Effect of large plastic deformation on microstructure and mechanical properties of a TWIP steel

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Abstract. The effect of cold rolling on the microstructure evolution and mechanical properties of a cold rolled Fe-0.3C-17Mn-1.5Al TWIP steel was studied. The plate samples were cold rolled with reductions of 20, 40, 60 and 80%. The structural changes were associated with the development of deformation twinning and shear bands. The average spacing between twin boundaries in the transverse section of the rolled plates decreased from ~190 to 36 nm with an increase in the rolling reduction from 20 to 40%. Upon further rolling to 80% reduction the twin spacing remained at about 30 nm. The cold rolling resulted in significant increase in strength as revealed by tensile tests at an ambient temperature. The offset yield stress approached 1440 MPa, and the ultimate tensile strength increased to 1630 MPa after rolling reduction of 80%. Such significant strengthening was attributed to the development of specific structure consisting of deformation nanotwins with high dislocation density.

Keywords: cold rolling, strain hardening, mechanical twining, microshear bands, TWIP steel

1. Introduction

High-Mn austenitic steels exhibit the excellent combination of tensile strength and ductility owing to the formation of mechanical twins with nanometric thickness (twinning induced plasticity, TWIP - effect) and have a great potential in applications for automotive industry. The TWIP effect is attributed to the extensive mechanical twining. Superior ductility of TWIP steel is accompanied by a high resistance to necking due to the strong work hardening. The exact strengthening mechanisms, responsible for this high work hardening is a subject for discussion [1-4]. It is obvious that almost all strengthening mechanisms resulting in high rate of work hardening are operative due to the low stacking fault energy (SFE) in high-Mn austenite [1–4]. In the case of a low SFE, the deformation mechanisms change from slip of perfect dislocations to slip of partials, to mechanical twinning and eventually to transformation into hcp ε-martensite and/or bcc/bct α’-martensite. It is worth noting that dislocation slip provides the major contribution to the total strain. The operation of other deformation mechanisms gives the minor contribution to the overall plastic flow. The deformation twinning occurs in alloys with SFE > 18 mJ m² [1], while the formation of ε-martensite requires lower SFE values. Allain et al. [2] calculated that twinning can occur for a SFE between 12 and 35 mJ m², whereas ε-martensite can be formed for SFE values <18 mJ m². According to Rémy and Pineau [3] the minimum SFE that is required for mechanical twinning is 9 mJ m², while ε-martensite can be formed when the SFE does not exceed 12 mJ m². Taking into account the difficulties in determining the SFE and the different compositions of the alloys in these studies, the agreement between the literature data could be summarize as following. Extensive twinning is observed in steels with medium stacking fault energy (between 20 to 40 mJ/m² [5-7]), whereas γ→ε phase transformation takes place in alloys with SFE ≤ 20 mJ/m²[8,9].
The high strain-hardening is commonly attributed to the reduction of the dislocation mean free path with the increasing fraction of deformation twins or martensitic phase as boundaries of these structural elements are considered to be strong obstacles to dislocation glide \[10-13\]. Detailed investigation of microstructure evolution in these steels is critical to understand their strain-hardening mechanisms and mechanical properties. Thus the aim of the present work was to investigate the deformation microstructures developed in a Fe-0.3C-17Mn-1.5Al steel subjected to extensive cold rolling and their effect on mechanical properties.

2. Experimental

The TWIP steel used in this study had the chemical composition given in Table 1. An ingot of steel was subjected to solution treatment at 1150°C for 4 hours. The ingot was forged from 140 mm to 50 mm thickness in 3 passes and subsequently annealed at 1150°C for 4 hours. The forged steel was hot rolled at an initial temperature of 1150°C to 10 mm thickness and then annealed at the same temperature during 1 hour. The plate samples were cold rolled to 8, 6, 4 and 2 mm sheets (reductions of 20, 40, 60 and 80% respectively). Structural investigations were performed on the sections parallel to the rolling axis, using a Quanta 600 FEG scanning electron microscope equipped with an electron back scattering diffraction pattern analyzer incorporating an orientation imaging microscopy (OIM) system and a Jeol JEM–2100 transmission electron microscope (TEM). For structural characterization by the TEM thin foils of 3 mm diameter were cut out parallel to RD–ND plane and grinded to 0.1 mm thickness. Then the discs were polished using a double jet TENUPOL-5 electrolytic polisher at voltage of 20 V at room temperature. An electrolyte contained 10% perchloric acid and 90% acetic acid. The foils were examined using a JEOL JEM-2100 TEM operated at an acceleration voltage of 200 kV. Also the dislocation density was determined by analysis of X-Ray diffraction profiles. An ARL-Xtra diffractometer operated at 45 kV and 35 mA and diffraction profiles were collected using Cu Ka radiation. The value of the dislocation density \( \rho \) was calculated from the average values of the crystallite size \( D \) and microstrain \( \langle \varepsilon^2 \rangle \) by using the following relationship \[14\]:

\[
\rho = \frac{3\sqrt{2}\langle \varepsilon^2 \rangle}{nb}
\]

where \( b \) is the Burgers vector \( (b = a/\sqrt{2} \) for the FCC structure where \( a \) is the lattice parameter). The misorientations between the twins boundaries were analysed by the conventional TEM Kikuchi line method with a converged beam technique \[15\]. Tensile tests were carried out by using an Instron 5882 testing machine on specimens with a gauge section of 1.5 mm × 3 mm cut out parallel to the rolling plane.

|     | Mn | Al | Si | Cr | S  | P  | Fe  |
|-----|----|----|----|----|----|----|-----|
| 0.3 | 17.7 | 1.5 | 0.01 | 0.07 | 0.007 | 0.02 | bal. |

3 Results and discussion

3.1 Initial microstructure

The microstructure of Fe-0.3C-17Mn-1.5Al TWIP steel after initial thermo-mechanical treatment is shown in figure 1. The initial treatment resulted in the formation of uniform
microstructure composed of equiaxed grains with an average size of \( \sim 24 \ \mu m \). This microstructure consists of austenitic grains with a significant amount of annealing twins. The fraction of \( \Sigma 3 \) CSL boundaries comprises 40%.

### 3.2 Microstructure evolution

Deformation microstructures developed after a rolling reduction from 20 to 80% are shown in figure 2. Commonly, the cold rolling leads to noticeable increase of the dislocation density and extensive mechanical twinning in some favorably oriented grains. The deformation microstructure after a rolling reduction of 20% (figure 2a) is very heterogeneous and consists of grains with one primary twinning system only, grains with more than one twinning system (primary and secondary twinning systems), and grains containing no twins. Primary twins pass over initial grain, mostly, while secondary twins extend from one primary twin to the other.

![Figure 1](image)

Figure 1. OIM image of the initial microstructure of Fe-0.3C-17Mn-1.5Al TWIP steel
Figure 2. Microstructures of a Fe-0.3C-17Mn-1.5Al (wt.%) TWIP steel subjected to cold rolling at a reduction of (a) 20%, (b) 40%, (c) 60% and (d) 80%. RD indicates the rolling direction.

The deformation twins appear as bundles of thin and straight twins. Numerous dislocations exist in the matrix between the twins (figure 3a). It should be noted, that the matrix lattice orientation remains practically unchanged. The misorientation angle between points 1 and 3 in figure 3b was calculated to be 1°, while the misorientation angle between points 2 and 3 is about 55° that corresponds to the misorientation between the austenite matrix and twin lamellae. The dark-field image suggests that the deformation twins appear as bundles of thin and straight twins. The formation of ε-martensite phase occurs rarely at the twins boundaries and it has the form of thin plates, as shown by the dark-field image of ε-martensite. Schematic illustration of the location of diffraction spots from the austenitic matrix plane {111}, ε-martensite plane {100} and the twin plane {111} are also shown in figure 3. The average spacing between twin boundaries is about 190 nm. It should be noted that the dislocation density rapidly increases to about $1.5 \times 10^{15}$ m$^{-2}$. Thus, after 20% rolling reduction extensive twinning and the formation of ε-martensite provides subdivision of initial grains to lamellae that highly facilitates accumulation of lattice dislocations.
Figure 3. TEM images of microstructure developed after 20% of cold rolling: (a) and (b) BF-images, and (c) diffraction pattern. Arrows show the dark-field images of ε-martensite and deformation twins.

At a reduction of 40%, a high density of deformation twins is observed, and narrow band-like regions of extensive mechanical twinning could be distinctly distinguished (figure 2b) [16, 17]. The amount of deformation twins increased substantially. In the most of grains the deformation twins belonging to several systems were found. Up to three activated twinning systems were observed in numerous grains, and primary twinning systems are highly in dominant. The average spacing between twin boundaries in the transverse section significantly decreases. The distance between twins becomes 36 nm. The deformation twinning as one of the main deformation mechanisms leads to the development of nanoscale layered structures because of the small thickness and spacing of the twins. The deformation twins tend to rearrange along the rolling plane (figure 4a). Figure 4b reveals the wavy bands that appeared owing to the formation of shear bands. The separate shear band passes over a grain and shears the previously formed deformation twins (figure 4b). Note here that the multiple deformation twining results in the development of complicated microstructures composed of frequently intersected twins belonging to different twinning systems (figure 4a, 4c). An increase in the rolling reduction from 20 to 40% is accompanied by a gradual increase in the dislocation density from about 1.5×10^{15} m^{-2} to 2×10^{15} m^{-2}. Thus, the increasing
rolling reduction from 20 to 40% leads to a strong increase in twin density and minor increase in lattice dislocation density. The formation of shear bands starts to occur.

![TEM images](image)

**Figure 4.** TEM images of microstructure developed after 40% of cold rolling: (a), (b) and (c) BF-images, (d) DF-image of deformation twins with diffraction pattern.

Further rolling induces the formation of numerous shear bands, which pass over a grain and shear the previously formed deformation twins (figure 2c). No significant increase in density of twins and lattice dislocations was found. Most of the grains (around 80%) contain the twins with different primary and secondary twinning systems activated. The increasing of the rolling reduction from 40 to 60% leads to an increase of the number of shear bands and their thickness, which in turn leads to the development of narrow regions of localized shear (figure 5a). The shear bands are composed of largely misoriented nanocrystallites. The dislocation density increases to about $2.5 \times 10^{15} \text{ m}^{-2}$. The grains without deformation twins contain a high density of planar dislocation structures (figure 5b). It is evident that the amount of twins increases with increasing the cold strain before the rolling reduction reaches 60%, which suggests that the mechanical twinning is active during the cold rolling at small to intermediate reductions. The previously formed twin boundaries tend to rearrange towards the rolling direction.

At large strains (reduction of 80%) the number of shear bands increases noticeably, and the respective shear bands tend to be subdivided to nanoscale crystallites (figure 2d). With increasing strain, the lamellar twin-matrix structure progressively aligns with the rolling plane as a consequence of dislocation slip in both matrix and twins. The TEM
observations show a band-like lamellar structure (figure 6a). Some of these lamellae contain twins. The fine structure of the twin lamellae is shown in detail figure 6b. Here, the [2 1 1] and the [3 2 3] are the zone axes for austenite matrix and deformation twins. The misorientation between the austenite matrix (point 2) and the twin (point 3) in figure 6b was calculated to be about 60°. It should be noted that rather large internal distortions are involved in the deformation fine structure. The crystal rotation within a matrix, i.e., the misorientation angle between point 4 and point 3 in the figure 6b, comprises about 4°. In contrast to sharp twin boundary TEM images at small rolling strains, the image of the twin boundaries on TEM micrographs become diffuse when the cold reduction reached 80% (cf. figure 5b and 6b). This also suggests that the severely strained fine structures are characterized by large lattice distortions, which can be attributed to the high dislocation densities which increases to $5 \times 10^{15}$ m$^{-2}$.

![Figure 5](image)

**Figure 5.** TEM image of microstructure developed after 60% of cold rolling: (a) and (b) BF-image; (c) DF-image of deformation twins with diffraction pattern.
Figure 6. TEM image of microstructure developed after 80% of cold rolling: (a) BF-image; (b) BF-image with diffraction pattern of deformation twins and austenitic matrix.
Figure 7. The effect of rolling reduction on the twin spacing and dislocation density.

Figure 7 shows the strain effect on the twin spacing and dislocation density. The twin spacing decreases to about 20 nm during rolling to 60% reduction. Then the twin density does not vary significantly upon further rolling. In contrast, the dislocation density increases through all rolling process and finally approaches $5 \times 10^{15}$ m$^{-2}$ after 80% rolling reduction.

3.3 Mechanical properties

A representative series of engineering stress-strain curves obtained by tensile tests at an ambient temperature is shown in figure 7. In the initial annealed condition, the Fe-0.3C-17Mn-1.5Al steel is characterized by elongation of 96% and relatively low offset yield stress of 240 MPa. Extensive strain hardening takes place up to failure providing exceptionally high necking resistance and, therefore, very high ductility. The cold rolling leads to significant strengthening of the steel. The mean yield stress of 840 MPa is achieved in the specimens after rolling at 20% reduction. In this case, the sample exhibits apparent steady state flow resulting in rather large uniform elongation above 20%. An increase in the cold rolling reduction results in further progressive increase in the yield stress. It should be noted that the samples subjected to the cold rolling with 40-80% reductions show well-defined peak stress on the tensile flow curve. The strengthening by cold rolling is accompanied by a degradation of plasticity owing to facilitating plastic instability. An increase in the rolling reduction to 80% increases the yield stress to 1440 MPa, and the ultimate tensile strength approached 1630 MPa, while the total elongation decreases to approx. 5%. It is obvious that the strengthening can be associated with the grain boundary strengthening, i.e. decrease in the twin spacing, and the dislocation strengthening. These both strengthening mechanisms are probably responsible for the yield stress increase after cold rolling at relatively small reductions. The twin spacing does not change at large rolling reductions, whereas the dislocation density increases during the rolling even in the range of large reductions of 60-80%. Therefore, the increase in the dislocation density is considered as a main contributor to yield stress for the samples subjected to cold rolling with large reductions above approx. 50%.
4. Summary

The microstructure evolution and their effect on the mechanical properties of a cold rolled Fe-0.3C-17Mn-1.5Al TWIP steel were studied. Cold rolling with a reduction of 20% brings about high dislocation density and the formation of numerous deformation twins belonging to primary and secondary twinning system. Also, the $\gamma \rightarrow \epsilon$ phase transformation was observed mainly on the deformation twin boundaries after a reduction of 20%. The dislocation density increases rapidly with strain. Further cold rolling with reductions above 40% leads to the formation of microshear bands, i.e., narrow sheet-like regions of concentrated plastic deformation. The deformation microstructure evolved after a rolling reduction of 80% consists of separate micron scale crystallites delimited by shear bands which are subdivided to nanoscale crystallites bounded by twin boundaries. The cold rolling results in significant strengthening of processed steel, which is accompanied by a remarkable degradation of plasticity. The offset yield strength increases from 840 MPa to 1440 MPa when the rolling reduction increases from 20 to 80%. Correspondingly, the elongation decreases from 29% to 5%.

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