Effects of hot rolling and homogenisation treatment on low alloy steel ingot

Mahdiyeh Baharvand, Amir Zanganeh, Hamed Mirzadeh and Mohammad Habibi Parsa

School of Metallurgy and Materials Engineering, College of Engineering, University of Tehran, Tehran, Iran

ABSTRACT
The effects of hot rolling and homogenisation treatment on the microstructure and mechanical properties of low-alloy transformation-induced plasticity (TRIP) steel were studied. With increasing the reduction in thickness during rolling, the band spacing and the average ferrite grain size decreased with the consequent enhancement of mechanical properties. Homogenisation treatment resulted in the gradual fading of the banded morphology, more homogeneous microhardness profile, and twice total elongation compared to the as-cast counterpart. The experimentally observed homogenisation time judged by the disappearance of the banded structure was in agreement with the obtained values based on the band spacing and elemental analysis. This work provided scientific evidence for the assumptions used to derive the homogenisation formulae.

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TRIP steel; high-temperature rolling; homogenisation; band spacing; segregation ratio

Introduction
The low-alloy steels containing Si/Al might be processed by heat treatment to obtain the transformation-induced plasticity (TRIP) steels [1,2]. These steels show good strength–ductility balance as a result of the inhibition of necking by the TRIP effect [3,4]. Besides the TRIP heat treatment (intercritical annealing and isothermal bainitic transformation), the controlling of the primary microstructure is an important factor to obtain an appropriate final microstructure [5,6].

These steels, with their special chemical composition, should be processed by casting, hot rolling, and cold rolling to obtain the required initial sheet. The inhomogeneity of the as-cast microstructure is primarily resulted from the microsegregation of alloying elements during solidification (especially Mn) and appearance of Mn-lean and Mn-rich regions in the austenite. During hot rolling, the elongation of these regions results in the appearance of the ferritic/pearlitic banded structure [7–11]. This, in turn, plays a key role in the overall thermomechanical processing of the material [12], where the distribution of the phases, especially the retained austenite, is significantly affected by these inhomogeneities [13]. Therefore, the homogenisation treatment can be considered as one of the primary processing routes to reduce the microsegregation effects of alloying elements [14].

The homogenisation treatment has been applied to many commercial alloys for the enhancement of their properties [15–21]. For a low-alloyed TRIP steel, a profound effect of homogenisation treatment on the decomposition of austenite, suppression of microstructural banding, and work-hardening capacity were observed [13]. In the production of high-manganese TRIP and TWIP steels, significant microsegregation effects were observed after solidification, where the cast alloy was treated by electroslag remelting (ESR). After a simple hot rolling procedure with a thickness reduction of 90% and without further heat treatment, microsegregation of manganese was reduced to ∼5 wt-% [22].

Hot rolling is used to replace the coarse dendritic microstructure with a recrystallised and equiaxed one, which is a required step to obtain a sheet with optimum microstructure. While hot rolling leads to the formation of banded structure, the band spacing decreases by increased rolling reduction, and hence, the distance between Mn-lean and Mn-rich regions decreases. The latter significantly reduces the diffusion distance during homogenisation and increases the kinetics of this process [7–9].

Since the homogenisation treatment is time-consuming, the optimisation of this process is important from the industrial standpoint [23,24]. However, the experimental evidences for the homogenisation (disappearance of the banded structure and elemental analysis) and its relation to the diffusional parameters need much more experimental work. Moreover, the assumptions used to derive the homogenisation formulae [24] should be rationalised from the scientific and experimental standpoints. The present work aims to deal with these subjects.
Experimental details

Processing

A low-alloy steel with the chemical composition (wt-%) of 0.249C–1.25Si–1.16Mn–0.905Al was used in this work, which is suitable for TRIP heat treatment [25,26]. The hot rolling operations were performed at 1050°C up to the reductions in the thickness of 20%, 35%, 50%, and 90%. Owing to the small thickness of the 90% rolled sample and its very fine microstructure, the reduction in the thickness of 50% was selected for further analysis to make the measurements more reliable. After rolling, the samples were furnace cooled to room temperature to induce the formation of the banded structure during cooling from the austenitic region [27,28]. The homogenisation heat treatment was performed by annealing the 50% hot-rolled specimen at 1100°C for holding times up to 9 h followed by furnace cooling. The choice of the homogenisation temperature and time was based on the preliminary estimation of the homogenisation kinetics by consideration of the observed hot-rolled microstructures. In fact, shorter homogenisation times can be achieved at higher homogenisation temperatures. Figure 1 shows a schematic representation of the processing routes used in the present work and the samples have been defined in Table 1.

Characterisation

The samples were etched in the 2% nital solution. An Olympus Vanox optical microscope and an FEI Nova field-emission scanning electron microscope (FE-SEM) were used for microstructural analysis. The average ferrite grain size was obtained based on the standard intercept method. Elemental analysis was based on the energy-dispersive spectroscopy (EDS) using a Bruker QUANTAX XFlash 6 detector (with incorporation of a true standardless P/B ZAF correction technology [29]). Vickers hardness test with loads of 0.05 and 30 kg were used for micro- and macro-indentation, respectively. Round tensile test specimens were prepared according to the subsize ASTM E8 standard with the gauge length of 16 mm. Tensile testing was carried out at room temperature by a computerised testing machine at the constant crosshead speed of 1 mm/min. The fractographic analysis of the broken tensile samples was also performed using the same FE-SEM.

Calculation of the homogenisation time

It is often of interest to be able to calculate the time taken for an inhomogeneous steel to reach complete homogeneity, which leads to the disappearance of the banded structure [7,24,30]. Leslie assumed that the variation of Mn concentration in a rolled plate occurs in only one direction and along a straight line normal to the bands. The concentration of Mn after homogenisation can be simply modelled by a sinusoidal function as shown in the below equation and Figure 2

\[
C - \bar{C} = \beta \sin(\pi x/l) \exp\left(-\pi^2Dt/l^2\right)
\]  

where \(C\) is the concentration of Mn at any point \(x\) through the thickness of the plate, \(\bar{C}\) is the average...
concentration of Mn and the band spacing is taken as $2l$ [24, 31].

At $t = 0$ and $x = l/2$, $C_{0m} - \bar{C} = \beta$. At $x = l/2$, $C_{tm} - \bar{C} = \beta \exp\left(-\pi^2Dt/l^2\right)$. Therefore, the following relation can be obtained:

$$\frac{(C_{tm} - \bar{C})}{(C_{0m} - \bar{C})} = \exp\left(-\pi^2Dt/l^2\right)$$  \hspace{1cm} (2)

Leslie assumed that when the amplitude of the concentration profile at $x = l/2$ reaches 1/10 of the initial value, the required homogenisation has been achieved. Therefore, $(C_{tm} - \bar{C})/(C_{0m} - \bar{C}) = 0.1$, and hence, the following equation can be proposed:

$$\frac{Dt}{l^2} = 0.233$$  \hspace{1cm} (3)

This equation was used to predict the homogenisation time based on the diffusion on Mn (the effect of temperature) and the band spacing as will be discussed later. It should be noted that the microsegregation of other alloying elements such as Al and Si also happens during solidification. However, it is a usual practice to consider the microsegregation of Mn, which is the most important element responsible for the appearance of the banded structure [7–9]. The microsegregation of Al and Si has been discussed in the previous work and the importance of the microsegregation of Mn has been revealed [5].

Results and discussion

Hot rolling

The optical micrograph of the as-cast specimen is shown in Figure 3(a), which is similar to the well-known ferritic–pearlitic steels [32–34]. The appearance of the pearlite microstructure in the interendritic regions is a good sign of the microsegregation during solidification. This inhomogeneity is the basis for the appearance of the banded structure during hot rolling [7–10] as shown in Figure 3(b–e). As can be seen in Figure 3(b), the dendritic structure is being destroyed by 20% hot rolling reduction and the banded structure can be discernible. Moreover, equiaxed ferrite grains as a result of recrystallisation can be observed. At higher rolling reductions (Figure 3(c–e)), the band spacing was reduced and the ferrite grain size was refined considerably. The results are summarised in Figure 3(f), where it can be seen that the changes in the ferrite grain size and band spacing are pronounced at low rolling reductions but become less significant at higher reductions in thickness.

The change in hardness of the hot-rolled sheets is also shown in Figure 3(f), where the increase in hardness by increasing the rolling reduction is evident. This can be related to the observed grain refinement. Therefore, it is interesting to relate the hardness to the average grain size ($d$) by the well-known Hall–Petch plot [35–37], which is shown in Figure 3(g) and resulted in the following relationship

$$\text{Hardness} = \frac{46.26}{\sqrt{d}} + 179.2$$  \hspace{1cm} (4)

The obtained Hall–Petch slope of 46.26 kg mm$^{-2}$ μm$^{0.5}$ and friction hardness of 179.2 kg mm$^{-2}$ are consistent with the previous reports on the Hall–Petch behaviour of mild steel [38].

Homogenisation

The 50% hot-rolled sheet was homogenised at 1100°C. The choice of final homogenisation time was based on the predictions of Equation (3). The band spacing of the
50% hot-rolled sample was determined as 30 $\mu$m, and hence, $l = 0.0015$ cm.

The diffusion of Mn in austenite can be expressed as follows [24]:

$$D(Dm/s) = 0.65 \exp(-276000/RT)$$  \(5\)

Therefore, at $T = 1100^\circ$C = 1373 K, $D$ can be determined as $1.7 \times 10^{-11}$ cm$^2$s$^{-1}$. As a result, based on Equation (3), the homogenisation time to reach 90% homogeneity is $\sim 8.5$ h. Accordingly, the homogenisation time of 9 h was used in this work.

The homogenised microstructures at various holding times are shown in Figure 4. In this low magnification images, it can be easily seen that, by increasing the holding time, the banded structure tends to vanish, which is a sign of homogenisation. However, the average grain size of the 50% rolled sample (holding time of zero) and the 9 h annealed sample is $\sim 11$ and $\sim 75$ $\mu$m, respectively. This reveals that the homogenisation treatment resulted in appreciable grain growth during elevated temperature exposure.

In fact, after the holding time of 9 h, the microstructural banding cannot be seen (Figure 4(d)), but at 8 h, the sign of banding can be still identified (Figure 4(c)). This reveals that the prediction of Equation (3) is reliable, which predicted the homogenisation time of 8.5 h. In fact, by consideration of holding time of 9 h, Equation (2) can be presented as follows:

$$\frac{(C_{tm} - \bar{C})}{(C_{om} - \bar{C})} = \exp(-\pi^2Dt/l^2) = 0.089$$  \(6\)

Therefore, the $(C_{tm} - \bar{C})/(C_{om} - \bar{C})$ value of 0.089 was used in this work, which is lower than the assumed value of 0.1. This insures the homogenisation of the ingot.

Besides the disappearance of the banded structure, the homogenisation of hot-rolled material can

![Figure 3. (a–e) As-cast and hot-rolled microstructures, (f) summary of microstructural results and hardness measurements, and (g) Hall–Petch plot for hardness.](image)
be directly studied by analysing the variation of elements (especially Mn) along a given distance, which is shown in Figure 5(a). It can be seen that the 50% rolled sample shows huge variation in the amount of Mn due to the microsegregation. However, after the homogenisation time of 2 h, the amplitude of the variations decreased considerably, indicating the achieved homogeneity. After the homogenisation time of 9 h, the mean amplitude of the variations can be compared to that of the initial amplitude

$$\frac{(C_{tm} - \bar{C})}{(C_{0,m} - \bar{C})} = \frac{(1.8 - 1.16)}{(5.8 - 1.16)} = 0.13$$

where $\bar{C}$ of 1.16 was taken from the chemical composition of the studied material and the values of $C_{tm}$ and $C_{0,m}$ were estimated from Figure 5(a). It can be seen that the value of 0.13 is comparable to the assumed value
of 0.1 by Leslie [24]. Therefore, the EDS analysis of the present work, in conjunction with the disappearance of the banded structure, provides experimental evidence for the assumptions made in Equations (1–3) for the homogenisation of hot-rolled ingots.

Since the model of Equation (6) depends on the band spacing \( \left(Dt/l^2 = 0.245\right) \) and the spacing between the bands \( (2l) \) or the diffusion distance can be reduced by increasing the reduction in thickness \( (r) \), it is possible to relate the homogenisation time \( (t) \) to \( r \), which is summarised in Table 2. This table reveals that the homogenisation time decreases significantly by decreasing band spacing through increasing the reduction in thickness during rolling.

The segregation ratio has been defined as \( SR = C_{\text{max}}/C_{\text{min}} \), where \( C_{\text{max}} \) and \( C_{\text{min}} \) are the maximum and minimum concentration of the segregating element, respectively [14]. Based on Figure 5(a), the values of \( SR \) can be determined as \( \sim 20 \), 3.8, and 2.8 for R50, H2, and H9 samples, respectively. Mathematically, complete homogenisation can be obtained when \( SR \) approaches the value of 1, which theoretically happens at \( t \to \infty \). Obviously, this is not applicable for industrial practice, and hence, consideration of a critical value for \( SR \) is reasonable. Based on the results of the present work, the value of \( \sim 2.8 \) seems to be appropriate for Mn. It is noteworthy that the \( SR \) values of 1.20, 1.26, and 1.41 for Cr, Mo, and V have been obtained by Han et al. [23] for homogenisation of steel ingot.

To investigate the effects of homogenisation on the homogeneity of properties, the microhardness profile of samples was taken into account as shown in Figure 5(b–d). The contours represent the relative percentage of microhardness \( (R_H) \) based on the following equation:

\[
R_H = 100 \times \left| \frac{H - \bar{H}}{\bar{H}} \right|
\]

Therefore, a distribution of lower \( R_H \) values is indicative of better homogeneity. Since Mn is a strong solution hardening element in ferrite [39], the segregation of Mn can be qualitatively related to the microhardness measurements. For the as-cast sample, it can be seen in Figure 5(b) that \( R_H \) varies from 5% to 30% in different locations. For the 50% hot-rolled sample (R50), \( R_H \) varies from 10% to 25% (Figure 5(c)), which reveals the presence of microsegregation. For the homogenised sample (H9), Figure 5(d) reveals that the \( R_H \) varies from 0% to 5%, which is an indication of the achieved homogeneity.

**Mechanical properties**

Tensile stress–strain curves are shown in Figure 6(a). The as-cast sample shows low strength (UTS of \( \sim 580 \) MPa) and low ductility (total elongation of \( \sim 23\% \)). The fractograph of this sample in Figure 6(b) reveals the

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**Table 2. Effect of reduction in thickness during hot rolling on the homogenisation time.**

<table>
<thead>
<tr>
<th>( r )</th>
<th>( 2l (\mu m) )</th>
<th>( t (h) )</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>60</td>
<td>36</td>
</tr>
<tr>
<td>35</td>
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</tr>
<tr>
<td>50</td>
<td>30</td>
<td>9</td>
</tr>
<tr>
<td>90</td>
<td>8.5</td>
<td>3</td>
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**Figure 6.** Tensile stress–strain curves and the corresponding fractographs of the broken tensile samples.
presence of dimples, large cleavage facets, and cracks. The inhomogeneous structure of as-cast sample with the potential presence of casting defects resulted in the relatively poor tensile properties of this sample [40]. Owing to the nature of fracture surface (large cleavage facets), this sample does not show post-uniform elongation in its tensile stress–strain curve.

The microstructural refinement introduced by hot rolling resulted in the enhancement of tensile properties of R35 and R50 samples as shown in Figure 6(a). The corresponding fractographs in Figure 6(c,d) show the prevalence of the dimple fracture. As a result, the post-uniform elongation can be seen in these samples, especially for the R50 sample.

After homogenisation (H9 sample), while the tensile strength becomes comparable to that of the as-cast sample (due to the coarsening of ferrite grains), the total elongation is significantly larger (more than twice). This justifies the application of homogenisation treatment for the enhancement of properties of the as-cast ingot. Compared to the R50 sample, the H9 sample shows higher ductility but much lower strength. However, the properties of the sheet have become more homogeneous (Figure 5(d)) and the banded structure has vanished, which might be advantageous for applications of the processed sheets.

Conclusions
Hot rolling and homogenisation effects on the microstructure and mechanical properties of low-alloy steel were studied. The following conclusions can be drawn:

1. Hot rolling resulted in the appearance of the banded structure instead of the dendritic one. With increasing the reduction in thickness during hot rolling, the band spacing and the average ferrite grain size decreased with the consequent enhancement of mechanical properties and a more homogeneous microhardness profile. The hardness of the hot-rolled sheets was successfully related to the average grain size via the Hall–Petch relation of Hardness = 46.26/√d + 179.2.

2. Homogenisation treatment resulted in the gradual fading of the banded morphology due to the back-diffusion of segregated Mn from Mn-rich regions. The value of ∼ 2.8 was found to be appropriate for the segregation ratio of Mn after homogenisation. The homogenisation resulted in the homogeneous microhardness profile and twice total elongation compared to the as-cast counterpart while maintaining the same strength level. Compared to the fine-grained hot-rolled sheets, the properties of the homogenised sheet have become more homogeneous and the banded structure has vanished.

3. The experimentally observed homogenisation time judged by the disappearance of the banded structure was in agreement with the calculated value based on the band spacing and that obtained from the EDS analysis. This provided scientific and experimental evidence for the assumptions used to derive the homogenisation formulae. Based on these findings, the effect of the reduction in thickness during hot rolling on the homogenisation time was also discussed. It was concluded that the main findings of this work do not change with increasing the rolling reduction or the homogenisation temperature; although the required homogenisation will be achieved at shorter times based on the band spacing and diffusion coefficient.

Disclosure statement
No potential conflict of interest was reported by the author(s).

ORCID
Hamed Mirzadeh © http://orcid.org/0000-0001-7179-0052

References


