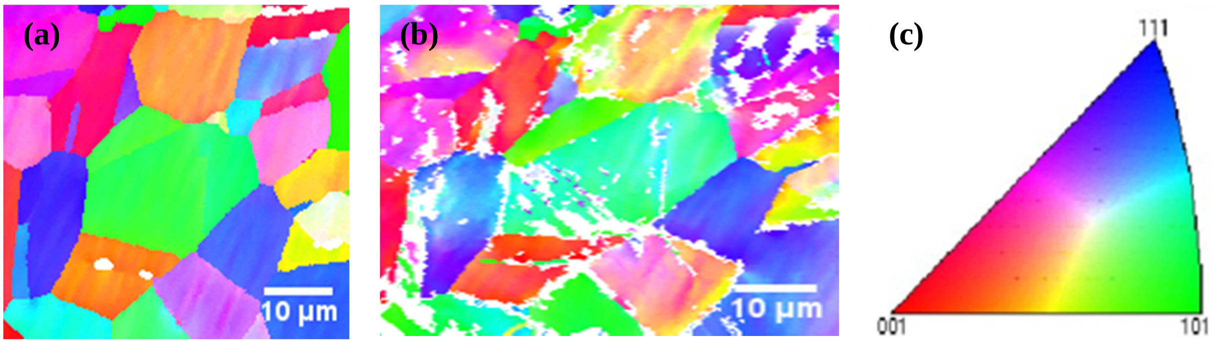

Figure 1: EBSD cartography: (a) at the initial state, before loading, (b) at the final loading step after the first crack appearance. The color legend is represented in the standard triangle in (c). (b) shows grain deformation, and intragranular misorientations compared to (a).

Focus was made on some specific grains to study in particular the influence of initial grain orientation on final misorientations (figure 1). At the end of the loading, some of the grains presented either shear bands or martensite variants, or for some grains both of these inelastic mechanisms. Combining SEM images and EBSD data we were able to identify the activated plasticity and phase transformation systems (figure 2). The tests performed with the XRD device allowed to follow and to quantify the evolution of martensite fraction during loading.

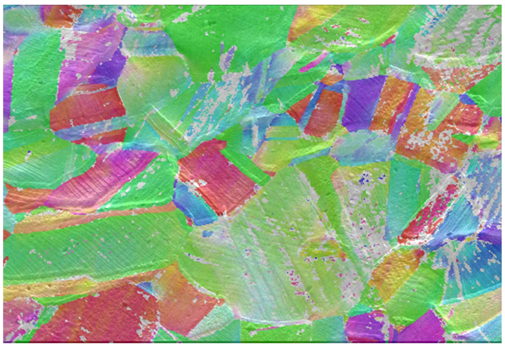

Figure 2:superimposition of SEM and EBSD images at the final loading step.

Finally, the same in situ biaxial tensile tests were also performed with both SEM and XRD, first at low temperature in order to study the influence of temperature on the material behaviour and especially on martensite formation, and then on the same steel that was heat treated in order to increase its grain size and to study the grain size effect.

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