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EBSD analysis of surface and bulk microstructure evolution during interrupted tensile testing of a Fe-19Cr-12Ni alloy

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1. Introduction

The Fe-Cr-Ni ternary alloy system is the backbone of austenitic stainless steels and a chromium content of 18 wt% with varying concentrations of Ni are the most commonly used alloy compositions, e.g. 18/8, 18/10, etc.. One of the reasons for changing the Ni concentration is to alter the stacking fault energy (SFE) \cite{1,2}. The SFE in a set of high-purity austenitic stainless steels with 19 wt% Cr and 12–31 wt% Ni was evaluated by both measurements and calculations in \cite{2}. It was found that SFE increases with increasing Ni content for this set of alloys. One of these alloys is investigated in the present work and another alloy from that study has been investigated in a previous study \cite{3}. The SFE is a material parameter of crystalline materials, which depends on the chemical composition \cite{1} and temperature \cite{8}, and is a measure of the extent to which a perfect dislocation dissociates into partial dislocations and forms a stacking fault. Low values of the SFE promote formation of stacking faults which are essential for initiating both martensite formation and deformation twinning. Low values also retard recovery for which climb and cross-slip of perfect, un-dissociated dislocations are the basic mechanisms. With increasing SFE the dissociation of perfect dislocations is suppressed and the deformation is thus controlled by dislocation slip \cite{4}. According to one study made on high-Mn steels a SFE \(\leq 20 \text{mJ/m}^2\) favors martensitic transformation while SFE > 20 mJ/m\(^2\) favors deformation twinning \cite{9}, whereas another study on high-Mn steels concluded mechanical twinning to be dominating at SFE > 12 mJ/m\(^2\) \cite{10}. Tian et al. \cite{11} studied the deformation microstructure of ternary Fe-18Cr-(10-12)Ni alloys by EBSD. They found a gradual transition from deformation-induced martensite to deformation twinning for SFE from about 7 to 12 mJ/m\(^2\), as evaluated by first-principles calculations. Both martensite formation and deformation twinning are important for the mechanical properties such as high ductility and strength \cite{5}.

Twinning was concluded to be important for the four stage strain hardening behavior for low SFE fcc metals as described in detail in

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The later study includes the stainless steel 316 which has a similar Ni content and SFE as the Fe-19Cr-12Ni alloy in the present study. Primary twinning was observed during the first stage of constant hardening in the true strain range ~0.1–0.2 and the decreasing rate of twinning was explained to be the reason for the next stage of falling strain-hardening rate. The onset of extensive secondary twinning at a true strain of ~0.55, where new twins intersects existing primary twins, was then correlated to the start of the second stage with a constant strain-hardening rate.

Different explanations of the role of deformation twinning on the strain hardening have been proposed. One is based on the fact that deformation twinning introduces new twin boundaries (TBs) in the grains which increase the overall content of high angle boundaries (HABs). The effective grain size is thereby reduced leading to a hardening similar to the Hall-Petch relationship [14]. Another explanation comes from the Basinski mechanism [15] which proposes that glissile dislocations transform to sessile inside the twins and thereby contributes to strengthening. A third mechanism refers to a softening caused by twinning by reorientation of the lattice providing more favorable orientation for slip inside the twins [16]. The twinning strain itself makes a small contribution to the total elongation [17].

The SFE also affects the deformation by cross-slip, which is a thermally activated deformation mechanism leading to hardening, pattern formation and dynamic recovery [18]. During cross-slip un-dissociated screw dislocations change slip plane. This enables the release of dislocations which have been trapped by various obstacles in their initial screw dislocations change slip plane. This enables the release of dislocations transforming to sessile inside the twins and thereby contributes to strengthening. A third mechanism refers to a softening caused by twinning by reorientation of the lattice providing more favorable orientation for slip inside the twins [16]. The twinning strain itself makes a small contribution to the total elongation [17].

Most dislocations are generated by Frank Read sources which operate when a critical resolved shear stress is applied. The ratio between the shear modulus and the distance between the anchoring points of the dislocation line in the Frank Read source approximately determines the minimum stress needed to generate new dislocations [20]. A dislocation line terminating on a surface may act as a Frank Read source with twice the line length, lowering the critical resolved shear stress needed to generate new dislocations. Dislocation generation from surface sources requires only half of the stress needed for dislocation sources located in the interior of a single crystal [21]. A layer close to the surface of a single crystal will hence be more strain hardened than the interior because secondary slip systems will also be activated at lower stresses near the surface [22].

Electron backscatter diffraction (EBSD) has become an attractive tool for studying the microstructure evolution in metals. When using EBSD in situ during e.g. tensile testing it is the behavior of the surface grains which are followed, whereas the bulk grains are studied after conventional tensile tests. By performing both in situ and conventional tensile tests and evaluating the microstructure using EBSD results in a similar way, it is possible to study the underlying mechanisms for strain hardening of surface and bulk grains. This kind of comparison has not been done previously to our knowledge. The in situ deformation also makes it possible to follow the deformation of individual grains [3,23–26].

The present work focus on characterizing the surface and bulk microstructure evolution in a high-purity Fe-19Cr-12Ni alloy with a SFE of 17 mJ/m$^2$ during interrupted tensile testing. The aim is to enhance the knowledge of the deformation mechanisms and the relation between microstructure and the mechanical properties. The evolution of low- and high-angle boundaries as well as twinning and texture are investigated, which has not been well described by in situ tests for this important class of engineering alloys previously.

2. Experimental Details

2.1. Sample Description

The chemical composition of the hot rolled, fully annealed, medium-nickel austenitic stainless steel used in this study is given in Table 1. A detailed description of the manufacturing process of the alloy and the production of the tensile specimens are given in [3]. The SFE has previously [2] been measured to 16.9 mJ/m$^2$ for the medium-nickel alloy investigated in this study and to 30.9 mJ/m$^2$ for the high-nickel alloy used in a previous study [3] by means of transmission electron microscopy using weak beam dark field imaging technique and the isolated dislocation method.

2.2. In Situ Tensile Test

A Deben Microtest tensile stage, fitted with a 5 kN load cell, was used within the SEM chamber for the in situ tensile test. For control of the stage and for real-time display of the force-extension curve the Deben Microtest acquisition software was used. Data for the applied force and for the extension were collected. The data sampling time was set to 500 ms and the rate of displacement was set to 0.1 mm/min. The in situ tensile test was interrupted repeatedly for strain calculations and EBSD measurements. The strain was calculated by measuring the distance between Vickers hardness indentation marks on the sample surface made prior to the test. Before EBSD measurements the load was reduced by approximately 50% to ensure that no stress relaxation took place in the sample during the data acquisition.

2.3. Conventional Tensile Test

Interrupted conventional tensile tests were performed to investigate the deformation microstructure of the bulk. This enables comparison of deformation of a free surface, as studied during the in-situ tensile test, with deformation of the bulk. The conventional tensile test was also used to derive the strain-hardening rate for the alloy. The conventional tensile tests were performed using a Shimadzu AGG 100kN tensile test machine with a Hegewald & Peschke Inspekt retrofit control system and an MFA 25 extensometer. The test speed was 1 mm/min up to a strain of 1%, thereafter 5 mm/min. The extensometer was removed at a strain of 1.5% and then the cross-head displacement was used to determine the applied strain. The tests were conducted to 2, 5, 9, 14, 26 and 40% true strain ($\varepsilon$) respectively. Tensile tests were also carried out at a test speed of 5 mm/min up to a strain of 1.2% followed by 20 mm/min to fracture. The purpose for the tests with the faster speed was to obtain the same test condition as in the study of the high-nickel alloy [3].

2.4. EBSD Data Acquisition

The SEM- and EBSD settings used during data acquisition are summarized in Table 2. The EBSD maps were taken in a Zeiss Ultra 55 FEG-SEM equipped with a HKL Nordlys F EBSD detector using the AZtech software from Oxford Instruments for data processing.
2.5. Analytical Procedures for Data Cleaning, Boundary Definitions and Creation of Texture Component Subsets

Most of the post-processing of the EBSD data and the generation of maps and inverse pole figures (IPFs) were conducted using the Channel 5 software from Oxford Instruments.

2.5.1. Data Cleaning

A careful data cleaning was performed prior to the EBSD analysis. The cleaning included replacement of incorrectly indexed isolated points, and filling of unindexed point which had at least 5 indexed neighbors, with copies of neighboring points. The filling of unindexed points was repeated a maximum of three times. Less than 5% of the data points were unindexed for true strains ≤ 11% for the in situ tensile tests, and < 2% for the conventional tests for true strains ≤ 26%, when acquired with 2 μm step size. The cleaning was thus in accordance with the guideline in the standard ISO 13067:2011 [27], which recommends that the percentages indexed should not be increased by > 5%. This guideline was also followed for the sample acquired with a step size of 0.1 μm.

However, at a true strain of 18% for the in situ sample, and at a true strain of 40% for the conventional sample, the percentage of unindexed points was reduced from 11% to 4% and from 10% to 1.4% respectively for the maps acquired with a step size of 2 μm.

2.5.2. Boundary Definitions

A boundary is identified if the misorientation between two neighboring data points exceeds a defined minimum threshold misorientation. Boundaries in the range 2°–10° are defined as low-angle boundaries (LABs) and boundaries > 10° are defined as high-angle boundaries (HABs). Boundaries are identified as twin boundaries (TBs) if they fulfill the requirement of having a misorientation of 60° about an 〈111〉 axis, within an allowed deviation of 5°. The boundary density, which for any range of misorientations is the boundary length divided by the indexed area [28], is used to quantify the evolution of the LABs, HABs and TBs. However, for measuring the TB density for individual texture components, the boundary length is divided by the indexed subset area.

2.5.3. Texture Component Subsets

Subsets for fiber textures with the direction 〈001〉, 〈101〉 or 〈111〉 parallel to the tensile direction (TD) were obtained allowing a maximum deviation of 20°. For measuring the boundary density corresponding to each texture component, the subset masks were dilated by one pixel to include any surrounding boundaries.

3. Results

3.1. Stress-strain Behavior During In Situ and Conventional Tensile Tests

In Fig. 1 the tensile curves from the in situ and the conventional tensile tests are compared.

Fig. 1. Technological tensile curves. Comparison between the tensile curve for the in-situ test (solid grey line, both lower and upper horizontal axis) and the conventional tests performed at two different test speeds (solid and dotted black lines, upper horizontal axis).

The lower horizontal axis shows the extension for the whole sample during the in situ tensile test. The upper axis shows the technological strain for the in situ tensile test, based on measurements of the distance between indentation marks, and for the conventional tensile tests. The negative spikes in the in situ curve show the effect of the unloading.

The in situ tensile curve is corrected in two ways, as explained in detail in [3], for both a misalignment of the sample during the initial loading and due to a bias in the measured force.

There is a very good agreement of the hardening behavior between the in situ test and the conventional test performed at a final test speed of 5 mm/min despite the difference in shape of the two test samples. The strain-hardening rate is therefore assumed to be comparable for the two different tensile test set-ups.

Fig. 2 shows the strain-hardening rate (dσ/dε), where σ is the true stress, for samples from the conventional tensile tests performed at final speeds 5 and 20 mm/min. The corresponding curve for the alloy with high-nickel content is included for comparison.

Initially the rate of hardening drops dramatically up to ε ~ 2% for the medium-nickel alloy. Thereafter follows a continuously decreasing rate with an inflection point around ε ~ 20%, with only a minor decrease in the strain range 10–30%, called the inflection-point region. When the tests were performed at a final test speed of 20 mm/min necking occurs at ε ~ 41%, whereas no necking is noticed for the slower test speed. This is in agreement with that the Considère criterion for necking, i.e. σ = dσ/dε [29] is far from fulfilled when the test was interrupted. As can be seen in Fig. 2, the high-nickel alloy shows a markedly different evolution of the strain-hardening rate, described in [3], with a region of an almost constant strain-hardening, in the strain range ~2–10%, followed by a large decrease up to a strain of ~30%.

3.2. Evolution of the Deformation Boundary Structures

EBSD boundary maps for surface grains for a sample deformed by in situ tensile testing, are shown in Fig. 3. The maps are all of equal size, acquired using a step size of 2 μm, and show approximately the same area after each deformation step. A few LABs (light blue) are seen at ε = 2.3%. At ε = 7.2% broad planar arrays of short fragments of LABs are gathering together both in the vicinity of grain boundaries and...
extending into the grain interior causing subdivision of the grains. These LAB structures densify with increasing strain at the same time as new branches of the LAB networks are formed. At \( \varepsilon = 2.3\% \) parts of the annealing twins are starting to lose their twin integrity which can be followed by the color change from red TBs to dark blue HABs in the boundary maps. At the same strain level deformation twins are detected for the first time. As the strain increases more annealing twins loses their integrity while more deformation twins are formed.

EBSD boundary maps for bulk grains in three of the six samples deformed by conventional tensile tests, at a final test speed of 5 mm/min, are shown in Fig. 4. These maps are acquired using a step size of 2 \( \mu \)m and the area of the maps are of equal size. Because different samples are studied after each conventional tensile test it is not possible to follow the evolution of individual boundaries as during the in situ test. It is clear, however, that the boundary evolution resembles that from the in situ test with the difference that higher strain levels are needed to obtain similar deformation structures.

Broad planar arrays of LABs are seen in the sample deformed to a strain of 13.7% and most of the grains have a dense LAB network extending throughout the whole grains when strained to \( \varepsilon = 26\% \).

Some of the annealing twins have been deformed and have partly lost their twin integrity as shown in Fig. 4a for \( \varepsilon = 9.1\% \) but already observed at \( \varepsilon = 2\% \). Deformation twins are on the other hand first detected in the sample strained to \( \varepsilon = 9.1\% \). They are visible in the form of a few lonely pixels and only detected before the clean-up routine. The deformation twinning increases thereafter with increasing strain while the annealing twins continuously lose their integrity.

3.3. Boundary Densities

In Fig. 5 the evolution of boundary densities with increasing strain are shown for samples deformed by in situ (surface grains) and conventional tensile tests (bulk grains).

The surface grains initially show a slight increase of the LAB density up to a strain of \( \sim 4\% \) (Fig. 5a) followed by a markedly, almost linear, increase for larger strains. The HAB density (Fig. 5b) also shows a linear increase for strains > 5% but at a lower rate. For the TBs a minor decrease is noticed for strains up to \( \sim 8\% \) (Fig. 5b) due to loss of twin integrity for parts of the annealing twins. This loss continues but the TB density starts to increase at strains > 8% due to formation of deformation twins. The increase of the TB density is somewhat lower compared to the HAB density. The HAB density increases not only as a consequence of formation of deformation twins, but also as a consequence of formation of deformation induced HABs from LABs. One example of a deformation induced boundary is pointed out by arrows in Fig. 3d–f.

The increase of the boundary densities with increasing strain starts at higher strains in the bulk grains than for surface grains for all boundaries. The curve shape is though similar for both kind of grains. The major linear increase in the density of LABs (Fig. 5a) in the bulk grains starts at a strain \( \sim 10\% \) compared to at \( \sim 5\% \) strain in the surface grains. A small decline in the increase of LABs is seen after 26% strain. This is probably an effect of the data cleaning procedure. The sample...
strained to 40% contained 9.8% unindexed points which was reduced to 1.4% after cleaning. As the unindexed points are filled with copies of neighboring data points no LABs can be identified in these areas. The loss of twin integrity for parts of the annealing twins causes the TB density to decrease with strain up to ~14% where after it increases continuously due to deformation twinning (Fig. 5b).

In Fig. 5c the TB density in three texture components for the surface grains is illustrated. The TB density for \( \langle 101 \rangle \parallel TD \) decreases with strain up to a strain of ~8% due to loss of twin integrity for parts of the annealing twins. For larger strains a minor increase is observed due to deformation twinning (Fig. 5b).

The change of the occurrence of misorientations in the twin range, i.e., 55–63°, is illustrated in Fig. 6c–d for strains \( \leq 4.8\% \) and \( > 4.8\% \) respectively. The total occurrence of misorientations increases slightly with increasing strain up to 4.8% but the “twin peak” (here with a maximum at 59.5°) decreases and broadens. This is due to accumulation of dislocations at the TBs which alters the misorientation axis and angle relationship for the boundaries leading to loss of twin integrity for parts of the annealing twins. For strains \( > 4.8\% \) an increase with strain is seen of the “twin peak” in combination with a continuing broadening. The increase is due to a more extensive formation of new deformation twins than loss of twin integrity of both annealing twins and

3.4. Misorientation Evolution

The boundary misorientation evolution in surface grains with strain is illustrated in Fig. 6 for different misorientation ranges. The occurrence of misorientations, i.e. the number of data points that are in a particular bin, for the whole misorientation range 0–63° is shown in Fig. 6a for the undeformed sample and after straining to \( \varepsilon = 18\% \). The straining is seen to have the strongest influence on the boundaries with misorientations \(< 20°\) and \( > 50°\), whereas the boundary misorientations in the middle range, 20–50°, are almost unaffected. The evolution with strain of the boundaries in the misorientation range \(< 20°\) is seen in Fig. 6b. The occurrence of LABs is seen to increase with strain which reflects the formation of new LABs. The misorientation is also shifted towards larger angles with strain which causes some of the LABs to develop into new HABs, so called deformation induced boundaries, causing fragmentation of the grains (pointed out by yellow arrows in Fig. 3d–f).
deformation twins formed at lower strains. The occurrence of misorientations in the “twin peak” has increased by over 50% after deformation to \( \varepsilon = 18\% \) strain compared to the undeformed sample.

### 3.5. Deformation Twinning During In-situ Tensile Testing

Two sites, A and B, were selected for studying the evolution of deformation twinning using a step size of 0.1 \( \mu \)m. Site A was selected because it contained grains of different orientations. In Fig. 7a–c, a small part of Site A is shown before deformation and after straining to 2.3% and 4.9% strain. Site B, shown partly in Fig. 7d–e at strains 2.3%, 8.2% and 14.0% respectively, was selected based on two criteria. First, it contained a blue IPF colored grain oriented with \( \langle 111 \rangle \parallel TD \) which represents a major fiber texture component after tensile deformation. Second, it showed lines in the band contrast map, acquired with a step size of 2 \( \mu \)m, indicating the possibility of being signs of deformation twinning. Both sites show deformation twins at 2.3% strain, which was the smallest strain required to detect deformation twins when using a step size of 2 \( \mu \)m. The higher number of unindexed points (white areas) in the deformation twins at higher strains is due to the surface topography, formed by the deformation, shown in the forescatter detector images in Fig. 7g–h.

In Site A deformation twins were first detected in a green grain initially oriented with \( \langle 101 \rangle \parallel TD \). The twinned parts of the grain have rotated and are colored red which corresponds to an orientation with \( \langle 001 \rangle \parallel TD \). Other parts of the grain have rotated towards the stable orientation with \( \langle 111 \rangle \parallel TD \) which is represented by a blue color. After straining to 4.9% more deformation twins appears in this grain and the initial ones are seen to have grown both in length and width. Larger parts of the grains have continued to rotate towards \( \langle 111 \rangle \parallel TD \). After straining to 4.9% deformation twins also appears in the blue grain with \( \langle 111 \rangle \parallel TD \) and in pink parts of a grain with \( \langle 114 \rangle \parallel TD \).

In Site B, the deformation twinning in a blue grain with \( \langle 111 \rangle \parallel TD \) is followed. A few thin twins are detected at 2.3% strain. When the strain has reached 8.2% these twins are both lengthened and widened. New deformation twins, in the same direction, have also been formed, doubling the total amount of deformation twins in the grain. At 14.0% strain, deformation twins are formed in new directions, growing between the initial twins.

The neighboring grain to the right, initially colored blue-green, has rotated to bring \( \langle 111 \rangle \parallel TD \) and turned almost blue at 8.2% strain. The deformation twins, which initially started in the blue grain, seem to have extended into this neighboring grain, passing through the grain boundary, see Fig. 7e. At 14.0% strain, this is even more clearly shown,
see Fig. 7f and h. Deformation twins have also formed in parts of this grain not shown in Fig. 7. At 8.2% strain, twinning has also started in the upper green grain with $\langle 101 \rangle ||_{TD}$ and the twinning is seen to continue with increasing strain. The twinned parts in these grains are all oriented with $\langle 001 \rangle ||_{TD}$.

### 3.6. Texture Evolution

Fig. 8 illustrates the evolution of three texture components in surface grains with strain. Included in the figure is also the corresponding average Schmid factor for the slip system $\langle 111 \rangle \langle 1\text{-}1\text{0} \rangle$ for the texture components and for the complete data set. A high Schmid factor represents orientations for which deformation by slip of perfect dislocations is easy whereas a low Schmid factor denotes an orientation where slip is less favored.

The component $\langle 101 \rangle ||_{TD}$ decreases almost linear with increasing strain while $\langle 111 \rangle ||_{TD}$ increase continuously but levels off for strains > 8%. The texture component $\langle 001 \rangle ||_{TD}$ is almost constant for low strains but increases markedly and linear for strains > 7%. The texture evolution results in a double fiber texture with $\langle 111 \rangle$ and $\langle 001 \rangle$ parallel to the TD with the relative proportion of $\langle 001 \rangle$ equal to 0.41 at a true strain of 18.2%.

The increase of the component $\langle 111 \rangle ||_{TD}$ and the decrease of $\langle 101 \rangle ||_{TD}$ both contribute to lower the Schmid factor for the complete data set whereas the increase of $\langle 001 \rangle ||_{TD}$ increases the Schmid factor. In spite of the large changes in the distribution of texture components the average Schmid factor for the complete data set only show a slight decrease with strain.

Fig. 9 follows the orientation changes in one surface grain with increasing strain as shown at the top of each column. The changes are illustrated in the upper row by IPF coloring of the grain, displaying the tensile direction in the crystal coordinate system. The middle and lower row shows inverse pole figures where the orientation at individual data points in the grain is plotted and displayed in a color corresponding to the Schmid factor for the slip systems $\langle 111 \rangle \langle 1\text{-}1\text{0} \rangle$ and twinning systems $\langle 111 \rangle \langle 1\text{-}2\text{1} \rangle$ respectively. The middle row corresponds to slip of perfect dislocations whereas the lower row is for slip of partial dislocations during twinning.

The grain’s initial orientation $\langle 101 \rangle ||_{TD}$ is quite uniform as illustrated by the completely green colored grain in the upper row and the
dense data point distribution in the middle and lower left IPF. At 4.8% strain, inner parts of the grain have turned weak bluish and the data points in the IPFs have started to scatter as a result of rotations towards \(<111||TD\). The rotations have contributed to higher Schmid factors for the slip system \(<111\langle1\bar{1}0\rangle\) and thereby made slip easier in these parts. At 7.2% strain, twinning has occurred in the bluish part of the grain causing an orientation change to \(<001||TD\) of the twinned parts of the grain. As straining continues, larger parts of the grain rotate further towards \(<111||TD\). The twinning in turn increases the fraction of the texture component \(<001||TD\). According to the coloring in the IPFs, the rotation towards \(<111||TD\) lowers the Schmid factor for slip of perfect dislocations continuously whereas the Schmid factor for slip of partial dislocations continues to be high up to strains ~11%.

3.7. Martensite Formation

The detected amount of martensite, indexed as iron bcc, is low in all deformed samples, but an increase can be noticed with increasing strain. The amount of martensite before data cleaning is only 0.003% (a few pixels) in the undeformed sample but increases to 0.19% and 0.26% at a true strain of 27% after in situ and at a true strain of 26% after conventional tensile testing respectively, when measured using a step size of 2 μm.

Fig. 10 shows phase maps, acquired with a step size of 0.1 μm, where martensite (blue) can be detected both after conventional (a–d) and in situ tensile tests (e–f). Phase maps after conventional tensile tests to a true strain of 9% in (a) and 40% in (b) are magnified in (c) and (d) respectively. The martensite is detected at the TBs both within and outside the twins.

Fig. 8. The evolution of different texture components in surface grains with strain. The texture components are illustrated by solid lines and markers. The lines without markers represent the average Schmid factors for the texture components and for the complete data set.

Fig. 9. Orientation changes in a surface grain with increasing strain are illustrated by IPF-coloring of the grain in the upper row and by IPFs in the middle and lower row. The coloring of the IPFs is based on the Schmid factor for the slip systems \(<111\langle1\bar{1}0\rangle\), middle row, and the twinning systems \(<111\langle1\bar{2}1\rangle\), lower row. The color legends are shown at the end of each row, and the Schmid factor (0.27–0.5) scale is at the bottom of the figure.
Fig. 10e–f show the same area after straining to $\varepsilon = 8\%$ and to $\varepsilon = 18\%$ during the in situ tensile test. The martensite is seen to mainly nucleate in the vicinity of the deformation TBs and grow with increasing strain.

4. Discussion

4.1. Comparison of the Microstructure Evolution in Surface - and Bulk-grains With Increasing Strain

During the in situ tensile test, the behavior of surface grains is studied, whereas bulk grains are studied after the conventional tensile tests. The surface grains have a higher degree of freedom to adapt to the applied stress due to the absence of neighboring grains in one direction. This makes it easier for the surface grains to meet the constraint condition compared to the bulk grains, which are surrounded by other grains in all directions. This explains the observation that the formation of new boundaries starts at lower strains for the surface grains and is also consistent with the explanations for dislocation slip in single crystals [21,22] which states that the stress for dislocation generation at surface sources is half that for an interior source of equal length.

For initiation of deformation twinning a critical shear stress has to be reached [30]. According to our results this critical stress differs between surface- and bulk grains as the strain needed for twinning initiation in surface grains is only about half of what is needed for bulk grains. This is also consistent with easier dislocation generation at the surface [21,22].

4.2. The Relation Between Boundary Evolution, SFE and Strain-hardening Rate

In this study different contributions to the strain-hardening rate behavior in the inflection-point region in Fig. 2, characterized by only a minor decrease with strain, has been identified. The start of the significant increase of the LAB density with strain in the medium-nickel alloy (Fig. 5a) during conventional tensile tests coincides with the start of the inflection-point region which is close to 10% true strain (Fig. 2). A more moderate increase in the boundary density is observed at higher strains for HABs and at even higher strains for TBs (Fig. 5b). This indicates that the formation of the LAB substructure is a main cause for maintaining a high strain-hardening rate in this region.

As the SFE is quite low in the medium-nickel alloy, the dissociation of perfect dislocations into partials is favored, which in turn leads to a reduced ability for cross-slip. The reduced ability for cross-slip decreases the possibility for dynamic recovery by annihilation of dislocations which otherwise would lead to a reduced strain-hardening rate [31]. As a consequence of the low activity of cross-slip, the LAB density increases (Fig. 5a) which causes the strain-hardening rate to be maintained. This is in contrast to the behavior of the high-nickel alloy, with a SFE of $\sim 30 \text{ mJ/m}^2$, where cross-slip was concluded to cause the significant drop in the strain-hardening rate for strains > 10% [3] as shown in Fig. 2.

Deformation twinning is favored in the medium-nickel alloy due to the low SFE. Deformation twinning is first observed at low strains but an overall increase in the TB density (Fig. 4b) is not seen in the bulk.
grains until the strain level is larger than 14% due to the simultaneous loss of twin integrity for the annealing twins. Although parts of the annealing twins lose their twin integrity they still continue to be HABs and effective barriers to dislocation slip. However, the formation of both deformation twins and new HABs, created from LABs, represents new obstacles which act as effective barriers to dislocation slip. The mean free distance for dislocation slip is thereby reduced when the HAB density increases with strain. The Hall-Petch effect, as a result of a reduction of the effective grain size, may add to uphold the strain-hardening rate [14].

Another possible contribution to the sustained strain-hardening rate is from the Basinski mechanism [15] which states that glissile dislocations transforms to sessile inside the twins. This kind of inner structure in the deformation twins was however not possible to resolve in this study.

The increase in the HAB density, including the deformation twin boundaries, rise the need of more geometrically necessary dislocations to maintain the compatibility condition [32] which also adds to sustain the strain-hardening rate.

Although the content of martensite is very low it increases slightly with increasing strain. The transformation from austenite to martensite contributes to obstructing dislocation slip because of the formation of new phase boundaries. As martensite has a higher hardness than the austenitic matrix it will also necessitate the formation of geometrically necessary dislocations in the austenitic matrix and thus increase the hardening rate [32].

The formation of LABs and HABs with increasing strain is more extensive in the medium-nickel alloy compared to in the high-nickel alloy as illustrated by the boundary densities in Fig. 5. It is worth pointing out that the LAB density is significantly higher in the medium-nickel alloy. No deformation twins or martensite were detected in the high-nickel alloy during the in situ tensile test which was performed up to a true strain of 24% [3]. Only after conventional tensile test to fracture a small amount of deformation twins was detected.

4.3. Texture Evolution and Deformation Mechanisms

The first deformation twin is detected in a grain with \(\langle 111\rangle||TD\) and in these grains the most extensive twinning is observed. Even though this is a stable orientation during tensile direction of fcc metals and alloys, the grain has to deform to meet the constraint conditions from the neighboring grains. This is accomplished by deformation twinning, making use of the twinning system \(\langle 111\rangle\langle 1\bar{1}2\rangle\) instead. Even though the critical resolved shear stress needed for slip on \(\langle 111\rangle\langle 1\bar{1}2\rangle\) always is lower than for twinning for annealed fcc crystals, twinning may be possible after strain-hardening if the stress level is increased sufficiently [33]. The deformation of the green grains in Figs. 7a-c and 9 clearly shows contributions from both slip and twinning as deformation mechanisms. At lower stresses, deformation occurs by slip in the grain and at higher stresses, deformation by twinning is added.

The reorientation caused by twinning may lead to the mechanism called textural softening [16] if the orientation inside the twins enhances the probability to slip. This behavior contributes to lowering the strain-hardening rate. As can be seen from Fig. 8, it is the texture component \(\langle 001\rangle||TD\) which show the largest increases with strain and also the largest Schmid factor. A large Schmid factor indicates that deformation by slip is easy, and a large fraction of \(\langle 001\rangle||TD\) therfore contributes to textural softening of the strain-hardening rate. The reduced increase of the texture component \(\langle 111\rangle||TD\) at larger strain in Fig. 8 is a consequence of twinning causing the orientation to change to \(\langle 001\rangle||TD\) in the twinned parts.

The overall texture evolution after tensile deformation of both the present studied medium-Ni alloy and the previous tested high-Ni alloy results in a double fiber texture with \(\langle 001\rangle\) and \(\langle 111\rangle\) parallel to the TD. The relative proportion of the \(\langle 001\rangle\) component is however larger for the medium-Ni alloy; 0.41 compared to 0.31 at a true strain of ~18%. The higher ratio of the component \(\langle 001\rangle\) in the medium-Nickel alloy is concluded to be a result of deformation twinning.

5. Conclusions

The use of both in situ and conventional tensile tests to study the microstructure evolution in a pure Fe-19Cr-12Ni provided good opportunities to compare the deformation behavior in surface and bulk grains. The different microstructure features investigated contribute to the understanding of the deformation behavior in the important class of engineering austenitic stainless steels which have a similar SFE. The following important conclusions were drawn from this work:

- The evolution of the boundary structure in surface and bulk grains displays a strong resemblance but the strain needed to obtain similar deformation structures is lower for the surface grains.
- Deformation takes place by both dislocation slip and twinning. The contribution from both is clearly shown in grains with the initial orientation \(\langle 101\rangle||TD\). The most extensive twinning is observed in grains with the orientation \(\langle 111\rangle||TD\).
- The critical shear stress needed to initiate deformation twinning is lower for surface grains than for bulk grains. The strain needed for twin initiation in surface grains is only about half of what is needed for bulk grains.
- Textural softening caused by an increase in the texture component \(\langle 001\rangle||TD\) caused by twinning contributes to reduce the strain-hardening rate.
- Martensite is observed to nucleate in the vicinity of deformation twin boundaries and to grow with increasing strain. The amount of martensite is however very low and its contribution to the mechanical behavior is concluded to be of minor importance.
- The significant increase of the LAB density with increasing strain, caused by the reduced ability to cross slip and by deformation twinning (both due to the low SFE), is concluded to be the main reason for maintaining a high strain hardening rate up to strains close to necking.

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Data Availability

The raw/processed data required to reproduce these findings cannot be shared at this time as the data also forms part of an ongoing study.

References


