Development of Grain Interior Strain Localizations during Plane Strain Deformation of a Deep Drawing Quality Sheet Steel

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Development of dislocation substructures was characterized in an aluminum killed deep drawing quality steel at four different plane strain deformations. At and above 20% reduction, the most significant substructural feature was micro bands (MBs). MBs appeared as paired dislocation walls of 0.2–0.4 μm thickness and were always at an angle of approximately 37° with rolling direction (RD). As the traces of the MBs were more than 5° of {110} and {112}, closed packed planes of the bcc system,—they were termed as first generation

have often described them by the generic term of “grain interior strain localizations”. Evidently, a more detailed understanding on the development of such strain localizations is needed—which is the objective of the present study.

The development of dislocation substructure in bcc low carbon steel has been a subject of limited studies in comparison with high stacking fault energy fcc systems. Conventional metallographic studies of deformed low carbon steels have often reported existence of grain interior “fish-bone” like structures—the so-called grain interior strain localizations. Orientation Imaging Microscopy (OIM) studies have identified such structures as “more frequent grain boundaries with higher than average misorientations” and inclined at approximately 37° with the rolling direction (RD). There seems to be two opinions about the exact nature of such strain localizations. (I) These were at times considered as in-grain shear bands. (II) Alternatively, Transmission Electron Microscope (TEM) observations in warm rolled IF (interstitial free) steel suggested that the so-called grain interior strain localizations are basically micro bands at angles of ±35° with RD. In absence of a comprehensive understanding regarding the nature and origin of the “fish-bone” like structures or grain boundary clusters at 37° with RD; previous studies have often described them by the generic term of “grain interior strain localizations”. Evidently, a more detailed understanding on the development of such strain localizations is needed—which is the objective of the present study.

The development of dislocation substructure in bcc low carbon steel has been a subject of limited studies in comparison with high stacking fault energy fcc systems. The usually acknowledged sequence of substructure development, i.e. as a function of increasing cold reduction, in a cell forming fcc metal can be generalized as: Taylor lattice (TL) → cell blocks (CBs) and dense dislocation walls (DDWs) → first and second generation micro bands (MBs) → shear bands (SBs). In the present study some of these terms and conventions, will be used/borrowed to describe the substructure development in bcc low carbon steel.

2. Experimental Methods

2.1. Material and Processing

Aluminum killed DDQ steel, chemical composition given in Table 1, was produced by vacuum oxygen degassing (VOD). Continuous cast billets were hot rolled to 50% reduction (1325, 900 and 650°C being the respective reheating, finishing and coiling temperatures). Hot rolled

Table 1. Chemical composition (in wt%) of aluminum killed DDQ steel used in the present study.

| C  | Mn  | Si  | P  | S   | Al  | N |
|----|-----|-----|----|-----|-----|---|
| 0.05 | 0.23 | 0.028 | 0.01 | 0.012 | 0.04 | 0.03 |
coil was subsequently cold rolled to 80% reduction in thickness and then batch annealed at 740°C, 16 h soaking. Fully recrystallized batch annealed material*1 showed grain sizes of 26, 14 and 14 μm respectively along rolling direction (RD), transverse direction (TD) and normal direction (ND). This was used for subsequent deformation studies using 100 mm flat bottom punch with in-plane stretching. Figure 1 shows the schematics of the tooling geometry and dimensions used for such tests. During in-plane stretching various strain states can be obtained by controlling the dimensions of the test samples. The plane strain condition is obtained by testing rectangular sheet samples of dimensions 140 by 178 mm. A rectangular sheet sample of identical dimension having a central hole of diameter 38 mm is used as the washer during the deformation. The sheet sample along with the washer blank is formed by means of a flat cylindrical punch. To avoid direct contact with the punch during forming, the actual specimen was separated from the die by the washer sheet. The whole assembly was lubricated with molybdenum disulphide grease and was then clamped between die and binder plates, as shown in Fig. 1. The forming was carried out on a 200 t single action press using 300 kN binder force. Circle grid analysis was used for measuring strains in the formed samples. Substructure developments in the following plane strain reductions were studied: 5, 20, 33 and 41%.

2.2. Examination of Microstructures

TEM foils were prepared from the rolling planes (i.e. containing RD and TD). On each foil, the rolling direction was marked by “v” grooving with a slow speed diamond cutter. The foils were electropolished in a Struers Teunopol 3 twin jet polisher, using 80 : 20 methanol : perchloric acid as electrolyte, at -20°C and 10 V dc. Foils were subsequently studied at 200 keV in a Philips CM200 TEM. Initially for local orientation measurements, a computer program (using the methodology described by Young et al.23) was used to solve the convergent beam electron diffraction (CBED) Kikuchi patterns. Subsequent measurements were obtained using the commercial TSL-ACT package. ACT i.e. advanced crystallographic technique is a tool for online local orientation measurement in TEM. The package has been developed and marketed by TSL-EDAX. Size measurements were obtained using online interface of CM200 series, which uses deflector coil movements to measure distances in the microstructures.

Orientations were generalized as F{111}⟨112⟩, E{111}⟨110⟩, I{112}⟨110⟩ and H{001}⟨110⟩. While the first two can be idealized as γ-fiber, the last two falls within generalized α-fiber (RD//⟨110⟩). Any region of the dislocation substructure within 20° of these ideal orientations were considered accordingly. If a deformed grain fell within 20° of more than one ideal component, it was considered as the component with least misorientation. Any deformed region misoriented by more than 20° from these idealized components was considered as “random”—i.e. deformed grain outside the usual α and γ-fibers. For each reduction and for each idealized component, at least 20 measurements each from at least 3 different samples were used.

3. Results

Conventional metallography followed by optical microscopy revealed grain interior strain localizations23 at and above 20% plane strain reduction. Optically these appeared as in-grain “fish-bone” like structures and were similar to the deformed structures commonly reported2,10,11–15 in low carbon steels. As metallographic observations of such grain interior strain localizations are rather common,11–13 these are not repeated in the present paper. To understand and characterize the origin/nature of such strain localizations, extensive TEM studies were carried out at all four reductions. TEM observations on the overall substructural changes and on the relative developments in different microtexture components are presented separately.

3.1. Overall Substructural Changes

At the lowest reduction of about 5%, the substructures consisted of large dislocation cells, as shown in Fig. 2. The cell size (d) was about 1 μm and the misorientation among the neighboring cells (θ) was typically of the order of 1.5°, see Table 2. The other interesting feature at this lowest reduction was the regions with higher dislocation densities, one such region is marked by a pair of parallel lines and arrows in Fig. 2. Such regions are somewhat similar to the dense dislocation walls (DDWs) commonly observed in fcc metals,11 and will be also be termed generically as DDWs. These DDWs were 0.2–0.6 μm thick, often aligned at an angle of approximately 37°±6° with RD, as shown in Fig. 2. At this reduction, DDWs were relatively few. Only one DDW, on average, was typically observed over a distance of 6 μm along TD. The average misorientation across the DDWs was 3°, while the average misorientation between adjacent cells was about 1.5°.

At 20% reduction, the most interesting feature of the dislocation substructure was the MBs, as shown in Fig. 3(a). These were essentially parallel pairs of dislocation walls of 0.2–0.4 μm thickness and often had substructure inside, and were always at an angle of about 37° with RD. MBs appeared mostly as paired dislocation sheets (PDS), although strings of small pancaked-shaped cells (SPC)17 were also

*1 Measured planar and normal anisotropies of the batch annealed material being 0.35 and 1.66 respectively.
The MBs observed in the present study were: (I) first generation or non-crystallographic and (II) second generation or crystallographic. The non-crystallographic or first generation MBs were at times part of preexisting oriented regions—having a spatial angle of 37° with RD. At 33 and 41% reductions, MB intersections were often observed, as shown in Fig. 2. Such intersections had two visible features on the substructure: (I) Regions with MB intersections often had a checkered pattern, as shown in Fig. 4. (II) MB intersection at times led to noticeable shear offsets, as shown in Fig. 5(a). It is to be noted that such shear offsets from intersection of first generation MBs were noticed in some cases (but not always) and shear offsets did not lead to the formations of larger misorientations or any significant “preferred” orientation(s). Perhaps the most significant effect of the presence of MBs was the ‘misorientation development’, see Figs. 3(b) and 5(b). At all three stages of reduction where MBs were visible, misorientation development across the MBs were far more significant than misorientation among ordinary dislocation cells.

### Table 2. Average cell size (\(d\) \(\mu\)m) and average misorientation among neighboring cells (\(\theta^\circ\)) for different microtexture components at four reductions.

| % Reduction | F \([\{111\} < 11\overline{2}]\) | E \([\{111\} < 11\overline{0}]\) | I \([\{112\} < 1\overline{1}0]\) | H \([\{100\} < 1\overline{1}0]\) | Random |
|-------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|---------|
| β (°)       | \(\theta^\circ\) (°)          | \(\theta^\circ\) (°)          | \(\theta^\circ\) (°)          | \(\theta^\circ\) (°)          | \(\theta^\circ\) (°) |
| 5%          | 1.5                           | 1.1                           | 1.4                           | 1.4                           | 1.2     |
| 20%         | 4.0                           | 0.7                           | 4.16                          | 0.68                          | 3.04    | 0.73   | 1.76  | 0.84 |
| 33%         | 5.0                           | 0.4                           | 5.5                           | 0.5                           | 4.6     | 0.56   | 2.1   | 0.82 |
| 41%         | 7.2                           | 0.35                          | 7.2                           | 0.36                          | 6.2     | 0.43   | 2.4   | 0.79 |

At the two higher reductions of 33 and 41%, MBs continued to be the significant feature of the overall substructure. Increasing reductions, in general, increased the frequencies of the MBs and misorientation across the same \((\theta_{\text{MB}}))\), as shown in Table 3. Even then all the MBs continued to be first generation—having a spatial angle of 37°±6° with RD. At 33 and 41% reductions, MB intersections were often observed, as shown in Fig. 4. Such intersections had two visible features on the substructure: (I) Regions with MB intersections often had a checkered pattern, as shown in Fig. 4. (II) MB intersection at times led to noticeable shear offsets, as shown in Fig. 5(a). It is to be noted that such shear offsets from intersection of first generation MBs were noticed in some cases (but not always) and shear offsets did not lead to the formations of larger misorientations or any significant “preferred” orientation(s). Perhaps the most significant effect of the presence of MBs was the ‘misorientation development’, see Figs. 3(b) and 5(b). At all three stages of reduction where MBs were visible, misorientation development across the MBs were far more significant than misorientation among ordinary dislocation cells.
of the MBs can be quantified from (a) the MB spacing along TD ($\lambda$), and (b) average misorientation across MBs ($\theta_{MB}$) in different microtexture components. Even at 20% reduction, $\lambda$ was less and $\theta_{MB}$ was more for F and E grains than I and H grains. With increase in reduction, this trend of $\lambda$ and $\theta_{MB}$ values did not change, rather relative differences increased, as shown in Table 3. The trend is similar to the expected trend in Taylor Factors. In other words, microtexture components or orientations with higher Taylor factors had more frequent and effective MBs, or lower $\lambda$s and higher $\theta_{MB}$s, — combination of which is reflected in the trend of stored energy differences between different microtexture components, as shown in Table 4.

Table 3. Micro band (MBs) spacings ($\lambda$ $\mu$m) and average misorientation across MBs ($\theta_{MB}$) as measured along TD in different microtexture components at 20–41% reductions. No MBs was observed at 5% reduction; estimated average DDW spacing and misorientation across DDWs were approximately 6 $\mu$m and 3°—values which were nearly equal between different microtexture components.

| % Reduction | F $\{111\}<11\bar{2}>$ | E $\{111\}<11\bar{0}>$ | I $\{11\bar{2}\}<11\bar{0}>$ | H $\{001\}<11\bar{0}>$ | Random |
|-------------|------------------|------------------|------------------|------------------|--------|
|             | $\lambda$ ($\mu$m) | $\theta_{MB}$ (°) | $\lambda$ ($\mu$m) | $\theta_{MB}$ (°) | $\lambda$ ($\mu$m) | $\theta_{MB}$ (°) |
| 20          | 3.0              | 5.8              | 3.0              | 6.11             | 4.6    | 6.04             | 3.3            | 4.4            | 4.1            |
| 33          | 2.0              | 10.2             | 2.1              | 10.31            | 7.5    | 5.88             | 5.4            | 3.0            | 7.17           |
| 41          | 0.96             | 20.6             | 1.1              | 20.19            | 15.3   | 5.5              | 10.6           | 1.8            | 15             |

Fig. 4. Dislocation substructure of E $\{111\}<11\bar{0}>$ component after 33% reduction. Two of first generation MBs are marked with double arrows.
4. Discussion

4.1. On the Possible Nature of the ‘Grain Interior Strain Localizations’

Unlike fcc metals, detailed substructural developments in low carbon bcc steels are relatively less charted. In a high stacking fault energy fcc system, the initial near random dislocation structure or Taylor Lattice is expected to be transformed into less randomized tangled dislocation structure, which, in turn, gets replaced/broken into cell blocks (CBs), consisting of equiaxed cells, by dense dislocation walls (DDWs). At subsequent stages of deformation, MBs, essentially parallel dislocation sheets, are observed. In fcc metals, MBs are distinguished as first and second generation. Second generation MBs are characterized by their crystallographic orientation, their traces falling within 5° of {111}, the closed packed plane of the fcc system. In a polycrystalline fcc metal, second generation MBs will not have a definite geometric orientation with respect to sample axis, as the traces of the closed packed planes will be different in different grains. First generation MBs, on the other hand, is expected to have a geometric orientation with respect to sample axis—aligned along the direction of maximum shear stress. Formation of first generation MBs is considered as the most frequent mechanism for the nucleation of new cell-bocks by splitting of pre-existing DDWs.

At 5% reduction, substructure was essentially made of dislocation cells. Such cells formed the so-called cell-blocks and individual cell blocks were separated by regions of higher dislocation densities, which are somewhat similar to the DDWs of the fcc metals. The DDWs and the cell blocks had a characteristic approximate 37° angle with RD. Misorientation across the DDWs were twice of that of ordinary neighboring cells. Approximate DDW spacings were 6 μm, somewhat similar between different microtextural components. This was perhaps reflected in the nearly identical stored energy values at 5% reduction. OIM studies at early deformation stages (at about 7% reduction) in IF steel also had similar observations.

At and above 20% reduction, the most interesting feature of the dislocation substructure was the MBs, as shown in Figs. 3(a), 4 and 5(a). These bands of morphologically different dislocation structure were termed as MBs because of their characteristic parallel dislocation walls. As discussed earlier in Sec. 3.1, the MBs were classified as first generation. Increasing reductions brought MB intersections and somewhat checkered dislocation substructure and at times shear offsets, as shown in Figs. 4 and 5(a). This has also been reported in pure aluminum. MBs were, however, always first generation and at an approximate angle of 37° with RD, at least for the range of reductions used in the present study. The main feature of the substructure with sufficient misorientation (i.e. other than pre-deformation grain boundaries) necessary for conventional metallographic or OIM visibility are the first generation MBs and the same can be considered as exact nature of the grain-interior strain localizations at least over the range of strain and strain path used in the present type of low carbon steel.

4.2. Orientation and Substructure

Role of orientation on substructural development is important not only in understanding deformation micromechanisms, but also in comprehending subsequent recrystallization behavior. In low carbon steel, formation of strong γ-fiber (ND/(111)) recrystallization textures is usually attributed to ‘preferred nucleation’. The two commonly referred mechanisms of such preferred nucleation

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**Table 4.** θ/d values (°/μm), as estimated from Table 2, for different microtexture components at four reductions. θ/d values are expected to scale with stored energies of deformation. Also included are standard deviations (SD) of estimated θ/d’s.

| % Reduction | F (111)<112> | E (111)<110> | I (112)<110> | H (001)<110> | Random |
|-------------|--------------|--------------|--------------|--------------|---------|
|             | θ/μm SD      | θ/μm SD      | θ/μm SD      | θ/μm SD      | θ/μm SD |
| 5%          | 1.36 / 0.66  | 1.4 / 0.66   | 1.17 / 0.65  | 1.4 / 0.64   | 1.36 / 0.66 |
| 20%         | 5.8 / 1.85   | 6.12 / 1.86  | 4.16 / 1.12  | 2.1 / 0.68   | 4.1 / 1.2 |
| 33%         | 10.2 / 3.6   | 10 / 3.3     | 7.5 / 2.3    | 2.6 / 0.78   | 7.17 / 2.4 |
| 41%         | 20.6 / 5.5   | 20 / 5.2     | 14.4 / 4     | 3 / 0.98     | 15 / 4.4 |

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**Fig. 5.** (a) Dislocation structure of E (111)/(110) after 41% reduction. An intersecting first generation MB with noticeable shear offset is marked by double arrows. Misorientation changes on the points marked in Fig. 5(a) are given on Fig. 5(b). Conventional misorientation representation is same as in Fig. 3(b).
are: (I) higher stored energies of deformed γ-fiber grains\(^{(2,3,6–9)}\) and (II) micro-growth advantage or selection of γ-fiber recrystallized grains by virtue of a special high mobility boundary such as 25–35°\(^{(110)}\).\(^{4,5,12}\) Direct experimental evidence\(^{(2,3,6–9)}\) for (I) seems to be convincing, although effectiveness of (II) is debated at times.\(^{2,3,7}\) It seems, however, that both schools of opinion now consider presence of grain interior strain localizations as a ‘requirement’ for either of the mechanism(s).\(^{2,12}\)

Bulk measurement techniques,\(^{9,25,26}\) like X-ray and neutron diffraction, had repeatedly shown that Taylor factors, grossly representing orientation against a particular strain tensor as a single scalar quantity,\(^{8}\) can be used as an index of stored energy in a polycrystalline material. Local orientation measurements had also shown\(^{(2,7,8,10,20,24,27)}\) that deformed structure of individual grains and their relative stored energies usually depend on orientation and a generalized trend in Taylor factors. It seems that there are only two kinds of exception reported\(^{(2,10,18)}\) against this generalized trend—higher Taylor factor indicates higher stored energy. (I) As observed in the present study and also reported in earlier research on low carbon steel,\(^{(2,10)}\) at the early deformation stages substructure and stored energy seems to be independent of Taylor factor or orientation. (II) At low Zener–Holloman factors\(^{(26)}\) or at high temperature and/or low strain rate deformation, reported for AA1050,\(^{(3)}\) the same is true. Interestingly in all these cases,\(^{(2,10,18)}\) the lack of dependence of stored energy on Taylor factor was observed to coincide with the absence of so-called ‘grain interior strain localizations’. In other words, grain interior strain localizations, having a specific spatial angle with RD, seems to be responsible for stored energy differences in grains of different orientations or Taylor factors—as reported both in low carbon steel\(^{(2,10)}\) especially the present study, and commercial purity aluminum.\(^{(18,20,29)}\)

The nature of such grain interior strain localizations, at least in the present study, is classified as first generation MBs. The present study has also shown that frequency and effectiveness, as quantified by \(\lambda\) and \(\theta_{\text{red}}\), of the first generation MBs depend on orientation or Taylor factor, as shown in Table 3. These, in turn, determine the stored energy, as estimated by respective \(\theta/d\) values in different microtexture components, as shown in Table 4.

Presence and effectiveness of the MBs not only determine the absolute value of the stored energies, as given in Tables 3 and 4, but also decide the relative spread or standard deviations (SD) of such stored energies. In other words, an orientation with higher stored energies and consequently higher SD of stored energies is more potential site for preferred nucleation.\(^{(5)}\) The γ-fiber grains satisfy this, as shown in Table 4. This, on the other hand, may explain the subsequent preferred nucleation behavior of recrystallized γ-fiber grains.\(^{(2,3,7)}\)

It is perhaps best to point out that maximum reduction of 41% used in the present study is generally inadequate to obtain a strong γ-fiber recrystallization texture in an aluminum killed DDQ steel, the optimum rolling reduction being 60–70%. Our results, on the other hand, clearly indicate stored energy advantage for γ-fiber grains at 41% reduction. There are two possible answers for this apparent discrepancy. (I) Other than stored energy advantage, deformed γ-fiber grains also need to be effectively pancaked, i.e. their thickness along ND needs to be 1–2 μm, for ‘effective’ preferred nucleation.\(^{(26)}\) In other words, a thicker (along ND) deformed γ-fiber grain even with a ‘stored energy advantage’ will not act as a potent site for preferred nucleation, as growth of recrystallized γ-fiber grains can be hindered.\(^{2,5}\) Present deformation of 41% is not adequate for effective pancaking, as deformed grain thickness after 41% reduction was estimated to be approximately 6 μm along ND. (II) Possibilities of differences in stress conditions and subsequent grain interior strain localization behavior between press forming, as used in the present study, and cold rolling, the usual industrial practice, may not be ruled out.

The other interesting issue is the observation that ‘frequency and effectiveness of MBs depend on orientation’. The answer for this is perhaps best sought in the frame work of Dillamore’s instability criteria.\(^{(30)}\)

\[
\frac{1}{\sigma} \left( \frac{d\sigma}{d\varepsilon} \right) = \frac{n}{\varepsilon} + \frac{m + \varepsilon}{\varepsilon} \left( \frac{d\varepsilon}{d\varepsilon} \right) + \frac{1 + n + m}{M} \left( \frac{dM}{d\varepsilon} \right) - \frac{m}{\rho} \left( \frac{d\rho}{d\varepsilon} \right) \equiv 0 \quad \ldots (1)
\]

where \(\sigma\) and \(\varepsilon\) are the macroscopic stress/strain, \(n\) and \(m\) are strain hardening exponent and strain rate sensitivity, \(\dot{\varepsilon}\) is the strain rate, \(M\) is the Taylor factor and \(\rho\) is the mobile dislocation density.\(^{(30)}\)

Formation of plastic instabilities or strain localizations is expected\(^{(30)}\) and observed\(^{(18,30–33)}\) to be determined by the instability criteria—Eq. (1). In a low solute material where dislocation softening termed as \((dp/de)\) is less significant, the instability criteria is expected to be dominated by tensile softening termed as \((dM/de)\).\(^{(32,33)}\) In other words, in a relative pure material negative \((dM/de)\) values for a grain or orientation is expected to ease formation of strain localizations.\(^{(18,31,32)}\) In general, but not always, the so-called hard orientations or orientations with large Taylor factors have higher chances of a negative \((dM/de)\).\(^{(18)}\) This, on the other hand, may explain our observation that both frequency and effectiveness, or overall formation, of first generation MBs were more in deformed grains of higher Taylor factors.

5. Summary

- At and above 20% reduction, the most significant features of substructure were parallel pairs of dislocation walls with morphologically different substructure inside—termed as micro bands or MBs using standard fcc nomenclature. These were deviated by more than 5° from traces of \{110\} and \{112\} planes, closed packed planes of bcc system, and were always at an angle of about 37° with RD. Following the usual fcc conventions, the MBs were classified as first generation or non-crystallographic.

- Other than pre-deformation grain boundaries, MBs were the only other substructural feature with large enough misorientation necessary for optical and OIM visibility. The first generation MBs can be considered as the exact nature of grain interior strain localizations, at least in the strain and strain path range of the study.
At the lowest reduction of 5%, overall dislocation substructures and the estimated stored energies were nearly equal between different microtexture components; while at and above 20% reduction dislocation substructure and stored energies did depend on Taylor factor or orientation. This was clearly related to the presence and effectiveness of MBs, as quantified by MB spacing ($\lambda$) along TD and misorientation across MBs ($\theta_{MB}$). In other words, overall formation of MBs appear to be easier at orientation with high Taylor factors.

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