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**Characterisation of strain-induced precipitation behaviour in microalloyed steels
during thermomechanical controlled processing**

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Abstract

The temperature at which thermomechanical controlled processing is undertaken strongly influences strain-induced precipitation (SIP) in microalloyed steels. In this study, the recrystallisation-precipitation-time-temperature curve was simulated to determine the full recrystallisation temperature, recrystallisation-stop temperature and the temperature where precipitation would occur at the shortest time. The calculated temperatures were verified by experimental testing for rolling between 1100°C and 850°C. On the basis of this a finishing deformation of 850°C was chosen in order to maximise the precipitate number density formed in a fully unrecrystallised austenite. The orientation relationship between the SIP in austenite, and subsequent transformation to ferrite was identified by calculation from the coordinate transformation matrix, and by electron diffraction in the transmission electron microscope. The NbC formed as coherent/semi-coherent precipitates in the austenite, and remained coherent/semi-coherent in the ferrite, indicating a Kurdjumov-Sachs orientation relationship between the austenite and ferrite on transformation.

Keywords: Microalloyed steel; recrystallisation-precipitation-time-temperature curve; strain-induced precipitates; orientation relationship.

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1. Introduction

Microalloyed steels represent an important class of steel used in the automotive, construction, transportation and energy sectors. They are relatively easy to fabricate, and have good strength and toughness with low carbon content and a corresponding increase in the microalloying elements such as Nb, Ti & V [1–7]. Commercial processing of flat product lines usually consists of thermomechanical controlled processing, where the finishing deformation passes take place in the fully unrecrystallised ferrite. However, due to these lower final deformation temperatures, problems may arise during production including high mill loads and a corresponding high energy consumption [5]. The aim of the process is to roll in the softer austenite region with the temperature lower than the recrystallisation stop temperature, which gives a high accumulated strain and results in a high nucleation rate of strain induced precipitation (SIP), refining the final grain size [6,7]. It is expected that the SIP in the austenite exhibits an orientation relationship between the precipitate and the matrix, which are coherent/semi-coherent with austenite. Following transformation to ferrite, an orientation relationship should be preserved, but this has not been examined in detail in the literature. The aim of this work was to determine the orientation relationship (OR) between precipitates and austenite transformed to ferrite.

2. Experimental procedure

The material used in the research was a microalloyed steel containing Nb (designated “Nb steel”) that was made by vacuum induction melting and casting to 220mm×65mm×28mm square laboratory ingots at ArcelorMittal. The chemical composition was analysed by Sheffield Testing Laboratories as Fe–0.067C–0.11Si–0.77Mn–0.03Nb–0.02Ni (wt.%). The ingots were soaked at 1300°C for 2 hr and were then hot-rolled into 12 mm thick plates in a 2-high experimental rolling mill, finishing at 1100°C. After hot-rolling, the plates were

quenched in ice water, and then machined into plane strain compression (PSC) specimens with dimensions of 60mm×30mm×10 mm. Prior to deformation via PSC, samples were preheated at 1100°C for 30s, and then air cooled to the deformation temperature at 850°C. Isothermal deformation of an equivalent strain of 0.3 was carried out at a constant true strain rate of 10s⁻¹ followed by water quench.

Optical microscopy and transmission electron microscopy (TEM) were used to investigate the microstructures, the morphology and composition of precipitates as well as the orientation relationship between precipitates and matrix. After cutting samples from the central areas of PSC specimens, samples were prepared for optical microscopy in the standard way. Samples were etched in picric acid to reveal the prior austenite grain boundaries. The average prior austenite grain size was measured at different rolling temperatures using the linear intercept method (ASTM E-112). To compute the prior austenite grain size in the Rolling Direction, 8 images at each deformation temperature were measured and the mean value of grain size was determined. To identify the composition and microstructure of the precipitates, carbon extraction replicas were prepared. Samples to produce TEM thin foil specimens were prepared by electropolishing. TEM was performed on a JEOL 2010, operating at 200 kV, to analyse the morphology and composition of nanometer sized precipitates as well as the OR between SIP in the parent austenite and ferrite matrix. Chemical analysis was conducted using an Oxford instruments energy dispersive X-ray spectroscopy (EDS) detector. The structures of precipitates have been observed by HRTEM. The OR between SIP and ferrite matrix were analysed by selected area diffraction (SAD).

3. Results

3.1 Recrystallisation-precipitation-time-temperature (RPTT) curve

The suppression of recrystallisation is important, because the extent of strain accumulation will determine the volume fraction of the SIP and the final grain size, which are key to the mechanical properties. It is well known that the recrystallisation behaviour for Nb steel can be influenced by the microalloy elements, because the recrystallisation is inhibited when the precipitates pinning force (F_{pin}) exceeds the driving force (FR) for recrystallisation [8], [9].

In the current work, the initial Dutta and Sellars's precipitation model [10] and the later Dutta, Palmiere and Sellar's model [11] were used to predict the precipitation behaviour by calculating the precipitation start temperature-time diagrams (Eq. 1).

$$t_{0.05} = 3 \times 10^{-6} [Nb]^{-1} \varepsilon^{-1} Z^{-0.5} \times \exp \frac{270000}{RT} \exp \frac{2.5 \times 10^{10}}{T^3 (\ln k_s)^2} \quad (1)$$

where $t_{0.05}$ is time for 5% precipitation; [Nb] is the niobium in solution; Z is the Zener-Hollomon parameter; k_s is the solute supersaturation ratio expressed by considering the Nb solution model by Palmiere et al. [12] [13]:

$$k_s = \frac{\log[Nb][C]}{2.06 - \frac{6700}{T}} \quad (2)$$

The [Nb] was calculated using carbon replica specimens after reheating to 1100°C for 30 s followed by water quenching. Using the experimental data from the Nb microalloyed steel, the typical "C" shaped curves were obtained and shown in Fig. 1. The nose of the precipitation start temperature diagram for the SIP is located at 0.3 s and 840 °C for this steel.

There are various definitions of the recrystallisation-stop and recrystallisation-complete temperatures. In this study, the recrystallisation-stop temperature is defined as having no greater than 5% recrystallisation, and the recrystallisation-complete temperature is defined as

having at least 95% recrystallisation [14-16]. Then, considering the effect of niobium in solid solution on the recrystallisation kinetics of austenite, Dutta and Sellars [10] provided another model to calculate the 5% recrystallisation curves for the Nb steel using Eq. (3).

$$t_{0.05x} = 6.75 \times 10^{-20} d_0^2 \varepsilon^{-4} \times \exp \frac{300000}{RT} \exp \left\{ \left(\frac{2.75 \times 10^5}{T} - 185 \right) [Nb] \right\} \quad (3)$$

where $t_{0.05x}$ is time for 5% recrystallisation, d_0 is original grain size in μm , and ε is strain. Finally, the full recrystallisation time expressed as a function of the recrystallized volume fraction (X) was calculated using the Johnson et al. model [17-19] and is given by Eq. (4).

$$X = 1 - \exp \left(-\ln(0.95) \left(\frac{t}{t_{0.05x}} \right)^2 \right) \quad (4)$$

Full recrystallisation is defined as the volume fraction of recrystallisation of at least 95%. Then, with 95% substituted into Eq. (4), the recrystallisation finish time (t_f) can be estimated as:

$$t_f = 7.66 \times t_{0.05x} \quad (5)$$

Using Equations (1), (3) and (5), the interaction of precipitation and recrystallisation with different rolling temperatures was carried out and is shown in Fig. 1. Then, the full recrystallisation (T_1) and the recrystallisation-stop (T_s) temperatures were calculated using the respective times for 5% and 95% recrystallisation equalling to the time for 5% SIP. Therefore, the recrystallisation behaviour for Nb steel could be determined with the $T_{5\%}$ at 917°C and $T_{95\%}$ at 957°C from the RPTT curve.

Fletcher predicted the recrystallization stop temperature, taking account of the strain [20]:

$$T_{5\%} = 203 - 310C + 657\sqrt{[Nb]} + 683e^{-0.36\varepsilon} \quad (6)$$

where ε is the pass strain. The predicted $T_{5\%}$ value in this study is at 909°C, which is as close as the calculated data of 917°C by Dutta's equation for Nb steel.

Thus, the recrystallisation-stop temperature is approximately 917°C, and the recrystallisation-complete temperature is approximately 957°C. Therefore, any deformation taking place between these temperatures will result in a partially recrystallised microstructure. As the nose temperature for the SIP of this steel composition is at 840°C, the final deformation temperature of 850°C was selected in order to obtain the highest volume fraction of precipitates while enabling for the deformation of fully unrecrystallised austenite.

3.2 A_{e3} and A_{r3} for Nb steel

The upper equilibrium temperature between ferrite and austenite, A_{e3} , and the ferrite start temperature, A_{r3} , can be calculated using different models considering the weight percentage of alloying elements. In this study, the equilibrium phase transformation temperature of A_{e3} was calculated by Eq. (7) for low carbon steels [21]. The Blas et al. [22] model was considered for the simulation of the $\gamma \rightarrow \alpha$ start transformation temperature (A_{r3}), which was derived for a low carbon content between 0.024-0.068 %wt and is expressed as Eq. (8):

$$A_{e3} = 871 - 254.4\sqrt{\%C} - 14.2\%Ni + 51.7\%Si \quad (7)$$

$$A_{r3} = 903 - 328(\%C) - 102(\%Mn) + 116(\%Nb) - 0.909v \quad (8)$$

where v is cooling rate with °C/s. In the present study, the carbon content was 0.065 %wt for the Nb steel, which satisfied the condition of the Eqs. (7) and (8). The A_{e3} was 870°C derived from Eq. (7). The A_{r3} temperature was calculated from Eq. (8) as 796°C, which is lower than the rolling temperature of 850°C. Therefore, the SIP formed in the austenite region for the rolling temperature at 850°C.

3.3 Prior austenite grain structure and recrystallization behaviour at different hot rolling temperatures

Fig. 2 shows representative optical images of prior austenite grains at six deformation temperatures viz. (a) 850 °C, (b) 900 °C, (c) 950 °C, (d) 1000 °C, (e) 1050 °C and (f) 1100 °C for Nb steel. The equiaxed prior-austenite grain size was stable with the hot rolling temperatures in the range of 1000 °C and 1100 °C for Nb steel. Fig. 3 gives the grain size as a function of temperature. With the hot rolling temperature being reduced to 950 °C, the prior austenite grains became slightly elongated in the rolling direction (RD). Decreasing the hot rolling temperature from 900 °C down to 850 °C, the prior austenite grains became fully elongated with size coarsening in RD.

The optical images in Figs. 2 and 3 indicate that the recrystallization stop temperature is higher than 900 °C. Partial recrystallization takes place at 950 °C, which is the temperature between the recrystallization limit temperature and recrystallization stop temperature. The prior austenite grains plateau at 1000-1100 °C, which further illustrates that full recrystallization occurs.

3.4 Strain induced precipitation

3.4.1 The volume fraction and composition of the strain induced precipitates

The precipitates at each rolling temperature were separated into two groups by the size precipitate size; one group had dimensions larger than 20nm, believed to precipitates that had not dissolved on heating, while the other group was strain induced precipitates with a size less than 20nm. In this study, it is only focused on the strain-induced precipitates. In order to identify the effect of the hot rolling temperatures on the strain-induced precipitation behaviour, the volume fraction of precipitates calculated using the TEM replica data shown in

Figs. 4. Then, using the amount of Nb in the precipitates, the volume fraction of precipitates can be also expressed using Eq. (9) [5], [19], [20] and the results are shown in Fig. 5.

$$f(t) = \left[\frac{\rho_f}{\rho_{\text{precipitate}}} \times \left(\frac{Z+1}{Z} \right) / 100 \right] \times \{ \text{Nb} \} \quad (9)$$

where $f(t)$ is volume fraction of precipitates as a function of holding time; ρ_f and $\rho_{\text{precipitate}}$ are the densities of austenite and precipitates, respectively; Z is the stoichiometric ratio of the precipitates; $\{ \text{Nb} \}$ is the Nb concentration in the precipitates. From Fig. 5, the volume fraction of precipitates as a function of rolling temperatures appears to be logical in the Nb steel. It can be seen that the volume fraction of precipitates increased as the deformation temperature decreased for Nb steel.

After being deformed at 850°C to an equivalent strain of 0.3 followed by water quenching, the morphology of the SIP was investigated by HRTEM and EDS spectrum. The HRTEM presented in Fig. 6 (a) and (b) was taken from the same sample shown in Fig. 4 (f). The nano-sized particle in Fig. 6 (a) and (b) has $d_{(111)}$ equal to 0.257nm. The EDS spectrum further demonstrated that the particle in Fig. 6 (b) contains Nb (Fig. 6 (c)). The combined HRTEM and EDS spectrum strongly suggest that the finer precipitate shown in Fig. 6 (a) is consistent with NbC.

3.4.2 The observation of OR of SIP using TEM and HRTEM

The location and the OR of precipitated carbides with respect to the ferrite matrix have been analysed following the deformation at 850 °C followed by water quenching, shown in Fig. 7. The carbides in ferrite could be clearly identified by bright and dark-field imaging and the dark-field image taken from the $(1\bar{1}1)_{\text{NbC}}$ reflection from thin foil samples. Fig. 7 (a) and (b) show that the SIP is mainly located on the dislocations. The SADPs have been used to analyse the OR between SIP and the ferrite matrix. Figs. 7 (c) and (d) show that the carbides,

which are NbC, obey the orientation relationship with ferrite as $[\bar{1}10]_{\text{NbC}}//[\bar{1}11]_{\text{Ferrite}}$ and $(111)_{\text{NbC}}//(101)_{\text{Ferrite}}$.

High-resolution TEM (HRTEM) was used to investigate the nanometer-sized carbides. A lattice image of the ferrite matrix containing carbides is shown in Fig. 8 which was taken from the steel after deformation at 850°C. The inverse fast Fourier transformation (IFFT) was used for the carbide shown in Fig. 8 (b). The zone axis of the ferrite matrix and carbides was identified as $[111]_{\text{ferrite}}$ direction in Fig. 8 (b) parallel with the $[110]$ zone axis of carbides, i.e.: $[110]_{\text{NbC}}//[111]_{\text{ferrite}}$. Under this condition, the lattice image of the carbide has been identified with the $d_{(111)}$ spacing equal to 0.251nm. There are two carbides, shown in Fig. 8 (b), which have the cube-cube OR with two different growing along directions of $g(1\bar{1}1)$ and $g(\bar{1}11)$. The OR has been identified by the fast Fourier transformed (FFT) diffractogram shown in Fig. 8 (c). The NbC precipitates obey the orientation relationship with ferrite matrix as shown by $[\bar{1}10]_{\text{NbC}}//[1\bar{1}1]_{\text{Ferrite}}$ and $(111)_{\text{NbC}}//(\bar{1}01)_{\text{Ferrite}}$.

4 Discussion

4.1 Deformation temperature

The microstructure after deformation at different temperatures has been observed using OM and shown in Figs. 2 and 3. There were fully equiaxed prior-austenite grains for the rolling temperatures between 1100 °C and 1000 °C for Nb steel, which indicated that full recrystallization had occurred (Fig. 1). The partial recrystallization structure was obtained with the deformation temperature at 950 °C. With the deformation temperatures at 900 °C and 850 °C, the prior-austenite grains were fully elongated. The observed microstructures at different deformation temperatures fit well with the simulated full recrystallisation and non-recrystallisation regions for Nb steels.

There was no strain-induced precipitation (assuming precipitate diameter less than 20nm) after deformation at 1100 °C in the Nb steel. At 1000 °C, the percentage of strain-induced precipitates was 4.8 %, which was too little to result in a sufficient Zener pinning force to halt the recrystallization. Down to 950 °C, more than 50% of precipitates were smaller than 20nm in the Nb steel. The mixed microstructures observed at 950 °C in the Nb steels (Fig. 2) indicated that recrystallization occurred at the beginning and was then halted by the rapid increase in fine precipitate numbers. As the volume fraction of strain-induced precipitates increased with reducing the rolling temperatures to 850 °C, the recrystallization was fully prevented. The highest volume fraction of strain-induced precipitates was at 850 °C for Nb steel, which fits well to the calculation of the nose precipitated temperature at 840 °C by Eqs. (1) and (2) (Fig. 1).

Normally, the final step of thermomechanical processing (TMP) occurs in the fully austenite non-recrystallisation region to avoid the partial recrystallization structures and refine the final grain size. In this study, the deformation temperature at 850 °C for Nb steel resulted in non-recrystallized prior austenite microstructures. Moreover, depending on the simulated PTT curve (Fig. 1), specimens deformed at 850 °C exhibited more rapid precipitation kinetics than at other rolling temperatures. Therefore, the deformation temperature for Nb steel was optimised at 850 °C.

4.2 Calculation of the OR between SIP and austenite, subsequently transformed to ferrite

The SIP formed in austenite can be related to the ferrite by the OR between austenite and ferrite when observed at room temperature. There would be no OR between the SIP and the ferrite matrix if recrystallisation had occurred in the austenite grains prior to any subsequent phase transformation [23]. Therefore, to understand the OR between the SIP, austenite and

ferrite, the final deformation temperature should be lower than the $T_{5\%}$ but higher than the A_{r3} . In this study, the final deformation temperature of 850°C satisfied this requirement.

The OR between the SIP, austenite and ferrite will be discussed using the corresponding coordinate transformation matrix and Kurdjumov-Sachs (K-S) OR. Davenport et al. [24] indicated that the SIP and austenite have a cube-cube relationship, i.e.: $[010]_{\text{precipitate}}//[010]_{\text{austenite}}$, $(100)_{\text{precipitate}}//(100)_{\text{austenite}}$. From the above OR of SIP (determined as NbC) and austenite, the corresponding coordinate transformation matrix, M_{γ}^p can be readily determined as

$$M_{\gamma}^p = \begin{bmatrix} 1.25 & 0 & 0 \\ 0 & 1.25 & 0 \\ 0 & 0 & 1.25 \end{bmatrix} \quad (10)$$

Then, an arbitrary pair of parallel directions, r_p and r_{γ} , will be related by

$$r_p = M_{\gamma}^p r_{\gamma} \quad (11)$$

With the OR matrix, the pair of conjugate planes and direction of NbC parallel with the pair of $[110]$ and (111) of austenite can be deduced as $[110]_{\text{NbC}}//[110]_{\text{austenite}}$, $(111)_{\text{NbC}}//(111)_{\text{austenite}}$. It is well known that the austenite transforms to ferrite and obeys the Kurdjumov-Sachs (K-S) OR [25]: $[110]_{\text{austenite}}//[111]_{\text{ferrite}}$, $(111)_{\text{austenite}}//(110)_{\text{ferrite}}$. The OR between SIP of NbC and austenite transformation to ferrite can be further indicated as $[110]_{\text{NbC}}//[110]_{\text{austenite}}//[111]_{\text{ferrite}}$, $(111)_{\text{NbC}}//(111)_{\text{austenite}}//(110)_{\text{ferrite}}$.

The coordinate transformation matrix of NbC and ferrite is shown in Eq. (11), which indicates that the precipitates cannot have the same characteristics as the N-W OR (fcc/bcc system) [26].

$$M_f^{NbC} = \begin{bmatrix} 0.479 & -0.431 & -0.049 \\ 0.42 & -0.479 & -0.108 \\ 0.108 & 0.049 & 0.636 \end{bmatrix} \quad (12)$$

Face-centered cubic (fcc) precipitates in a body-centered cubic (bcc) ferrite can be indicated by $\{111\}_{\text{fcc}}//\{110\}_{\text{bcc}}$ and $\langle 110 \rangle_{\text{fcc}}//\langle 111 \rangle_{\text{bcc}}$. On a given (011) bcc plane (two-fold rotation axis), there are two $\langle 111 \rangle$ directions: $[11\bar{1}]_{\text{bcc}}$ and $[\bar{1}11]_{\text{bcc}}$, which are perpendicular. As there are six two-fold rotation axes of $\{110\}$ in bcc matrix, six variants of SIP and ferrite matrix OR can be derived, which are listed in Table 1. The OR between the precipitates and the matrix shown Figs. 7 and 8 are the same with the calculated OR from the corresponding transformation matrix shown in Table 1.

Thus, the calculated cubic-cubic OR between precipitate and austenite shows that the NbC precipitates were coherent or semi-coherent with the austenite matrix when formed by strain-induced precipitation. The OR between NbC and the matrix was retained in the ferrite following phase transformation on cooling, and this shows that there was a K-S OR between austenite and ferrite. Thus, these precipitates maintained their coherency/semi-coherency with the ferrite following transformation. This coherency/semi-coherency was only observed in the finer (<10nm) NbC precipitates, while the coarser (much greater than 10nm) precipitates were incoherent with the ferrite. Therefore, this shows that the NbC that forms in the austenite with a cubic-cubic OR are expected to be finer and more likely to contribute to the Zener pinning effect, and therefore strength enhancement.

5 Conclusions

1) The deformation temperature of 850°C is between the recrystallisation-stop temperature and the A_{r3} , indicative of deformation taking place in fully unrecrystallised austenite as well as obtaining the maximum volume fraction of SIP. The deformation temperature was higher than the A_{r3} , which indicates that the SIP had occurred in the fully

austenitic region.

- 2) The SIP has been examined via analyses of HRTEM and EDS. The results strongly suggest that these carbides are NbC which have a NaCl-type crystal structure and lattice parameters of 0.447 nm.
- 3) Finer (<10nm) NbC observed in the ferrite was coherent or semi-coherent with the matrix. Assuming a Kurdjumov-Sachs orientation relationship between the austenite and ferrite on transformation, this indicates that the NbC formed in the austenite as coherent or semi-coherent precipitates with a cube on cube orientation relationship between the NbC and the austenite. Given that the larger NbC observed was incoherent with the matrix, this suggests that the NbC that forms coherently or semi-coherently in the austenite with a cube on cube relationship will contribute more greatly to Zener pinning.

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Table 1
Six variants of NbC and Ferrite ORs in FCC/BCC system

Ferrite -NbC Variant	Plane BCC//FCC	Direction BCC//FCC
1a	$(011)_{\text{bcc}}// (111)_{\text{fcc}}$	$[\bar{1}\bar{1}\bar{1}]_{\text{bcc}}// [\bar{1}\bar{1}0]_{\text{fcc}}$
1b		$[\bar{1}\bar{1}1]_{\text{bcc}}// [\bar{1}\bar{1}0]_{\text{fcc}}$
2a	$(101)_{\text{bcc}}// (111)_{\text{fcc}}$	$[\bar{1}\bar{1}1]_{\text{bcc}}// [\bar{1}\bar{1}0]_{\text{fcc}}$
2b		$[\bar{1}\bar{1}\bar{1}]_{\text{bcc}}// [\bar{1}\bar{1}0]_{\text{fcc}}$
3a	$(110)_{\text{bcc}}// (111)_{\text{fcc}}$	$[\bar{1}\bar{1}\bar{1}]_{\text{bcc}}// [\bar{1}\bar{1}0]_{\text{fcc}}$
3b		$[\bar{1}\bar{1}1]_{\text{bcc}}// [\bar{1}\bar{1}0]_{\text{fcc}}$
4a	$(\bar{1}10)_{\text{bcc}}// (111)_{\text{fcc}}$	$[\bar{1}\bar{1}\bar{1}]_{\text{bcc}}// [\bar{1}\bar{1}0]_{\text{fcc}}$
4b		$[111]_{\text{bcc}}// [\bar{1}\bar{1}0]_{\text{fcc}}$
5a	$(\bar{1}01)_{\text{bcc}}// (111)_{\text{fcc}}$	$[\bar{1}\bar{1}\bar{1}]_{\text{bcc}}// [\bar{1}\bar{1}0]_{\text{fcc}}$
5b		$[\bar{1}\bar{1}1]_{\text{bcc}}// [\bar{1}\bar{1}0]_{\text{fcc}}$
6a	$(0\bar{1}1)_{\text{bcc}}// (111)_{\text{fcc}}$	$[111]_{\text{bcc}}// [\bar{1}\bar{1}0]_{\text{fcc}}$
6b		$[\bar{1}\bar{1}\bar{1}]_{\text{bcc}}// [\bar{1}\bar{1}0]_{\text{fcc}}$