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Microstructure development in a high-nickel austenitic stainless steel using EBSD during in situ tensile deformation

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ABSTRACT

Plastic deformation of surface grains has been observed by electron backscatter diffraction technique during in situ tensile testing of a high-nickel austenitic stainless steel. The evolution of low- and high-angle boundaries as well as the orientation changes within individual grains has been studied. The number of low-angle boundaries and their respective misorientation increases with increasing strain and some of them also evolve into high-angle boundaries leading to grain fragmentation. The annealing twin boundaries successively lose their integrity with increasing strain. The changes in individual grains are characterized by an increasing spread of orientations and by grains moving towards more stable orientations with ⟨111⟩ or ⟨001⟩ parallel to the tensile direction. No deformation twins were observed and deformation was assumed to be caused by dislocation slip only.

1. Introduction

Electron backscatter diffraction (EBSD) is a powerful tool for obtaining detailed information about the microstructural development during plastic deformation and is well suited for both qualitative and quantitative studies of deformation structures [1].

To further increase the understanding of the microstructural evolution upon plastic deformation in situ interrupted tensile test can be performed in the scanning electron microscope (SEM). This technique enables the same area to be investigated at different strain levels and the microstructure evolution of individual surface grains to be studied [2–5]. Using the EBSD technique detailed information on the evolution of the boundary network and changes in crystallographic orientation can be followed. The boundary network can be characterized by measuring the misorientation between neighboring data points. Low angle boundaries (LABs) give information on the evolving substructure inside individual grains and high angle boundaries (HABs) give information about changes of the grain structure such as formation of deformation twins and loss of integrity of annealing twins.

During plastic deformation by slip, regions of different orientations develop within the grains leading to grain subdivision and fragmentation which will here be studied using in situ testing and EBSD. If the orientation change is large, geometrically necessary dislocations are needed to accommodate this difference [6]. On a finer scale this subdivision is accomplished through the development of cells and subgrains and on a coarser scale by formation of deformation bands (DBs) [7]. DBs are parallel sequences of volume elements with alternating average lattice orientation [8]. The DBs are separated by deformation induced grain boundaries if the orientation change between the DBs is sharp, or by a transition band if the orientation change is more diffuse [7].

Early theoretical works on the lattice rotations in cubic crystals, for slip on {111}⟨110⟩ in tension, demonstrated that the deformation of single crystals can follow different orientation paths, with equal Taylor factor, for the same initial orientation. This leads to a mixed end-texture with ⟨111⟩ and ⟨100⟩ parallel to the load direction [9]. By means of in situ tensile testing the actual paths for crystallographic orientation changes, i.e. the texture evolution, in individual grains can be traced as a function of tensile load, and illustrated using inverse pole figures (IPFs) [10].

The relationships between SFE and alloying composition and between SFE, deformation mechanisms and mechanical properties for high-Mn austenitic steels have been studied previously [11–15]. The plasticity induced phase transformation and deformation twinning are responsible for the high strength and ductility in these steels. In high-Mn steels martensitic phase transformation takes place for SFE ≤ 20 mJ/m^2 and deformation twinning for SFE > 20 mJ/m^2 [11,12]. A steel with a SFE of 39 mJ/m^2 exhibited deformation twins in 25% of the grains at 0.1 true strain [15] and a steel with an estimated SFE of 63 mJ/m^2 showed extensive twinning at 0.3 true strain [14]. The same

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relationships for the austenitic Fe-Cr-Ni stainless steels have however not rendered the same attention.

To add to the knowledge the SFE for a set of high-purity austenitic stainless steels with 19 wt.% Cr and different Ni content in the range 12–31 wt.% was both measured and calculated. The SFE was found to increase with increasing nickel content from ~17 to ~30 mJ/m^2 [16]. In this article, we focus on the microstructure evolution during deformation of the alloy with the highest nickel content and highest SFE. By performing interrupted in situ tensile testing, in combination with EBSD measurements and forescatter detector (FSD) imaging, the deformation of individual surface grains are described and related to the mechanical properties. The aim of the study is to improve the understanding of the deformation mechanisms in the selected alloy with defined SFE, and to investigate if twinning, as indicated above, is an active deformation mechanism in this steel.

2. Experimental Details

2.1. Material Preparation

The chemical composition of the hot rolled, fully annealed, high-nickel austenitic stainless steel used in the study is given in Table 1. The SFE has previously been calculated and measured to 29.4 mJ/m^2 and 30.9 mJ/m^2 respectively [16]. The alloy was melted in a vacuum induction furnace and cast as 270 kg ingots. The as-cast ingot was hot forged to a dimension of 136 × 56 mm after soaking at a furnace temperature of 1250 °C. After forging, the material was quench-annealed; at a furnace temperature of 1200 °C, with a holding time of 30 min followed by water quenching. Hot rolling was performed down to a final thickness of 15 mm. The furnace temperature during hot rolling was 1210 °C. After the final rolling pass the material was quench-annealed again for 20 min at 1210 °C and quenched in water.

Tensile test samples, for both conventional and in situ experiments, were machined from the hot rolled plates in such a way that the tensile stress direction coincides with the rolling direction. The test samples for the conventional test were prepared with a round cross-section diameter of 5 mm and a parallel length of 60 mm.

For the in situ sample the plate was machined down to a thickness of 3 mm. The thin plate was then ground on sand stone to remove any surface defects from the machining operation, followed by standard metallographic procedures. The final shape of the sample, shown in the drawing in Fig. 1a was obtained by electrical discharge machining (EDM). After the EDM the sample was re-polished to remove any contaminations from the EDM process. A photo of the sample after deformation is shown in Fig. 1b. As can be seen by the shape changes the deformation was mainly concentrated to the middle part of the sample even though the material close to the hole became deformed in the process.

2.2. Conventional Tensile Test

Conventional tensile testing was performed using a Shimadzu AGG 100kN tensile test machine with a Hegewald & Peschke Inspect retrofit control system and an MFA 25 extensometer. The test was conducted to fracture. The test speed was 5 mm/min up to a strain of 1.2%, thereafter 20 mm/min. The extensometer was removed at a strain of 1.5% and after that, the cross-head displacement was used to determine the applied strain.

2.3. In Situ Tensile Test

For the in situ tensile test a Deben Microtest tensile stage fitted with a 5 kN load cell was used within the SEM chamber. A Deben Microtest acquisition software was used for control of the stage and for real-time display of the force-extension curve. The data sampling time and rate of displacement was set to 500 ms and 0.1 mm/min, respectively. Data for the applied force and for the extension were collected. The in situ tensile test was periodically interrupted for EBSD and strain measurements using the Vickers indentation marks made on the sample surface prior to the in situ test. The indentation marks were made along the tensile direction (TD), but outside the investigated area. Prior to EBSD measurements the sample was unloaded to half the tensile force, to ensure stable conditions during the EBSD acquisition.

2.4. EBSD Data Acquisition

The EBSD measurements were performed using a Zeiss Ultra 55 FEG-SEM equipped with an Oxford Instrument HKL Nordlys F EBSD detector. The EBSD data was acquired using the AZtec software from Oxford Instruments. The SEM- and EBSD settings, see Table 2, were all optimized to ensure accurate orientation mapping in combination with high speed of data acquisition. Good spatial resolution is vital for the study of deformed substructures and the ability to perform quick and repeated EBSD measurements of the same sampling area, while maintaining good electron backscatter Kikuchi diffraction pattern quality, is dependent of a high signal strength and speed of data acquisition.

2.5. Analytical Procedures for Data Cleaning and Boundary Definitions

2.5.1. Data Cleaning

Prior to the EBSD analysis a cautious data cleaning was performed. Isolated points which have been incorrectly indexed were replaced with copies of neighboring points. Unindexed points with at least 5 indexed neighbors were filled in by using copies of neighboring points. This step was repeated a maximum of three times. The correction was not allowed to increase the percentage indexed by > 2.5% for strains ≤ 23.6%, and by 4% above 23.6% strain. This corresponds well with the guidelines in the standard BS ISO 13067:2011 [17], which recommends that the percentage of indexed points should not be increased by > 5%.

2.5.2. Boundary Definitions

LABs are defined as having a misorientation in the range 2–10°, and HABs are defined as having a misorientation > 10°. The HABs which fulfills the requirement of having a misorientation of 60° about an ⟨111⟩ axis, within the allowed deviation of 5°, are defined as being twin boundaries (TBs).

3. Results and Discussion

3.1. Conventional Tensile Test

Conventional tensile tests were performed for two reasons. First, to get information about the strain hardening for the alloy in order to aid the planning of the in situ tensile test. Secondly, for comparison of the strain hardening obtained for the two techniques performed at different strain rates and shapes of the tensile samples.

Fig. 2 shows both the true stress-strain curves (σ for true stress and

Table 1
Chemical analysis of sample composition (wt.%).

Fe	Ni	Cr	Mn	Si	Al	C	N	S	P	Trace elements
49.8	31.41	18.57	0.03	0.02	0.013	0.006	0.006	0.005	0.003	0.137