Grain Boundary Engineering of Medium Mn TWIP Steels: A Novel Method to Enhance the Mechanical Properties

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The grain boundary engineering (GBE) approach was employed on a medium manganese (Mn) based twinning induced plasticity (TWIP) steel to improve its mechanical properties. Two specially designed thermo-mechanical processing (TMP) routes, one involving unidirectional rolling (UDR) and the other one employing multi-step cross rolling (MSCR), with intermediate short term annealing treatment have been used. The annealing temperatures are chosen considering recrystallization and grain growth. Of the two routes, the one involving MSCR, has shown a higher fraction of special or coincident lattice site (CSL) grain boundaries. A detailed grain boundary microstructural analysis has been carried out by electron backscatter diffraction (EBSD) for differently processed samples. A significant improvement in ductility is observed in MSCR processed samples due to increase of CSL boundary fraction.

KEY WORDS: thermo-mechanical processing; grain boundary engineering; microstructure; texture; mechanical properties.

1. Introduction

The interest in studying Mn based steels, twinning induced plasticity (TWIP) steels and transformation induced plasticity (TRIP) steels has increased in the past few decades because of the good combination of strength, ductility and formability that they offer, which is much suited to automobile applications.1–6) Even though these steels have a good mix of mechanical properties, they are not used extensively due to drawbacks such as delayed cracking, issues pertaining to melting and homogenization with manganese addition and hydrogen embrittlement. These shortcomings can be overcome by reducing the amount of Mn and adding other suitable elements. However, a decrease in Mn content affects ductility and other mechanical properties. Grain Boundary Engineering (GBE) is a possible way to compensate for the trade off in properties due to Mn addition.

It is well known that GBE is an effective approach to suitably tailor properties such as ductility, stress corrosion and intergranular cracking, creep and other functional properties of polycrystalline materials.7–13) In this approach, the above mentioned properties can be improved by changing the grain boundary character distribution (GBCD), specially by increasing the fraction of Coincident site lattice (CSL) boundaries in the overall grain boundary character distribution. Watanabe and co-workers14–16) introduced the concept of GBE in polycrystalline materials by thermo-mechanical processing. Time, temperature and accumulated strain during processing are the main parameters that control the grain size, shape and boundary microstructures. Although there is no thumb rule to tailor suitable grain boundary character distribution; it can be achieved by suitable iterative thermo-mechanical methods. Iterative strain and controlled annealing are the process parameters that increase Σ3 boundaries in steels and other materials with face-centred cubic (FCC) structures.17–19) The formation of Σ3 and other related boundaries have been previously reported by many authors in austenitic stainless steel and other FCC materials.21–23)

Deformation twinning and associated strain hardening is the most important phenomenon that makes TWIP steels attractive as structural materials. The nucleation and growth of deformation twins is anisotropic and is controlled by dislocation substructures.24,25) In addition, activation of multiple slip systems and dislocation pile-ups determine the nucleation, propagation and growth of deformation twins. Beladi et al.26) reported that deformation twins are mostly nucleated at grain boundaries and propagate through the grains. As grain boundaries are anisotropic in nature, hence the formation of deformation twins also depends on the character and plane orientation of these boundaries.27,28) In this way, the character and distribution of grain boundaries may contribute to extended mechanical twinning during deformation in TWIP steels.

The present investigation deals with several important issues concerning character and plane distributions of grain boundaries, size and texture of grains, as applied to a medium Mn TWIP steel. Through this investigation, an attempt has been made to understand the effect of strain path and annealing temperature on GBCD and texture evolution. Two different critical rolling paths and two different temperatures have been introduced to the thermo-mechanical schedule to increase the fraction of CSL boundaries. Further, the so-obtained combination of GBCD and texture has
been examined for their influence on mechanical properties.

2. Experimental Procedure

2.1. Materials and Processing

A medium Mn TWIP steel with the nominal composition Fe-12Mn-0.5C (wt%) was used for the present investigation. The material was prepared by arc melting under argon atmosphere. To break the solidification microstructure, the sample was cold cross-rolled up to ~50% reduction in thickness and then annealed at 800°C for 15 min to get a completely recrystallized microstructure. This was followed by the actual thermo-mechanical processing intended for grain boundary engineering, involving two different rolling paths: (i) unidirectional rolling and (ii) multi-step cross rolling (Fig. 1). In each of the rolling procedures, 5% thickness reduction per pass was given followed by annealing at 750°C (recrystallization temperature) and 850°C (grain growth temperature) for 5 min each. The process was continued up to 50% thickness reduction with annealing at 750°C and 850°C for 5 min each in between. Finally, the samples were annealed at each of the above temperatures for 15 minutes. A summary of the thermo-mechanical processing schedule is presented in Table 1.

2.2. Microstructural Characterization

The microstructural characterization of recrystallized and processed samples was carried out using a field emission scanning electron microscope (FESEM) (FEI Sirion model) operated at 25 kV and equipped with an Electron Back Scatter Diffraction (EBSD) system. Data acquisition and analyses were carried out using the TSL-OIM™ software. EBSD was performed to obtain the size, shape and microtexture of the grains and character distribution of the grain boundaries. The reconstructed grain boundary line segments were extracted from the TSL software. In this procedure, more than 200 000 line segments per sample were used to calculate the GBCD. Grain boundaries with $1 \leq \Sigma \leq 29$ CSLs were considered for calculation following the restriction imparted by Brandon’s criterion. The size of the scan step was kept constant (200 nm) for all samples. Microstructural analyses were performed on the longitudinal plane normal to transverse direction (TD). The grain size distribution was fitted with the log-normal size distribution function to obtain mean and variance. The distribution function can be written as:

$$f(x) = \frac{1}{(2\pi \sigma_m^2)^{1/2}} \exp\left[-\ln(x/m)\right]^{2/2\sigma_m^2}$$

where, $m$ and $\sigma$ are the median and variance respectively. EBSD generated inverse pole figure map (IPF) and grain boundary microstructure of the starting material are shown in Fig. 2.

2.3. Tensile Testing

The mechanical properties of starting materials and thermo-mechanically processed samples were evaluated by tensile testing. Flat, dog bone-shaped specimens (gauge dimensions: 6 mm length, 2 mm width and 0.7 mm thickness) were used for the test. The tests were carried out in a servo-hydraulic universal testing machine (UTM) with a constant strain rate of $10^{-3}$ s$^{-1}$ up to fracture.

<table>
<thead>
<tr>
<th>Table 1.</th>
<th>Thermo-mechanical processing route for different GBE conditions.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Processing condition</td>
<td>Temperature</td>
</tr>
<tr>
<td>Unidirectional rolling (UDR)</td>
<td>750°C</td>
</tr>
<tr>
<td>850°C</td>
<td>UDR850</td>
</tr>
<tr>
<td>Multi-step cross rolling (MSCR)</td>
<td>750°C</td>
</tr>
<tr>
<td>850°C</td>
<td>MSCR850</td>
</tr>
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</table>

Fig. 1. Schematic diagram represents the critical rolling path. (Online version in color.)

Fig. 2. Microstructure of starting material, (a) Inverse pole figure (IPF) map and (b) grain boundary microstructure, the black coloured line is random boundary and coloured lines are special angle boundaries. Grain size distribution has been shown in inset in Fig. 2(a). (Online version in color.)

3. Experimental Results

3.1. Microstructural Evolution

Figure 3 shows the EBSD microstructures for samples processed by the two employed TMP routes involving different strain paths and temperatures. The grain size distribution has been presented in the inset along with the mean grain size and variance. The microstructures clearly reveal the presence of annealing twins in different fractions. The samples subjected to intermediate annealing at 750°C after UDR and MSCR schemes of rolling show lower grain size than the samples annealed at 850°C between the rolling passes. The average grain sizes were 13 μm and 26 μm for UDR750 and UDR850 samples respectively. However, in the case of MSCR processed samples, the grain sizes were 23 μm and 31 μm for MSCR750 and MSCR850 samples respectively. It is expected that the recrystallized microstructure will have a narrow log-normal type grain size distribution. Along with mean grain size and variance, the microstructure can be characterized by the coefficient of variation of grain size distribution $\sigma_m/m$. The $\sigma$ value indicates the states, that is, whether the microstructure is recrystallized or if there is abnormal grain growth. The highest $\sigma_m$ (5%) was observed in UDR750 samples and lowest (2%) in MSCR850 samples.

In Fig. 4, the misorientation angle distribution plots of the
starting material and GBE samples are shown. The maximum frequency of distribution was observed at $\Sigma 3$ ($60^\circ$) and $\Sigma 9$ ($39^\circ$) positions. A very small fraction of low angle misorientation was observed in the starting material and the UDR750 samples. Other than the UDR850 samples, all the GBE samples showed similar fractions of $\Sigma 3$ (50%) and $\Sigma 9$ (5%) boundaries. A small fraction of $\Sigma 27a$ boundary was also observed at $\sim 32^\circ$, which suggests the formation of third order $\Sigma 3$ boundary.

3.2. Grain Boundary Character Distribution

It is well known that GBCD and grain boundary connectivity govern the grain boundary-related bulk properties in polycrystalline materials.30,31) Figure 5 presents the number fraction of different grain boundaries. The CSL boundary fraction increases from 40% to 60% in GBE samples. The UDR850 sample shows less CSL fraction compared to the starting material. In addition, the fraction of CSL boundary was inversely proportional to low angle grain boundaries (LAGBs). However, the fraction of high angle grain boundaries (HAGBs) remained nearly constant in the starting material and GBE samples.

The quantitative fractions of all types of CSL boundaries are presented in Fig. 6 for the starting material and GBE samples. In both starting material and GBE samples, the highest fractions were for the $\Sigma 1$, $\Sigma 3$, $\Sigma 9$ and $\Sigma 27$ CSL boundaries. Other than $\Sigma 3$, very small fractions of $\Sigma 5$, $\Sigma 7$, $\Sigma 11$, $\Sigma 15$ and $\Sigma 23$ boundaries were also observed. As the

![Fig. 3. Microstructure of GBE processed materials, (a) UDR750, (b) UDR850, (c) MSCR750 and (d) MSCR 850. Log normal type grain size distribution has been plotted in inset. (Online version in color.)](image)

![Fig. 4. Misorientation distribution of (a) starting material, (b) UDR750, (c) UDR850, (d) MSCR750 and (e) MSCR850. (Online version in color.)](image)

![Fig. 5. Grain boundary character distribution of starting material and GBE processed samples. (Online version in color.)](image)

![Fig. 6. CSL distribution of (a) Starting material, (b) UDR750, (c) UDR 850, (d) MSCR750 and (e) MSCR850. (Online version in color.)](image)
contribution to GBCD is mainly from the $\Sigma^3$ boundaries, the improved properties of GBE samples could be attributed to the formation of $\Sigma^3$. The UDR750 samples possess the highest fraction of $\Sigma^3$ boundary (51%) while the MSCR750 and MSCR850 samples show nearly 48% of $\Sigma^3$ CSL boundary in the microstructure. The UDR850 materials show the lowest $\Sigma^3$ CSL fraction (30%) whereas the starting material has 38%.

3.3. Grain Boundary Plane Distribution

Randle et al.\textsuperscript{32} pointed out that CSL boundaries processed by GBE do not always improve properties in the desired manner. Moreover, it also does not give information related to the non-CSL grain boundary network. Based on these points, the authors argued that there should be a greater emphasis on CSL boundary planes. This provides the inspiration to focus on the population and distribution of grain boundary planes in the present study. The importance and methodology of analysis is given in references.\textsuperscript{33–35} Following the same approach, the misorientation distribution has been calculated for the planes corresponding to $\Sigma^3$, $\Sigma^9$ and $\Sigma^{27a}$, as these CSL boundaries have maximum population in the microstructure.

The distribution of $\Sigma^3$ plane displays a strong peak at (111) position and the grain boundary planes are perpendicular to the [111] disorientation axis. This corresponding twin boundaries of the type {111}||{111} are known as coherent symmetric twist $\Sigma^3$ boundaries.\textsuperscript{13,20,27,35} The misorientation peak for $\Sigma^9$ boundary lies at 38.9° for (1-14) plane along the [110] orientation. This corresponds to symmetric tilt boundaries. The peak for $\Sigma^{27a}$ boundary is at 31.6° for (1-15) plane along the [110] disorientation axis, which corresponds to symmetric tilt boundaries.\textsuperscript{13} Figure 7 shows the grain boundary plane distribution for $\Sigma^3$, $\Sigma^9$ and $\Sigma^{27a}$ CSL boundaries at angles 60°, 39° and 32° respectively. The units are in multiples of random distribution (MRD). It was noticed that in both TMP routes, the MSCR samples (MSCR750 and MSCR850) show very high $\Sigma^3$ boundary intensity (1 300 and 1 170 MRD respectively) compared to the UDR samples (UDR750 and UDR850) and starting material (523, 625 and 665 MRD respectively). The highest population has been observed for $\Sigma^9$ and $\Sigma^{27a}$ were 8.0 MRD and 7.5 MRD respectively among GBE and non-GBE samples (Fig. 7). In contrast, the population of $\Sigma^3$ boundaries was always significantly higher than $\Sigma^9$ and $\Sigma^{27a}$ boundaries in all cases.

3.4. Texture in GBE Processed Samples

The overall texture of all the differently processed samples is shown in Fig. 8. The texture is presented by inverse pole figures (IPFs) in three directions, namely RD (100), TD (010) and ND (001). The IPFs were calculated from EBSD data. In almost all the samples, very sharp maxima are observed at the <111>, <110> and <112> orientations, but the sharpness of textures has been different. In GBE samples, a very strong <110> orientation along the (010) direction is observed, whereas a very sharp <112> orientation was present in the starting material.

3.5. Tensile Properties

In order to examine the effect of increased fraction of CSL boundaries (after thermo-mechanical processing) on the mechanical properties, tensile tests were carried out. Figure 9 shows the engineering stress–strain curves of the starting material as well as thermo-mechanically processed samples. The curves show significant improvement in

![Figure 7](image_url)

Fig. 7. Grain boundary plane distribution at fixed disorientations, 60°[111] for $\Sigma^3$, 38.9°[110] for $\Sigma^9$ and 31.6°[110] for $\Sigma^{27a}$ of (a) starting material, (b) UDR750, (c) UDR850, (d) MSCR750 and (e) MSCR850. (Online version in color.)
ductility after thermo-mechanical processing. The highest ductility is observed for the MSCR850 samples. The yield strength, ultimate tensile strength and ductility values for different samples are given in Table 2.

### Table 2. Yield strength, ultimate tensile strength and ductility of starting material and thermo-mechanically processed materials.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Yield Strength (0.2% proof strength) (MPa)</th>
<th>Ultimate tensile strength (MPa)</th>
<th>Ductility (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Starting material</td>
<td>320 ± 30</td>
<td>920 ± 50</td>
<td>40 ± 5</td>
</tr>
<tr>
<td>UDR750</td>
<td>320 ± 30</td>
<td>970 ± 50</td>
<td>50 ± 5</td>
</tr>
<tr>
<td>MSCR750</td>
<td>290 ± 30</td>
<td>960 ± 50</td>
<td>48 ± 5</td>
</tr>
<tr>
<td>UDR850</td>
<td>290 ± 30</td>
<td>880 ± 50</td>
<td>51 ± 5</td>
</tr>
<tr>
<td>MSCR850</td>
<td>260 ± 30</td>
<td>930 ± 50</td>
<td>57 ± 5</td>
</tr>
</tbody>
</table>

4. Discussion

Two types of thermo-mechanical processing routes were adopted to obtain the optimum fraction of CSL boundaries to improve the mechanical properties of medium Mn TWIP steels. The materials were characterized in terms of grain boundary character distribution and grain boundary plane distribution. The GBCD results show an increase in CSL boundary fraction after thermo-mechanical processing in UDR processed samples while the GBPD results show opposite trends. The formation and role of CSL boundaries in mechanical properties are discussed in the following sections.

4.1. Grain Boundary Character Distribution (GBCD) vs. Grain Boundary Plane Distribution (GBPD)

Multiple twinning is a commonly observed feature in the microstructures of low SFE FCC materials. The interaction of Σ3 and higher orders of Σ3 boundaries is a result of multiple twinning during recrystallization and grain growth. The formation of Σ9 and Σ27 boundaries is a geometric consequence of the interaction of Σ3 boundaries at triple points. Abou-Ras et al. estimated the probability of formation of Σ9 boundaries from Σ3 boundaries at triple junctions as (n^2) where n is the number fraction of Σ3 boundaries. Similarly, when a Σ3 and Σ9 boundary interacts, the probability of formation of Σ27 is n×m, where m is the number fraction of Σ9 boundary.

In the present investigation, the formation of Σ9 grain boundaries has been less than 5%. These Σ9 boundaries coincide with the {114} symmetric tilt boundary. Beladi et al. showed the population of Σ9 boundaries in TWIP steels to be 1.5%. The formation of different fractions of CSL boundaries (Σ3, Σ9 and Σ27) is dependent on composition, size and morphology of grains and texture. In the present study, grain sizes of the differently processed GBE materials were different; however, the grain morphology was equiaxed in all the cases and the texture was also similar (Figs. 3 and 8). Randle et al. reported that a decrease in grain size increases the population of CSL boundaries. The maximum CSL fraction was observed in UDR750 samples calculated by GBCD, with a grain size of 13 μm.

The grain boundary plane distribution of Σ3, Σ9 and Σ27 CSL boundaries is shown in Fig. 7. The GBE processed samples display a higher population of CSL boundaries than the starting material. Both the MSCR samples exhibit a very high population of CSL boundaries than the corresponding UDR samples (Fig. 7). However, the GBCD plot shows maximum intensity for the UDR750 sample while the GBPD shows an exactly opposite trend (Fig. 5). It is to be noted that GBCD does not distinguish between coherent and non-coherent CSL boundaries. Hence, it is appropriate to characterise CSL boundaries using GBPD rather than GBCD.

4.2. Role of Strain Path in the Evolution of CSL Boundary

Schuh et al. reported that a minimum CSL boundary fraction of ~40% is required to achieve the advantage of grain boundary engineering. In the present investigation, a CSL boundary fraction of ~60% was achieved by the newly
adopted GBE processing route, namely cross rolling. Figure 5. Figure 10 shows the grain boundary microstructure and CSL boundary network obtained from two different processing routes. Cross rolling is reported to lead to activation of more number of slip systems in polycrystalline materials. However, more interaction between dislocations and LAGBs as well as other slip systems reduces the intra-granular misorientation in cross rolled samples. Annihilation takes place in (111) slip plane and <110> slip direction, hence at very low strain these are the most favourable active slip plane and slip direction. Both edge and screw dislocations are taken part in annihilation mechanism. In case of edge dislocation, annihilation take place in parallel slip plane, whereas annihilation can take place in non-parallel slip plane for screw dislocation. The mechanism involved progressive annihilation of dislocations near angle grain boundaries, which transforms some of the LAGBs and HAGBs to CSLs boundaries during multi-steps cross rolling and intermediate annealing. It can be understood that cross rolling and subsequent annealing destroy the LAGBs and increase the tendency to form HAGBs as well as CSL boundaries.

The deviation of Σ3 and Σ9 CSL boundaries from the ideal coincidence angle is shown in Figure 11 for both non-GBE and GBE processed samples. The deviation has been calculated according to Brandon’s criterion. The maximum deviation was found to be 8.67° for Σ3 and 5° for Σ9. No significant CSL deviation was observed in Σ3 and Σ9 in UDR750, MSCR750 and MSCR850 samples. The low deviation suggests that the special boundaries coincide with their ideal positions. It also gives information regarding the number of dislocations present in the CSL boundary, as the deviation increases with an increase in the number of dislocations. The starting material and UDR850 samples show comparatively higher Σ3 and Σ9 boundary deviations, which is a manifestation of more dislocations present in the boundaries or that the boundaries are not completely coherent.

4.3. Role of Texture in Grain Boundary Character Distribution

The Figs. 5, 7 and 8 show the GBCD, GBPD and annealing texture respectively. The annealing texture plays a role in the formation of CSL boundaries. It is reported that strongly textured materials contain a very high fraction of LAGBs and high Σ boundaries. There is a strong correlation between grain boundary character and sharpness as well as type of texture; this helps in the prediction of CSL boundaries from textured materials. Garbacz and Grabski reported a correlation between sharp texture and formation of CSL boundaries in polycrystalline FCC materials. Further, a correlation between grain boundary misorientation distribution and CSL boundary distribution with texture was firmly established.

A specific Σ boundary is oriented in a preferential direction and it can be predicted from possible CSL orientation relations from the <100>, <110> and <111> textures. Sinha et al. showed the presence of weak Bs ([110]<112>), Goss ([110]<001>) and Cu ([112]<111>) components in austenite stainless steel. According to their observation, samples containing optimum CSL fractions (GBE samples) have stronger texture than non-GBE samples. Among all the CSL boundaries, Σ3 and Σ9 have the highest fractions. The Σ3, 60°/<111> , is aligned in the <111> direction and Σ9, 38.9°/<110> is aligned in the <110> direction; this can be correlated to sharp <111> and <110> texture formations (Fig. 8).

4.4. Mechanical Properties

Twinning plays a crucial role in the improvement of ductility in FCC materials with low stacking fault energy (SFE). Two plausible mechanisms are established that can explain the improvement in ductility with an increase in CSL boundaries. In TWIP steels, deformation twins mainly nucleate at the grain boundaries. During deformation, dislocations accumulate at random grain boundaries as well as annealing twin boundaries. The progressive accumulation of dislocations at boundaries leads to the nucleation of deformation twins. In the case of random boundaries, the accumulation is relatively faster and twins nucleate in the early stages of deformation. On the other hand, CSL boundaries act as nucleation sites for twinning at intermediate to high strain levels. The strain hardening as well as total elongation increases due to twinning at later stages of deformation. The grain reference orientation deviation (GROD) map for tensile deformed specimen in Figs. 12(a) and 12(b) for starting material and MSCR850 samples show strain accumulation due to geometrically necessary dislocation (GND) at random as well as CSL boundaries. The microstructure was taken at near tip at the crack region of the tensile
sample. The map indicates the origin of lenticular shaped deformation twins (indicated by red arrow) from grain and twin boundaries (in Fig. 12). The blue arrows show the pre-existence of CSL boundaries. After the accumulation of strain, the CSL boundaries have deviated from their exact locations due to accumulation of dislocations. The maximum fraction of twins was observed at CSL twin boundaries; this indirectly explains the improvement in ductility after the increase of CSL boundaries.

Two types of cracks are mainly observed in polycrystalline materials that are based on grain boundary structure, namely intergranular cracks (that propagate through grain boundaries) and transgranular cracks (that propagate through grain interiors).14) It is reported, in literature, that intergranular cracks cause early failure whereas transgranular cracks increase overall ductility. The CSL boundaries are low energy grain boundaries and are known to fracture less than random boundaries during straining.46,47) In the present investigation, the CSL boundary fraction increases significantly after processing. This could be the possible reason for improvement in ductility.

5. Conclusions

In the current study, an attempt was made to improve the special angle grain boundaries in medium Mn TWIP steels by specially designed thermo-mechanical processing routes. The outcomes of detailed microstructural characterization and assessment of tensile properties led to the following conclusions.

(1) The specially designed thermo-mechanical processing scheme for grain boundary engineering led to an increase in the CSL boundary fraction by ~60%.

(2) Special angle grain boundary fraction has been calculated using grain boundary character distribution (three parameter approach) and grain boundary plane distribution (five parameter approach). Grain boundary plane distribution shows higher fraction of CSL boundary in MSCR processed samples. The MSCR processed sample showed higher ductility compared with UDR process samples. It has been shown that grain boundary plane distribution is much better method for the estimation of special angle boundary fraction.

(3) Activation of more number of slip systems during cross rolling enhanced the formation of high fraction of CSL boundaries in MSCR processes samples.

(4) Interaction of CSL with 9 and 27 boundaries enhances the formation of CSL boundaries in the samples processed at grain growth temperature. During grain growth, the frequency of different kind of special angle boundary is always very high compared with recrystallization temperature.

(5) An appreciable enhancement in ductility was observed after thermo-mechanical processing due to improvement in CSL boundary fraction in the microstructure. The highest ductility was observed in MSCR850 samples.

(6) Annealing texture coincides with <111> and <110> directions help in the formation of special angle grain boundary. Stronger the along the <111> and <110> forms very high fraction of CSL boundaries.

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