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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 diffusive [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/m² and deformation twinning for SFE > 20 mJ/m² [11,12]. A steel with a SFE of 39 mJ/m² exhibited deformation twins in 25% of the grains at 0.1 true strain [15] and a steel with an estimated SFE of 63 mJ/m² showed extensive twinning at 0.3 true strain [14]. The same
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/m² [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/m² and 30.9 mJ/m² 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 1210 °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 retrofitted 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 ε for true strain).
The strain hardening can be divided into three stages prior to necking. Initially the rate of hardening drops dramatically up to ~2%. Thereafter follows a region, with a constant or only slightly decreasing strain hardening for strains in the range of ~2 – 10%. Finally, a region with a larger continuous decrease in the hardening rate up to a strain of ~30%.

The drop in the strain hardening rate, corresponding to the initial parabolic part of the stress-strain curve, can be attributed to the variation of the stress level needed to initiate plastic deformation in the individual grains [18]. The region with an almost constant strain hardening rate can either correspond to classical stage II hardening [19] or to the first region with constant strain hardening rate described for multiple-stage hardening [15,20]. According to the classical stage II hardening multiple slip systems are active and dislocation generation is responsible for the strain hardening. The latter alternative is described to also coincide with the onset of deformation twinning. Twinning was detected in an alloy with a SFE as high as 39 mJ/m² during this first region with constant hardening rate. As reported in Section 3.3.1, no deformation twins are however observed. The larger continuous decrease in the hardening rate for strain > 10% is concluded to be a consequence of thermally activated cross slip according to classical stage III hardening.

3.2. In Situ Tensile Test

Fig. 3a shows the in situ tensile test curve and data for the local technological strain measured at the microstructure level using indentation marks. The tensile test was successively interrupted, and the distance between the indentation marks along the TD were measured. The lower horizontal shows the extension collected from the in situ tensile stage and the upper axis the calculated strain. The small negative spikes in the curve are caused by the relaxation of the sample during stops. The larger negative spikes show the effect of the unloading of the sample that was made before the EBSD measurements. It should be noticed that the system is very stable without any apparent drift in the drawing force or extension during the un/re-loading. Indentation marks are used for strain calculations instead of extension data from the tensile stage. The deformation extended outside the length of the parallel reduced section of the sample, see Fig. 1b, which makes it difficult to use the extension data for strain calculations. The extension data also have to be corrected due to a slight misalignment of the sample during the in situ tensile test, giving rise to the s-shaped look at the beginning of the tensile curve. This is done by reducing the measured extension with a fixed value corresponding to the slack caused by the misalignment, see Fig. 3a.

In Fig. 3b, the tensile curves from the in situ and the conventional tests are compared. The curve for the in situ measurement is presented with two different x-axis; the lower shows the extension for the in situ tensile test and the upper shows the technological strain from the indentation measurements. Due to a bias in the measured force during the in situ tensile test, the stress level for the whole curve is shifted upwards to make the level comparable with the conventional tests. The shape of the curve from the in situ experiment coincides well with the curves from the conventional tensile test, despite the difference in shape of the two test samples and in strain rates. It is therefore reasonable to assume that also the strain hardening rate is comparable for the two different tensile test set-ups.

3.3. EBSD Measurements

3.3.1. Boundary Evolution

For each unloading of the in situ tensile test EBSD boundary maps displaying the evolution of boundaries with increasing strain are shown.
in Fig. 4a–f. Fig. 4g–l shows the corresponding FSD images, depicting the changes in surface topography during the deformation. The maps are all of equal size and show approximately the same area after each deformation step. The changes in appearance of the grains are directly related to the degree of deformation.

It can be noted in the EBSD maps that, as the deformation progresses, LABs (light blue) starts to form in the vicinity of grain boundaries or in some cases inside grains extending through the whole grain. The misorientation angle for some of the LABs increases with strain and as a result some LABs develops into HABs (dark blue) forming so-called deformation induced boundaries. The amount of LABs increase with increasing strain as can be seen in Fig. 4a–f. At 9.5% strain, planar arrays have started to form, and at 14.3% strain some LABs are also seen to subdivide parts of the grains. With increasing strain short segments of LABs are also gathering together forming broader structures. These tangled LAB regions can be described as representing transition bands, which intersects the grains and separates DBs, as well as regions with high density of geometrically necessary dislocations near some of the grain boundaries.

When comparing the grains labeled G1 and G2 in Fig. 4a with the corresponding FSD image in Fig. 4g it is clear that the deformation of these grains does not show up as LABs in the EBSD images even if it shows extensive formation of slip bands in the FSD images. Straight slip lines are visible in the FSD up to a strain of 9.5%, see Fig. 4g–h, thereafter more and more curved slip lines appears, see Fig. 4i–l, which is an indication of cross slip [18,21].

The annealing twins lose some of their integrity with increasing strain which can be seen in Fig. 4a–g by that parts of the red colored TBs are transformed into dark blue colored HABs. Furthermore, no deformation twins can be detected. The loss of the twin integrity is further investigated in Section 3.3.3.

A clear topography is seen in the FSD image already at a strain of 4.8% and it increases with increasing strain up to 14.3%. However, no significant differences can be seen in the surface topography with further increase of the strain. It is worthwhile to mention that the indexing during the EBSD measurements was surprisingly good in spite of the evolving topography; the zero solutions in the original maps (before data cleaning) are under 2% for the samples strained up to 19.0% and 3.3% and 6.0% for the samples strained to 23.6% and 27.5% respectively. The tangled LAB regions mirror the topography of the surface caused by the deformation seen on the FSD images.

There are some minor defects in terms of scratches in the investigated area shown in Fig. 4a–f. The defects are not present in the detailed area shown later in Figs. 6–7. The defects give an enhanced value for the measured length of the LABs. However, it is assumed that the effect can be viewed as a constant bias value throughout the whole deformation series.

Changes in the microstructure with applied strain as shown in Fig. 4 are presented quantitatively in Fig. 5. The changes of the occurrence of misorientation angles (normalized to the same number of pixels) are shown for the range 2–20° in Fig. 5a and for the angular region of 2°–10° in Fig. 5b. In Fig. 5c the evolution of HABs, TBs and LABs with strain is shown by using their respective boundary density (length of boundary/indexed area).

Misorientations in the LAB range (2–10°) increases with increasing strain for strains ≥ 9.5% as demonstrated in Fig. 5a and c. The LAB density follows an almost linear increase with increasing strain. The increase of the misorientations for lower angles can partly be attributed to formation of cells and subgrains and the increase of misorientations for higher angles can partly be attributed to development of DBs [22]. The number increase at misorientations in the LAB region in Fig. 5a is also accompanied by a shift towards larger misorientation angles with increasing strain. At strains ≥ 14.3% this shift includes formation of new boundaries with misorientation larger than 10°, HABs, seen in Fig. 4, which were not present in the undeformed sample. This shift towards higher misorientation angles with strain clearly demonstrates the evolution of LABs into HABs.

A correlation between the microstructure and the mechanical properties can be found by comparing the evolution of the LABs with the strain hardening curve (Fig. 2). The increase of both the number of misorientations and the misorientation angles starts at a strain of 9.5% which coincides with the transition from the second to the third stage in the strain hardening rate curve (Fig. 2). This shift is explained to mark the onset of cross slip [18], which enables the formation of subgrains.

The peak in the misorientation range 55–63° shown in Fig. 5b includes TBs. The peak decreases and broadens with increasing strain. This is due to the loss of TBs; the TB density decreases with increasing strain, as seen in Fig. 5c, as consequence of deformation of annealing.
twins and the lack of deformation twinning.

The HAB density shows a slight decrease up to a strain of 9.5% but thereafter an increase with increasing strain caused by formation of deformation induced HABs. The decrease up to 9.5% is most probably caused by new parts of grains entering and some leaving the analyzed area due to the shape change introduced by plastic deformation.

To follow the evolution of LABs, HABs and TBs by measuring their respective density is maybe the simplest and most straightforward way to describe the changes in the boundary network caused by the deformation. The deformation process is otherwise often described by reporting the changes in grain and subgrain sizes. However determining the grain and subgrain sizes in deformed samples unambiguously is challenging due to, e.g., difficulties with incomplete boundaries. Incomplete boundaries occur when the measured misorientation changes along the length of the boundary and fall below the defined grain boundary angle [17]. By using boundary densities the microstructure evolution can be described without having to rely on grain measurements based on the requirement that a grain should be completely enclosed by grain boundaries. But, as with grain size measurements the step size used has to be taken into account when comparing results from different measurements. One major advantage with using the boundary density is when reporting the twin content. The twin content is often presented as the percentage of HABs while the TB density is independent of the total length of HABs. Since deformation causes an increase of HAB, as can be seen in Fig. 5c, this will lead to a reduction in the percentage of TBs, even without an actual decrease in the amount of TBs. The TB density thus gives a more accurate value of the actual amount of existing TBs.

3.3.2. Subdivision of a Grain

A small area, marked with a rectangle in Fig. 4a, was mapped using a finer step size of 0.2 μm to obtain a higher spatial resolution. In Fig. 6 these maps are shown in IPF colors. The deformation can be followed by the various color changes, due to rotation of the crystallographic

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Fig. 4. EBSD boundary maps (a–f), acquired with a step size of 2 μm, and the corresponding FSD images (g–l). The strain level is indicated in the upper right corner of each map and image. In (a–f) the LABs are shown in light blue, the HABs in dark blue and the TBs in red. Unindexed points are colored black. The region highlighted with a black rectangle in (a) is also acquired with a step size of 0.2 μm (see Fig. 6). The TD is horizontal in all maps and images. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)
lattice, which occur in different parts of the grains with increasing strain.

The initial yellow grain in the lower right corner is subdivided into different regions of orange and pink due to the rotation of different parts of the grain in different directions. This subdivision leads to formation of DBs. The DBs are not sharply separated, but instead separated by regions of short segments of LABs forming a broader structure which may be regarded as transition bands.

The blue triangular grain in the left part of the maps displays that rotation in different directions in different parts of the grain can lead to grain fragmentation. The grain is partly subdivided by a LAB at a strain of 14.3%, which partly develops into a HAB at strains ≥ 23.6%. This boundary represents a so-called deformation induced grain boundary, which is starting to fragment the grain into separate grains. The deformation of this grain with increasing strain is shown separately in Fig. 7a–d, using coloring based on the calculated Taylor factor for the individual data points in the grain. This coloring shows how the propensity for slip is affected by the rotational changes within the grain. In the part of the grain with high Taylor factor, slip is more restricted because lower resolved shear stresses are acting on the slip systems. In

Fig. 5. Boundary misorientation evolution with increasing strain. The number of misorientations in the range 2–20° in (a) and 55–63° in (b). The boundary densities for HABs, TBs and LABs are shown in (c).

Fig. 6. IPF colored maps (a–e), acquired with a step size of 0.2 μm, showing the evolution of orientation changes in the grains with increasing strain. The strain level is indicated in the upper right corner of each map. LABs are shown in light blue, HABs in black and TBs in red. Unindexed points are colored white. The IPF coloring in (f) is used to display which crystal direction is parallel to the TD. The TD is horizontal in all maps.
the other part of the grain, with lower Taylor factor, slip is instead more favored. The Taylor factor differs greatly in different parts of the same grain; at strains ≥ 9.5% the Taylor factor varies from ~3.1 to ~3.6. However, the average Taylor factor in the grain only increases slightly with strain, from 3.45 in the undeformed grain, to 3.54 at a strain of 27.5%. The two evolving parts have clearly distinct glide properties, as viewed by the differences in their Taylor coloring (Fig. 7a–d), indicating that different combinations of slip systems are active in the different parts.

The misorientation for the evolving boundary in the middle of the grain increases with increasing strain, as visualized in Fig. 7e by using misorientation profiles (MOPs). The MOPs display the changes in orientation along a line drawn parallel to the TD in the center of the grain across the boundary. When the strain reaches 23.6%, a part of the boundary has even developed into a HAB.

3.3.3. Effect of Deformation on Annealing Twins

In order to follow the annealing TBs response to an increasing tensile strain, one area including annealing TBs were mapped, using a step length of 0.2 μm, see Fig. 8. The undeformed area in Fig. 8a consists mainly of a 〈101〉 texture component parallel to the TD, resulting in large resolved shear stresses on the slip systems. The twin in the center of the map is elongated in the TD with increasing strain, from 96 μm in the undeformed state to 117 μm at a strain of 27.5%. The largest misorientation changes are located in the inner part of this twin, caused by the rotation of 〈101〉 away from the TD. In the FSD image, in Fig. 8d, slip lines are clearly displayed. The slip lines intersect the TBs running along the TD, whereas they pass almost parallel to the TBs which are running at large angles to the TD.

The TBs are found to be varying in their ability to maintain their twin integrity with increasing strain. The TBs which run in directions at large angles relative to the TD maintain almost all of its twin integrity up to a strain of 14.3% and around half of it at a strain of 23.6% while the TBs running almost parallel to the TD has lost almost all of their twin integrity at a strain of 14.3%.

If the orientation of the annealing TBs remain such, that high resolved shear stresses acts on the intersecting slip systems, the TBs will lose their integrity. The loss is due to formation of dislocation pile ups and the generation of geometrically necessary dislocations near the boundary, which disturbs the twin relationship. In Fig. 8e LABs are seen to form parallel to the slip lines seen in the FSD image (Fig. 8d), indicating a high dislocation activity in the vicinity of the former annealing TBs. The loss of the TB integrity, as can be seen in Figs. 4 and 8, is the cause of the decrease and broadening of the HAB peak in the misorientation range 55°–63° (Fig. 5b) and the decrease in the TB density (Fig. 5c), with increasing strain.

Fig. 7. Subdivision of a grain with increasing strain illustrated by EBSD maps using Taylor coloring in (a–d) and by showing MOPs in (e). LABs are shown in light blue, HABs in black and TBs in red. Unindexed points are colored white. The MOPs are along a line parallel to the TD in the center of the grain. The TD is horizontal in all maps.

Fig. 8. Illustrations of the same area, containing annealing twins, at different strains using EBSD maps (a–c, e) and a FSD image (d). The IPF colored EBSD maps in (a–c) visualize the orientation change caused by increased strain, using the TD to display the orientation according to the coloring legend in (f). In (d) the orientation of slip lines are shown using a FSD image. In (e) the band contrast (an imaging quality factor) is used for the same EBSD data as in (b). All EBSD maps display HABs in black and TBs in red and in (e) LABs are is drawn in light blue. The strain level is indicated in the upper right corner of each figure and the TD is horizontal in all figures.
3.3.4. Grain Orientation and Misorientation Profiles

The orientation change in the grains with increasing strain can be followed by the change in their IPF coloring, see Fig. 9a–b. In the undeformed sample each grain is uniformly colored. With increasing strain, the coloring starts to vary between different parts of the grain. Overall, the orientation changes results in a double fiber texture with \(\{111\}\) and \(\{001\}\) parallel to the TD. The relative proportion of \(\{001\}\) being 0.32 at a strain of 27.5%. This fiber texture develops by rotation of grains away from \(\{101\}\) towards \(\{001\}\) or \(\{111\}\). This is clearly demonstrated by looking at the grain on the left edge of the maps in Fig. 9a–b. In the undeformed state the whole grain is green, which means that the TD is parallel to \(\{101\}\). With increasing strain one part turns bluish and one reddish, i.e. different parts of the grain rotate towards \(\{001\}\) or \(\{111\}\) with increasing strain.

The variation in the MOPs at different strains is shown in Fig. 9c for the grains marked G1–G4 in Fig. 9a. The MOPs, shown by white lines in the maps, are drawn from right to left into the middle of the grains parallel to the TD. The misorientation is given relative to the first point of the line. The distances, \(x\), are corrected for the technological strain, \(e\), using \(x_{\text{corr}} = x/(1 + e)\), to make it easier to follow the development of the substructure.

The misorientation along the lines in the undeformed grains is small. With increasing strain the misorientation increases. The greatest orientation change is seen in grain G3 and G4 where the misorientation reaches \(\sim 18^\circ\) at a distance of 100 \(\mu\)m from the grain boundary at a strain of 14.3%. The corresponding misorientation in grain G1 and G2 grain reaches \(\sim 7^\circ\). The more wavy shape of the MOP at higher strains reflects the developed substructure.

The initial large increase in the MOPs for the orange grain G4 (Fig. 9c) with increasing distance from the grain boundaries at strains \(\geq 9.5\%\), is not accompanied by formation of discernable LABs (Fig. 4). This shows that no well-ordered structures are being formed in the vicinity of the grain boundary. This grain shows the steepest increase in the MOPs near the grain boundary. The MOP starts at a junction between four grains, where all the other grains have the tensile texture stable orientation \(\{111\}\) parallel to the TD. The stable orientation of the neighboring grains causes the orange grain to take up a large proportion of the required shape change, imposed by the deformation, to meet the compatibility conditions. This shape change results in rotation of the crystal lattice which causes the sharp initial increase in the MOP. The misorientations can be ascribed to the accumulation of geometrically necessary dislocations which enables the maintenance of compatibility between neighboring grains during the deformation.

The orientation change in the four grains, marked G1–G4 in Fig. 9a, with increasing strain is visualized in Fig. 10 by means of IPFs where the tensile direction is displayed in the crystal coordinate system. The crystallographic orientation in the undeformed grains is homogeneous as can be seen by the dense distribution of the individual data points in the grains respective IPF in the left column of Fig. 10. The orientation change in the grains, caused by the deformation, can be followed by the increased spread and displacement of the data points representing the tensile direction. The displacement is accompanied by a change in the IPF coloring.

At a strain of 4.8%, the data points in G1 (pink) and G4 (orange) are still quite uniformly distributed, only more scattered, whereas in G2 (blue) and G3 (green) the data points are more unevenly spread. As the straining continues the grains begin to rotate. G1 (pink) and G3 (green) rotates towards \(\{111\}\), while G4 (orange) rotates towards \(\{001\}\). Initially, G2 (blue) rotates even closer to \(\{111\}\) but at a strain of 9.5% parts of the grain start to rotate away from \(\{111\}\), along the boundary line of the unit triangle connecting \(\{111\}\) and \(\{001\}\).

The orientation changes followed in the inverse pole figures (Fig. 10) are in agreement with the overview of early calculations of the rotation-orientation field, for tensile elongation of a fcc polycrystal.
given in the chapter written by Mecking in [23], and in [9].

G1 (pink) and G2 (blue) display a similar small overall increase in their MOPs, minor variations in their IPF coloring and a low content of LABs. The initial orientation of G2 (blue) represents a major component in the final tensile fiber texture. The grain deforms therefore mainly by slip on a few slip systems, which can be seen in the FSD images (Fig. 4g–l), in order to retain its initial orientation. Both the displacement away from the stable ⟨111⟩ direction and the small increase of the misorientation seen near the grain boundary (Fig. 9c) can be attributed to the requirement of compatibility. The stable orientation of G1 (pink) can be explained by its position in the IPF triangle (Fig. 10; near the boundary line connecting the ⟨111⟩ and ⟨001⟩ directions. At this position the shear stresses are equal for rotation towards ⟨111⟩ and ⟨001⟩ [16]. Small parts move towards either of the directions but the overall orientation change for the grain is small.

G4 (orange) rotates towards ⟨001⟩ and G3 (green) towards ⟨111⟩ contributing to the final double fiber texture dominated by ⟨111⟩. The overall large orientation difference between the grains interior and their border region after strains ≥ 9.5% results in the formation of LABs (Fig. 4).

4. Conclusions

The use of in situ tensile tests for studying deformation structures gives good opportunities to follow the evolution of individual boundaries and their changes in misorientation angles and to study the orientation changes within individual surface grains.

- For low strains only straight slip lines were observed in the FSD images and concurrently only a few LABs were formed. At strains ≥ 9.5% the number of LABs increases significantly and there is a tendency for the slip lines to become wavier. This behavior correlates well with the stress strain curve which displays an almost linear hardening up to a strain around 10%. Indicating that cross slip becomes significant first at higher strains.
- Both LABs and HABs densities increase with increasing deformation while TBs decreases. Different parts of a grain have in some cases been observed to rotate in different directions eventually leading to grain subdivision by LABs or even grain fragmentation by HABs.
- No deformation twins were observed and deformation was assumed to be caused by dislocation slip only. This is contrary to the high-Mn steels where deformation twinning is an active deformation mechanism in alloys with similar SFE.
- Annealing TBs lose their integrity due to accumulation of dislocations at and in the vicinity of the boundaries.
- The fiber texture developed during deformation is a mixture of ⟨001⟩ and ⟨111⟩ with the ratio of ⟨001⟩ being 0.32.

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References


