Strain Localizations in Ultra Low Carbon Steel: Exploring the Role of Dislocations

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Ultra low carbon steel samples were near plane strain deformed at different strains, strain rates and temperatures. Density of grain interior strain localizations in $\gamma$ (ND//<111>) and $\theta$ (ND//<100>) fibres were evaluated against micro-stress estimates through X-ray line profile measurements. The patterns were remarkably different between the fibres. The increase in strain localizations were associated with increased dislocation density. This effect was more pronounced in $\gamma$-fibre. In $\theta$-fibre, however, the peaks were increasingly more asymmetric and dislocation substructures were stipulated to have lesser recovery. Discrete dislocation dynamics simulations for single crystal pure iron also brought in different behavior for $\gamma$ and $\theta$ fibres: increase in dislocation density in the former was estimated to be ~5 times more. A combination of textural softening and large/positive increase in dislocation density appears to justify the preference for strain localizations in $\gamma$-fibre.

KEY WORDS: deformation; low carbon steel; dislocation; X-ray diffraction; X-ray line profile analysis; strain localizations; warm working.

1. Introduction

Low carbon steels became popular because of their excellent deep drawability.1–10) This, often generalized in terms of normal anisotropy or the Lankford value,1,2,9,10) is attributed to the presence of strong $\gamma$-fibre (ND//<111>) or corresponding absence of $\alpha$ (RD//<110>) and $\theta$ (ND//<100>) fibres.1,2,9,10) In a typical TMP (thermo-mechanical processing),10) the desired changes in crystallographic texture are obtained through maximizing $\gamma$-fibre through recrystallization. Though, controversies remain on the effectiveness of preferred nucleation vs. micro-growth advantage/selection in such recrystallization,1,2,4–16) where the preferred/selective appearance of ‘grain interior strain localizations’ is often considered as the governing factor.17–22)

An interesting aspect of deformed low carbon steels is the formation of ‘grain interior strain localizations’.18–20,22,23) Borrowing terminology from deformed fcc material,24) these were classified as first generation micro-bands.19) They have been found to have a preference for grains/orientations with high Taylor factor ($M$)19,20) and/or grains/orientations with textural softening: negative $dM/d\varepsilon$.25) where $\varepsilon$ is the true strain. A previous study26) has reported clear evidence of improving the preferred appearance of ‘grain interior strain localizations’ through warm plane strain deformation. This was rationalized in terms of enhanced role of textural softening.

This study is a continuation of a previous study26) which used extensive EBSD (electron backscattered diffraction) data and highlighted preferred formation of ‘grain interior strain localizations’ at different warm working conditions. The present study looked at the same samples and also used some of the past EBSD data, but involved extensive X-ray line profile analysis to bring out possible roles of localizations and recovery in $\gamma$ and $\theta$ fibres. In addition, discrete dislocation dynamics simulations were carried out to study differences, if any, in dislocation generation between the dominant crystallographic fibres: $\gamma$ (ND//<111>) fibre and $\theta$ (ND//<100>) fibre.

2. Experimental Methods

Hot rolled fully recrystallized ULC (ultra low carbon) steel, with the chemical composition given in Table 1, was subjected to plane strain compression (PSC) tests. The tests were conducted by strip PSC set-up in an MTS deformation simulator. In a typical strip PSC set-up, the heated samples were placed between two heated dies and then compressed employing boron nitride powder as lubricant at elevated temperatures and oil at room temperature. Friction

| C   | Mn | S   | P   | Si  | Al  | Ti  | N   | Fe  |
|-----|----|-----|-----|-----|-----|-----|-----|-----|
| 0.0014 | 0.145 | 0.0002 | 0.001 | 0.022 | 0.029 | <0.001 | 0.0017 | balance |

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between die and sample partially restrains material flow along transverse direction (TD). The present study involved a total of 24 different deformation conditions: three strains (40, 60 and 70% reductions), four different temperatures (20, 200, 400 and 600°C) and two strain rates (0.1 and 1 s⁻¹). Samples were quenched immediately after deformation in water at temperature 11°C. Though this does not rule out possibilities of post-deformation recovery, the quenched samples did not carry any signs of recrystallization. Afterwards, the deformed samples were subjected to detailed EBSD and XRD (X-ray diffraction) characterizations. The EBSD data were acquired from the mid-thickness section of the long-transverse plane (plane containing rolling direction (RD) and normal direction (ND)), while the X-ray data were acquired in the mid-thickness section of the rolling plane (plane containing RD and TD). Samples for EBSD and X-ray measurements 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.

Details of EBSD measurements and analysis are reported elsewhere. Individual γ and θ fibre grains, within 15° of ideal texture components, were identified. They were then cropped individually: a total of 3 800 grains, involving 24 deformation conditions, were cropped. The analysis, used in the present study, includes (i) estimates of ‘grain interior strain localizations’ in γ-fibre and θ-fibre grains and (ii) grain average misorientation or GAM values for the respective orientations. (i) was measured along RD, while GAM represented average point-to-point misorientation within a grain.

XRD measurements were carried out employing a Panalytical MRD system. Use of X-ray lenses and multi-channel textures 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.

\[ W = \frac{3}{2} E_{\text{hl}} \left( \frac{(B_{γ}^2 - B_{θ}^2) / 4 \tan^2 \theta}{1 + 2v_{\text{hl}}} \right) \]  

where \( W \) is the stored energy of cold/warm work, \( E_{\text{hl}} \) and \( v_{\text{hl}} \) are the orientation dependent Young’s modulus and Poisson ratio respectively. These were taken as 283.286 GPa and 0.368 for γ-fibre and 131.578 GPa and 0.218 for θ-fibre respectively.29) \( B_{γ} \) and \( B_{θ} \) are the measured full width at half maximum (FWHM) values for the deformed and recrystallized samples respectively and θ is the Bragg angle.

The dislocation density (ρ) and dislocation arrangement parameter (\(<s^2>\)) were determined according to the methodology described by Groma.30,31) According to Wilson32) and Groma,30 the second and fourth order restricted moments of the X-ray line profiles have the form:

\[ M_s(q) = \frac{1}{π} \left\langle \frac{L}{K} \right\rangle \ln \left( \frac{q}{q_b} \right) \]  

where \( q = \frac{4\pi}{\rho} \left( \sin θ - \sin θ_0 \right) \), \( e_F \) is the average column length33) or the area weighted particle size measured in the direction of the diffraction vector g, K is the Scherrer constant, \( L \) is the so called taper parameter, \( \left\langle ρ \right\rangle \) is the average value of dislocation density, \( q_b \) and \( q_0 \) are the fitting parameter not interpreted physically, \( \left\langle ρ^2 \right\rangle \) is the average of the square of the dislocation density, \( Λ \) is a geometrical constant describing the strength of the dislocation contrast and \( θ_0 \) is the exact Bragg angle. The dislocation densities are calculated by fitting Eq. (3) to the linear part of \( \frac{M_4(q)}{q^2} \). This method provides internal checking, since that value of dislocation density is chosen for which second order and fourth order moment gives almost same particle size, \( <s^2> \) is determined from the third order restricted moment30) given as:

\[ M_3(q) = -6 <s^2> \ln \left( \frac{q}{q_1} \right) \]  

The degree of peak resolution of the K\(_{α1}/K\(_{α2}\) doublet has been described quantitatively using the X-ray resolution function (XRD-RF) \((I_{min} - I_0)/(I_{max} - I_0)\) where \( I_{min} \) is the minimum intensity of the K\(_{α1}/K\(_{α2}\) doublet, \( I_{max} \) is the maximum peak intensity of the K\(_{α1} \) peak and \( I_0 \) is the background intensity. XRD-RF have been used, effectively, in the past in quantifying the recovery.28,34–36)

Bulk X-ray texture measurements were obtained by inversion of 4 incomplete pole figures and using the standard series expansion method37) and the software MTM-TFM.38) The orientation distribution functions (ODF) were calculated. The standard \( θ_2 = 45° \) ODF section37,39) were used along with the values of maximum ODF intensity and texture indices. The later was estimated as \( f_{I(p)}d^2g : f_{I(g)} \) representing the ODF intensities. Texture index can be used as an estimate for relative texturing or anisotropy. Texture simulations, involving the program MTM-Taylor,38) were carried out to estimate the Taylor factor (M) and textural softening/hardening (\( \pm dM/dε \)) for the respective fibres. A small strain increment (\( dε \)) of 0.001 was used under near plane strain deformation and full constraint Taylor approach.

This study also involved discrete dislocation dynamics (DD) simulations using the software ParaDis.\(^{40,42}\) The inputs were possible orientations of \( γ \) and \( θ \) fibres, orientation sensitive Young’s modulus and Poisson ratio of the respective fibres\(^{29}\) and an initial random dislocation array of 10\(^6\) m\(^{-2}\). Random dislocation tangles were discretized into segments. The force on each segment was then calculated using the Peach-Koehler formulation\(^{40,42}\) and the segments moved according to a pre-specified mobility law and boundary conditions. The DD simulations are inherently computation intensive and hence the plastic strains were restricted to \( 1–5×10^{-7} \); imposed in 50 000 steps under plane strain condition.

3. Results

Figure 1 collates the results of bulk crystallographic tex-
ture developments at different deformation conditions. Apart from some minor changes in texture indices\(^1\) and also changes in the relative intensities along the respective fibres, deformation under the same strain (but different strain rate and temperature) did not affect the overall crystallographic textures significantly. More specifically, the volume fractions (taken at 11° or higher Gaussian spreads) of the three major fibres (\(\gamma, \alpha\) and \(\theta\)) remained similar. Though \(M\) and \(dM/d\varepsilon\) vary within a fibre (see Fig. 2), \(\gamma\)-fibre typically has higher \(M\) and more textural softening than \(\theta\)-fibre. This may justify\(^25\) preferred formation of strain localizations in the \(\gamma\)-fibre; but does not provide a ready explanation for the observed\(^26\) changes in the relative appearance of ‘grain interior strain localizations’. As reported\(^26\) in a previous study, at the intermediate working temperatures 70% plane strain compressed \(\gamma\)-fibre grains had noticeably more strain localizations than \(\alpha\)-fibre or \(\theta\)-fibre grains.

Preferred appearance of strain localizations, in deformed \(\gamma\)-fibre grains, does play an important role during recrystallization.\(^17\)–\(^22\) In this study, appearances of strain localizations were related to the number of grain interior high angle boundaries. The latter quantity was related to different parameters obtained from X-ray peak profile estimates: to bring out roles, if any, of dislocations on the preferred appearance of ‘grain interior strain localizations’. As shown in the Fig. 3, the features of the deformed microstructures, which changed remarkably with deformation conditions were in-grain misorientations (the GAM values) and the relative presence of the ‘grain interior strain localizations’. The grain interior strain localizations were estimated as number

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\(^1\)For example: texture indices of the 70% deformed sample dropped with increase in working temperatures.
of high angle boundaries, along rolling direction (RD) per 100 μm length. GAM and estimated number of grain interior high angle boundaries scaled, almost linearly, for both fibres: a similar scaling factor or slope of 0.02±0.001.

As pointed out in the introduction, the aim of this study was to exploit data on X-ray line profiles: to bring out roles, if any, of dislocations on the preferred appearance of ‘grain interior strain localizations’. The XRD information is summarized in Figs. 4 to 7. Data start with relatively simple estimates of stored energy of cold work – see Fig. 4. Stored energy increased with increased frequency of strain localizations: the increase being ~2.5 times more in γ-fibre than in θ-fibre. However, two points need to be noted. (i) Though the linear scaling between strain localizations and stored energy appear reasonable for γ-fibre (Fig. 4(a)), the same is rather questionable for θ-fibre (Fig. 4(b)). (ii) The Stibitz formula, Eq. (1), is based on the assumption of elastically isotropic material with random orientations. Even after using the orientation dependent Young’s modulus and Poisson ratio, the validity of stored energy estimates in an anisotropic material is debatable.

It was hence decided to use more elaborate micro-stress measurements, the so-called momentum method: Eqs. (2)–(4), for measuring dislocation density and dislocation arrangement parameter \(<s^2>\). The former is shown in Fig. 5. As shown in the Fig. 5, the increase in dislocation density also approximated, linearly, with increase in number of strain localizations. However, the slope for γ-fibre was ~3.6 times that of θ-fibre. Dislocation arrangement parameter \(<s^2>\), Fig. 6, is a measure of asymmetry of X-ray peak profile. The asymmetry is expected to vanish with equal population of dislocations with opposite signs of Burger vector. As in Fig. 6, \(<s^2>\) was more in θ-fibre. \(<s^2>\) for both γ and θ fibres increased with increased presence of strain localizations: increase being ~3 times more in θ-fibre.

Dislocation structures can also be affected by recovery. It is impossible to separate plastic deformation and dynamic recovery and also difficult to quantify recovery through conventional property/microstructure measurements. XRD data provides an alternative in the form of XRD-RF or X-ray resolution function. In X-ray diffraction, a fully recrystallized structure has appropriate separation of the doublets \(K_{α1}\) and \(K_{α2}\) and corresponding values of XRD-RF. Plastic deformation and associated defects fudge such
separation and increase XRD-RF values, while recovery or formation of lower energy dislocation sub-structures is expected to enhance the doublet separation and reduce XRD-RF. As shown in Figs. 7(a) and 7(b), in \( \gamma \)-fibre XRD-RF did increase with increased numbers of grain interior strain localizations; while in \( \theta \)-fibre the scope/quantum of increase was much less and a clear pattern between XRD-RF and increase in strain localizations did not emerge. In a fully recrystallized structure, estimated XRD-RF values were 0.079 for \( \gamma \)-fibre and 0.17 for \( \theta \)-fibre. As shown in Figs. 7(a) and 7(b), \( \gamma \)-fibre had lower XRD-RF than \( \theta \)-fibre at all deformation conditions. As shown in Fig. 7(c), though the difference was more significant at higher working temperature and lower strain rate (e.g. lower Zener-Holloman \( (Z) \) parameter); even otherwise it remained more that the estimated difference (0.091) in the fully recrystallized state. In other words, \( \gamma \)-fibre is stipulated to have more recovery or lower energy dislocation structures than \( \theta \)-fibre: at all (especially low Z) deformation conditions.

4. Discussion

Though DSA or dynamic strain aging has long been discussed as an important mechanism for microstructural developments in low carbon steel, the present as well as the previous study did not find direct experimental evidence for or against the DSA. Explanations on grain interior strain localizations were taken more from plastic instability criterion. Research on strain localizations, especially on aspects of orientation dependence, have often

Fig. 4. Stored energy as a function of grain interior strain localizations (number of grain interior high angle boundaries per 100 \( \mu \)m length) for (a) \( \gamma \) and (b) \( \theta \) fibres.

Fig. 5. Dislocation density as a function of grain interior strain localizations (number of grain interior high angle boundaries per 100 \( \mu \)m length) for (a) \( \gamma \) and (b) \( \theta \) fibres.

Fig. 6. Dislocation arrangement parameter as a function of grain interior strain localizations (number of grain interior high angle boundaries per 100 \( \mu \)m length) for (a) \( \gamma \) and (b) \( \theta \) fibres.
used Dillamore’s plastic instability criterion\(^{58}\)
\[
\frac{1}{\sigma} \frac{d\sigma}{d\varepsilon} = \frac{n}{\varepsilon} + m \frac{d\dot{\varepsilon}}{d\varepsilon} + 1 + n + m \frac{dM}{d\varepsilon} - \frac{m}{\rho_m} \frac{d\rho_m}{d\varepsilon} \leq 0 \quad \ldots (5)
\]
where \(\sigma\) and \(\varepsilon\) are the macroscopic stress and strain, \(n\) and \(m\) are strain hardening exponent and strain rate sensitivity, \(\dot{\varepsilon}\) is the strain rate, \(M\) is the Taylor factor and \(\rho_m\) is the mobile dislocation density. Preferred or orientation sensitive appearance of the strain localizations were typically generalized through high \(M\)\(^{9,20}\) or negative \(dM/d\varepsilon\)\(^{25}\) values. Though role of \(dM/d\varepsilon\) have usually been acknowledged,\(^{25,58}\) the role of dislocations on the orientation sensitivity of strain localizations have rarely been explored. This has been the novelty of the present study.

It is apparent from the experimental data (Figs. 4–7) that dislocations and dislocations structures were different in \(\gamma\) and \(\theta\) fibres. These, in turn, were associated with differences in the preferred appearance of ‘grain interior strain localizations.

Following points try to generalize the differences:
- Increase in strain localizations was associated with larger increase in dislocation density for \(\gamma\)-fibre.
- However, asymmetry in peak profile (the so-called
\(<\vec{f}>^3\)\(^{30}\) was more for \(\theta\)-fibre. In other words, \(\theta\)-fibre is expected to have more dislocations with burgers vectors of similar sign than \(\gamma\)-fibre.

- The last point may offer an explanation, albeit indirect and qualitative, for the observed higher XRD-RF values, and hence an expected lower recovery, for \(\theta\)-fibre.

To explore these differences even further, it was decided to use discrete DD simulations. DD simulations have limitations.\(^{40-42}\) They are typically valid for single crystal pure iron and even with large computation resources the imposed plastic strains are often ‘limited’. For the present study, several orientations from \(\gamma\) and \(\theta\) fibres were taken and multiple simulations, with different dislocation configurations, were conducted. Of course strains were restricted, but the reproducibility between the simulations was excellent and results from the representative simulations are shown in Fig. 8. As shown in Fig. 8(a), the stress\(^2\) strain behaviour between \(\gamma\) and \(\theta\) fibres was remarkably different. Stress at \(1\times10^{-7}\) plastic strain was ~1.5 times in \(\gamma\)-fibre as compared to \(\theta\)-fibre. Interestingly, this is nearly identical as the simulated difference between the respective average Taylor factors – Fig. 2(a). In other words, the DD simulation could capture, quantitatively, the differences between texture-estimated Taylor factors for \(\gamma\) and \(\theta\) fibres. The other interesting aspect, as highlighted in Fig. 8(b), was noticeably different \(dp/d\varepsilon\) values between \(\gamma\) and \(\theta\) fibres: for the same plastic strains the \(\gamma\)-fibre had ~5 times \(dp/d\varepsilon\) than \(\theta\)-fibre. Significantly higher \(dp/d\varepsilon\) for \(\gamma\)-fibre also signifies preference for higher instability or more ‘grain interior strain localizations’ – see Eq. (5).

It is also important to evaluate the present results in reference to the previous study.\(^{26}\) A clear increase in the density and preference (for \(\gamma\)-fibre) of ‘grain interior strain localizations’ was reported,\(^{26}\) for the same material, at the intermediate working temperatures. A straight and equivalent (between \(\gamma\) and \(\theta\) fibres) recovery, arguably, reduces the \(dp/d\varepsilon\) advantage for \(\gamma\)-fibre. However, it needs to be pointed out that the DD simulations are conducted for pure iron. ULC, on the other hand, has interstitial content – see Table 1. Interactions between dislocations and interstitial atoms may reduce the \(dp/d\varepsilon\) advantage for \(\gamma\)-fibre as well. Hence increase in working temperature can have two effects: reduced dislocation-interstitial interactions and increase in overall recovery. These two counter-balancing effects, and not a straight forward recovery, may offer a logical explanation for the previous report\(^{29}\) on a clear increase in the preference for ‘grain interior strain localizations’ in deformed \(\gamma\)-fibre grains at the intermediate working temperatures. The other alternative is, of course, possibilities of orientation sensitive recovery: the possibility that recovery itself have contributed to the formation of grain interior strain localizations. This possibility, though often acknowledged,\(^{10,39,59}\) cannot be accounted in a plasticity based instability criterion, such as Eq. (5).

This study comes up with several interesting observations on the role of dislocations in the preferred appearance of ‘grain interior strain localizations’ in low carbon steel. It also raises questions on the exact role of recovery on the formation of strain localizations. Future research needs to be tailored to explore such questions. However, it is apparent from the present study that preferred appearance of ‘grain interior strain localizations’ in deformed \(\gamma\)-fibre grains is aided by both textural softening (negative \(dM/d\varepsilon\)) and strong increase in mobile dislocation density (\(dp/d\varepsilon\)). Though exact role of orientation sensitive recovery needs to be tested further, the basic observations and inferences of this study remain statistically sound, reproducible and novel.

5. Summary

- \(\gamma\) (ND/(<111>) fibre was estimated to have higher \(M\) and more negative \(dM/d\varepsilon\) than the \(\theta\) (ND/(<100>) fibre. This may explain, partly, the preference for strain localizations in \(\gamma\)-fibre. Samples subjected to the same strain (different working temperatures and strain rates), however, had similar textures. Hence textural aspects are not anticipated to affect, significantly, relative appearance of strain localizations between the fibres.
- Density of ‘grain interior strain localizations’ scaled,
linearly, with grain average misorientation (GAM). The scaling was similar for both γ and θ fibres. XRD estimated stored energy of cold work, by the Stibitz formula, also scaled with density of strain localizations. The scaling was steeper for γ-fibre. As the standard Stibitz is based on elastically isotropic material, the analysis was extended to more involved micro-stress measurements/analysis.

- Micro-stress measurements, with the momentum method, brought out several interesting observations. For both fibres, increased density of strain localizations coincided with increase in dislocation density (ρ). This effect was ~3.6 times in γ-fibre than in θ-fibre. However, asymmetry in peak profile was more for θ-fibre. θ-fibre is hence expected to have more dislocations with burgers vectors of similar sign than γ-fibre.

- γ-fibre also showed higher X-ray resolution function (XRD-RF), indicating presence of lower energy dislocation substructures (and hence higher recovery) than θ-fibre. This effect, though more pronounced at higher working temperature and lower strain rate, was apparent at all deformation conditions.

- DD simulations had shown ~5 times dp/dε in γ-fibre than in θ-fibre. In other words, preferred appearance of strain localizations in γ-fibre appears to be supported by a combination of textural softening (dMdε) and large dp/ε.

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