ND//<111> Recrystallization in Interstitial Free Steel: The Defining Role of Growth Inhibition

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Hot band, fully recrystallized, interstitial free (IF) steel samples were cold rolled to 10–80% thickness reductions. After characterizing the developments in deformed microstructures, 50–80% deformed samples were fully recrystallized at 650°C. Though different annealing times were used, use of a relatively lower annealing temperature and repeated trials ascertained absence of significant post-recrystallization grain coarsening. Recrystallization brought in a steady improvement in γ-fibre (ND//<111>) and drop in θ-fibre (ND//<100>). The only exception was 80% prior deformed microstructure, where the trend was reversed: enforcing a drop in texture estimated normal anisotropy or \( \tilde{\gamma} \) value. The study brought out growth inhibition of recrystallized γ-fibre grains, caused by non-γ-fibre bands and by orientation pinning of recrystallized γγ boundaries, as the mechanism behind the observed trend reversal.

KEY WORDS: IF steel; recrystallization; deformation; texture; stored energy; orientation pinning; lankford constant.

1. Introduction

The formability of low carbon (LC) steels, in general, and interstitial free (IF) steels, in particular, are related to the formation of strong γ-fibre (ND//<111>) recrystallization texture.1–18) The development of strong γ-fibre recrystallization texture is often considered from the mechanisms of preferred nucleation (oriented nucleation - ON) and/or growth (oriented growth - OG) of γ-fibre grains from the deformed grains/bands of similar orientation(s).2,4–7,9–11,17–19) The classical definition of ON and OG, though nearly half a century in circulation, has one problem. They were originally designed to address the recrystallization mechanisms, but fail to provide a clear demarcation between nucleation and growth.2,4–7,9–11,20,21) A recrystallized grain can be distinguished from rest of the deformed matrix by its strain-free nature and a minimum size.10,19,22,23) This is the only way to distinguish a recrystallized grain from a large sub-grain.10) The ‘minimum’ size criterion, on the other hand, brings an interesting contradiction. To achieve a minimum size, and thus being ‘qualified’ as a recrystallized grain, both nucleation and limited or local growth could have been involved. To avoid this contradiction, an alternative approach is to use ‘frequency advantage’ and/or ‘size advantage’ of the recrystallized grains/bands. In other words, strengthening of a crystallographic texture ‘i’ by recrystallization can be caused by the presence of more ‘i’ grains and/or by the fact that ‘i’ grains are larger in size. This can be formulated10,20,24–26) as:

\[
\alpha = \frac{i_{\text{Re}}} {i_{\text{Random}}} 
\]

\[
\beta = \left( \frac{d_{i}} {d_{\text{average}}} \right)^3
\]

where \( i_{\text{Re}} \) and \( i_{\text{Random}} \) are the respective number fractions of ‘i’ oriented grains in the recrystallized material and in a random textured material; \( d_{i} \) and \( d_{\text{average}} \) are the mean grain sizes for ‘i’ oriented grains and average grains. It is to be noted that \( \alpha \) can be affected by preferred nucleation and/or by micro-growth advantage/selection; while rationales for ‘size advantage’ may range from faster nucleation kinetics to overall growth advantage/disadvantage.10,20,24–26)

The γ-fibre recrystallization texture, in LC steels, is expected to be strengthened by prior reductions.9,10,17) This, of course, would depend on the prior deformation grain size and texture and also on the developments in deformed microstructures.10) What is not very apparent, in the domain of published literature, are the possible ‘limitations’, if any, in the exact developments of γ-fibre recrystallization texture with prior deformation. This, on the other hand, defined the main objective of the present study.

2. Experimental Methods

Hot rolled and fully recrystallized IF steel (4.5 mm thickness), with the chemical composition shown in Table 1, was used in the present study. The hot band was cold rolled in a
laboratory rolling mill to 10, 20, 40, 50, 60, 70 and 80% reductions in thickness. The 50, 60, 70 and 80% deformed samples were annealed in a salt-bath furnace at 650°C for various times. The lower annealing temperature and multiple trials (or use of different annealing periods) ascertained complete recrystallization with relatively insignificant post-recrystallization grain coarsening.

The deformed and recrystallized samples were subjected to detailed characterization through X-ray and electron diffraction. The former was taken on the mid-thickness section of the rolling plane (plane containing rolling direction (RD) and transverse direction (TD)), while mid-thickness section of the long-transverse plane (plane containing RD and normal direction (ND)) was used for EBSD (electron backscattered diffraction). In both cases, samples were prepared by standard metallographic polishing followed by electro-polishing at −20°C and at 11 volts DC, using an electrolyte containing methanol and perchloric acid in 80:20 ratio.

X-ray diffraction (XRD) measurements were taken in a Panalytical X’Pert PRO MPD system. Peak profiles of (222) (γ-fibre (ND//<111>)) and (200) (θ-fibre (ND//<100>)) were taken using a step size of 0.02° and a time period of 60 s at each step. The XRD system was used in the line focus mode with 0.02 rad soller slits on both incident and diffracted side and a point detector. The analysis, of the peak profiles, involved multiple aspects: ranging from relatively simple peak broadening (full width at half maximum - FWHM) to more involved micro-stress and X-ray resolution function measurements. The discrete data points of the line profiles were fitted with a pseudo-Voigt (pV) function using X’Pert Highscore software and then FWHM was determined from the fitted function. The data points usually do not follow a pure Gaussian or pure Lorentzian distribution and hence it was necessary to use the pV function to get accurate values of FWHM.

Bulk X-ray texture measurements were obtained, in a Panalytical X’Pert PRO MRD system with X-ray lens and multi-channel solid state area detector, by inversion of 4 or more scans. Step size of 0.5 μm was used while all the EBSD scans were taken under identical beam and video conditions. Measurement points above 0.1 CI (confidence index: an index of accuracy in automated indexing) were cropped or separated manually, so that subsequent analysis, of the in-grain strain localizations were quantified in terms of ‘cropped’ grain average misorientation.

The dislocation density (ρ) was determined according to the methodology described by Groma. The second and fourth order restricted moments of the X-ray line profiles has the form:

\[
M_0(q) = \frac{1}{\pi^2 \varepsilon_F} q - \frac{L}{4\pi^2 \varepsilon_F^2} + \frac{\Lambda(\rho) \ln(q / \rho_1)}{2\pi^2} \quad (3)
\]

\[
M_2(q) = \frac{1}{3\pi^2 \varepsilon_F^2} q + \frac{3\Lambda^2(\rho^2)}{4\pi^2 q^2} - \ln(q / \rho_1) \quad (4)
\]

where \( q = \frac{4\pi}{\lambda} (\sin \theta - \sin \theta_0) \), \( \varepsilon_F \) is the average column length and \( \theta \) is the exact Bragg angle. The dislocation densities were calculated by fitting Eq. (3) to the linear part of \( M_0(q) \).

This method provides internal checking, since that value of dislocation density was chosen for which second order and fourth order moment gave almost same particle size. Momentum method has been effectively used in the past to find dislocation density in deformed LC steels.

The degree of peak resolution of the K_α/K_α doublet has been described quantitatively using the X-ray resolution function (XRD-RF). XRD-RF is defined as ((I_min-I_b)/(I_kα−I_0)): where I_min is the minimum intensity of the K_α/K_α doublet, I_kα is the maximum peak intensity of the K_α peak and I_0 is the background intensity. XRD-RF has been used, effectively, in the past in quantifying the recovery/recrystallization. The present study, however, used XRD-RF to bring out difference in dynamic recovery, if any, between γ and θ fibres.

TSL (Tex Scan Ltd.) EBSD systems attached on FEI Quanta-200HV and FEI Quanta-3d FEG (field emission gun) SEMs (scanning electron microscope) were used. For each sample, at least an area of 2 mm × 2 mm was covered in 4 or more scans. Step size of 0.5 μm or less was employed, while all the EBSD scans were taken under identical beam and video conditions. Measurement points above 0.1 CI (confidence index: an index of accuracy in automated indexing) were used for subsequent analysis. Data points above 0.1 CI represent at least 95% accuracy. A total of 1,500 grains, representing different deformation conditions, were ‘cropped’. The ‘cropping’ was a tedious, albeit necessary, procedure: for details the reader may refer elsewhere. Visible grains/deformed bands of γ and θ fibres were cropped or separated manually, so that subsequent analysis is representative and appropriate. From each ‘cropped’ grain average misorientation was estimated, while the in-grain strain localizations were quantified in terms of number of high angle boundaries (>15° misorientation) along RD per 100 μm length inside the respective ‘cropped’ grains. The ‘cropped’ data was also used to estimate thicknesses (along ND) of the deformed bands.

3. Results

Figure 1 collates the X-ray ODFs and EBSD inverse pole figure (IPF) maps of the IF steel samples at various stages.
of deformation. The hot rolled (HR) IF steel had a weak transformation texture. This strengthened with the increase in strain: texture index increased from 1.5 (hot band) to 3.4 (80% deformed). With the increase in thickness reduction, the grain shapes had also undergone expected changes with concurrent developments in misorientations. Figure 2 expands on the ODFs of Fig. 1 by providing information on the volume fractions of the three important fibres: $\gamma$ (ND//<111>), $\alpha$ (RD//<110>) and $\theta$ (ND//<100>). As shown in the Fig. 2, volume fractions of $\gamma$ and $\alpha$ fibres increased with increase in strain, while the volume fraction of $\theta$-fibre did not change significantly.

Developments in the stored energy of cold work is an important aspect, especially in terms of recrystallization behaviour, of any deformed microstructure. Figure 3 brought this out through simple micro-hardness measurements (Fig. 3(a)) and different parameters of X-ray peak profiles (Figs. 3(b)–3(d)). The typical LC steel texture is often generalized as $\gamma$, $\alpha$ and $\theta$ fibres. It is important to note that: (i) the fibres have overlap(s) in Euler space and (ii) $\gamma$ and $\theta$ fibres are the so-called ND fibres and hence has corresponding XRD peaks. The maximum misorientation was kept within 15° (or 11° Gaussian spread for ODF estimated volume fractions) to address (i). This, on the hand, is reflected in the relatively lower numbers for fibre volume fractions. Finally, the $\gamma$ and $\theta$ fibres were considered for establishing the frequency and size advantages, while the $\alpha$ fibre was avoided in such estimates. $\gamma$-fibre and $\theta$-fibres were selected based on the fact that both can be directly measured by EBSD and XRD. The ratio of strength of $\gamma$ and $\theta$ fibres has also been used in the past as a representative of normal anisotropy or $\overline{\gamma}$. Figures 3(b)–3(d) show higher stored energy (in terms of both peak broadening and estimated dislocation density) and stronger dynamic recovery (in terms of X-ray resolution function) for deformed $\gamma$-fibre grains. As discussed in previous studies, the stored energy difference is typically caused by the preferred appearance of grain interior strain localizations. As in Fig. 4, this was estimated in terms of more frequent high angle boundaries and correspondingly larger misorientation in deformed $\gamma$-fibre grains.

As discussed earlier in the introduction, the primary objective of this study was not to describe the developments in deformed microstructures, but to explore the possible 'limitations' in $\gamma$-fibre recrystallization texture developments. For this, 50–80% deformed IF steel samples were subjected to complete recrystallization. Though the recrystallization temperature was kept at 650°C, trials with different annealing time ascertained complete recrystallization with relatively insignificant post-recrystallizations grain coarsening. As shown in Fig. 5, the crystallographic texture and microstructure of the recrystallized samples differed, significantly, with prior deformation. For example, the tex-
ture indices went up from 3.3 to 5.74, while average recrystallized grain size decreased from 27 to 13 μm. The most significant changes were, however, in the estimated volume fractions of the γ and θ fibres. The respective fibres had a relative increase (γ-fibre: 0.28 → 0.37) and drop (θ-fibre: 0.05 → 0.02) till 70% prior deformation and then the trend was exactly reversed. From 70 to 80% prior reduction, the volume fractions of γ and θ fibres dropped (γ-fibre: 0.37 → 0.33) and increased (θ-fibre: 0.02 → 0.04). As in Fig. 6(a), the changes in the ratio of γ and θ fibres also brought in cor-
responding changes in texture estimated $\tilde{r}$ values.

The remarkable trend reversal, see Fig. 6(a), was then viewed from frequency and size advantage of recrystallized $\gamma$ (Fig. 6(b)) and $\theta$ (Fig. 6(c)) fibre grains. As shown in the figures, the trend reversals (below and above 70% prior reduction) was caused by a combination: (i) reduced size advantage of recrystallized $\gamma$ (Fig. 6(b)) and (ii) increased frequency/size advantage for $\theta$-fibre (Fig. 6(c)). (i) was of responsible for reduced volume fraction of $\gamma$ grains, while (ii) caused near doubling of $\theta$ volume fraction. The partially recrystallized microstructures (annealing time - 10 min at 650°C) were explored to comprehend the possible mechanism(s). As in Fig. 7, the growth of recrystallized $\gamma$-grains was clearly restricted by non-$\gamma$ fibre bands and by recrystallized $\gamma-\gamma$ grain boundaries. Such clear growth inhibition of recrystallized $\gamma$ had two effects: it restricted size advange for recrystallized $\gamma$ fibre grains and allowed sufficient time and space for $\theta$-fibre nucleation and growth. While the first point is apparent in the high resolution EBSD images (as in Fig. 7), the second appears slightly speculative and both are discussed further in the next section.

4. Discussion

$\gamma$-fibre recrystallization, in LC steel, is a relatively well documented subject. It is generally acknowledged that the preferred nucleation, due to higher stored energies of deformed $\gamma$-fibre grains, plays a critical role. Preferred appearance of grain interior strain localizations is often attributed as the mechanism behind such stored energy advantage. It is important to point out, at this stage, that though the role of stored energy advantage is universally acknowledged, debates still do continue on the relative effectiveness of micro-growth advantage and selection. In bcc steel, the $27^\circ$<$110$ boundary is expected to have higher mobilities. Presence of such boundaries in the deformed $\gamma$-fibre grains, or in the immediate neighbourhood, may provide a local or limited growth advantage for recrystallized $\gamma$-grains. Such micro-growth advantage is, however, very difficult to quantify. The preferred nucleation, as quantified by typical microscopic tools, may include components of micro-growth advantage: the so-called ‘vanishing evidence’. Frequency advantage would, of course, bring out signatures of all aspects of preferred nucleation: both stored energy and micro-growth advantage. As in Fig. 6(b), frequency advantage of recrystallized $\gamma$-fibre grains actually improved, from 2.0 to 2.7, as the prior deformation increased from 70 to 80%; while the size advantage dropped from 1.2 to 0.7. These are average estimates, the exact numerical values/distributions are expected to account for the observed drop (Fig. 6(a)) in recrystallized $\gamma$-fibre fraction.

Frequency advantage or preferred nucleation can be further quantified through the so-called ‘Nucleation Factor’. This concept was originally proposed in a study on cube recrystallization in fcc aluminium, and subsequently extended to bcc LC steel. It was proposed that a recrystallization texture of a component ‘i’ can be seen as:

$$A_i = \frac{N_i d_i}{\lambda_i} \quad \text{(5)}$$

where $A_i$, $d_i$, and $\lambda_i$ are the respective area fraction, average recrystallized grain size and deformed band separation for ‘i’ orientation: all estimated along ND in a long transverse plane (containing RD and ND). $N_i$ defines the nucleation factor: the number of ‘i’ recrystallized grain per ‘i’ deformed band estimated by linear intercepts along ND. Equation (5) is purely geometrical, but $N_i$ can provide numerical estimates of preferred nucleation.

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1It needs to be noted that past studies had shown that changes in texture estimated $r$-bar scales well with conventional mechanically tested $r$-bar values.

2Similarly, the estimated increase in $\theta$ frequency/size advantage should correlate with the changes in the recrystallized $\theta$-fibre volume fraction.
\( \gamma \) and \( \theta \) fibre recrystallized grains are collated in Table 2. For 70–80% prior deformation – the so-called regime of trend reversal (Fig. 6(a)), the table shows relatively insignificant changes (or a small increase) for \( \gamma \)-fibre nucleation factor; while a small but clear increase in \( \theta \)-fibre nucleation factor was also noted. It needs to be pointed out that stored energy difference, between \( \gamma \) and \( \theta \), remained nearly identical or even increased (depending on the characterization/estimation method: Figs. 3(b)–3(c) and 4(b)–4(c)) between 70 and 80% cold work. In other words, the small but significant increase in \( \theta \)-fibre nucleation factor does not appear addressable from a straight-forward argument on the driving force of recrystallization.

This is an interesting situation. As the separation between deformed \( \gamma \)-fibre bands (\( \lambda_i \)) reduces\(^3\), even with the same magnitude of nucleation factor an increase in recrystallized \( \gamma \)-texture is expected.\(^{18,22,25,47}\) This was not the case: see Figs. 5 and 6(a). The clear drop in \( \gamma \)-fibre recrystallization, between 70 and 80% prior reductions, were caused by growth inhibition of recrystallized \( \gamma \)-fibre grains. This is reflected on the reduced size advantage of recrystallized \( \gamma \)-fibre grains (Fig. 6(b)), while frequency advantage actually increased. The growth inhibition of the recrystallized \( \gamma \)-fibre is brought out pictorially in Fig. 7. As shown in the

\(^{24} \rightarrow ^{14} \mu m \) as the deformation increased from 50 to 80%.
Fig. 7. 80% deformed and partially recrystallized IF steel sample (annealing time: 10 min at 650°C) showing pinning of recrystallized γ-fibre grains by other γ-fibre grain and non-γ-fibre bands. Included are a relatively large area scan plus a magnified image showing clear evidence of growth inhibition.

Fig. 6. (a) Volume fraction (as measured by X-ray ODF for 11° Gaussian spread) of recrystallized γ-fibre/θ-fibre and texture estimated r-bar and frequency/size (Eqs. (1) and (2)) advantage parameters of recrystallized (b) γ and (c) θ grains. (a)–(c) are plotted as a function of prior thickness reductions. A maximum misorientation of 15° from the ideal fibre orientations was considered, while hot band microstructure was taken as random to solve Eq. (1). For automated grain classification, the presence of a continuous boundary above 5° misorientation was used: the relatively small misorientation criterion was essential to bring out subtle differences in frequency and size advantages.

Table 2. Nucleation factor, \( N_i \), for recrystallized γ and θ fibre grains: Eq. (5). \( \lambda_i (\mu m) \) was estimated using the deformed microstructures, while \( A_i \) (fraction) and \( d_i (\mu m) \) were measured from fully recrystallized structures.

| Percentage Deformation | γ-fibre | θ-fibre |
|------------------------|---------|---------|
| Prior Deformation      | \( \lambda_i \) | \( d_i \) | \( A_i \) | \( N_i \) | \( \lambda_i \) | \( d_i \) | \( A_i \) | \( N_i \) |
| 50%                    | 24      | 0.2764  | 0.286 | 52     | 24.83 | 0.0445  | 0.093 |
| 60%                    | 21      | 0.3006  | 0.317 | 29     | 17.44 | 0.0336  | 0.055 |
| 70%                    | 17      | 0.3731  | 0.345 | 25     | 12.65 | 0.0195  | 0.038 |
| 80%                    | 14      | 0.3266  | 0.352 | 22     | 11.83 | 0.0349  | 0.064 |

grains, extremely ‘sluggish’ and even impossible at the lower annealing temperatures. Such bands, on the other hand, appear to sub-divide the recrystallization microstructure, see Fig. 7, and possibly account for the time and space for θ-fibre recrystallization. The other source of growth inhibition was impingement of recrystallized γ-fibre grains. It is apparent that multiple sources of recrystallization, within close proximity, can lead to such growth inhibition: an effect often termed as orientation pinning. Yet another source of growth inhibition is the presence of fine precipitates (carbides and carbo-nitrides of titanium – TiC and Ti(C,N) in the present case). A dense dispersion of fine precipitates strongly suppresses the growth of recrystallized grain through pinning force, exerted by the particles on the boundary mobility, in the recovered matrix. The driving force for the growth of the newly formed recrystallized grains is the grain boundary surface free energy, which is substantially
smaller in magnitude than the driving force for primary recrystallization. Thus, Zener drag force\(^{10,19}\) on the boundary mobility is expected to be even more dominating in growth inhibition. Though, the relative effectiveness of these growth inhibition sources can be argued, it is clear from the present study that growth inhibition has a defining or ‘limiting’ role in \(\gamma\)-fibre recrystallization texture.

5. Summary

- During deformation, both \(\gamma\) (ND/\(<111>\)) and \(\alpha\) (RD/\(<110>\)) fibres increased, while there was insignificant changes in \(\theta\) (ND/\(<100>\)) fibre. With increasing deformation (0–70%), a clear and increasing difference in the estimated stored energies of \(\gamma\) and \(\theta\)-fibre grains/ bands was also observed. However, stored energy difference, between \(\gamma\) and \(\theta\) fibre, was relatively unaffected as rolling reductions increased from 70 to 80%.

- After recrystallization, \(\gamma\)-fibre increased till 70% deformation then dropped, while the reverse trend was observed for \(\theta\) fibre. The combination brought in an interesting pattern in texture estimated \(r\)-bar values. It increased till 70% prior deformation and then dropped. In other words, a significant trend reversal, below and above 70% reduction, in \(\gamma\) and \(\theta\) fibre recrystallization was captured in the present study.

- The trend reversal appeared to be associated with two factors: (i) reduced size advantage for recrystallized \(\gamma\)-fibre and (ii) increased frequency/size advantage for recrystallized \(\theta\)-fibre. These affected the changes in the respective volume fractions and thus defined the final recrystallization texture. (i) was related to growth inhibition of recrystallized \(\gamma\)-fibre grains: pinning by non-\(\gamma\)-fibre bands undergoing extended recovery and by impingement or orientation pinning by recrystallized \(\gamma\)-fibre grains. Extended pinning by non-\(\gamma\) fibre bands was stipulated to sub-divide the recrystallizing volume and thus offer space and time for \(\theta\) fibre recrystallization. The study brings out the defining role of growth inhibition of recrystallized \(\gamma\)-fibre grains as the ‘limiting’ parameter.

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