Slip System Partitioning as a Possible Mechanism for Ultrafine Grain Formation in Fe–3%Si Bicrystals

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An Fe–3%Si bicrystal was deformed in compression to a strain of 0.9 at ambient temperature. In the interior of deformation bands, characteristic band structures with high orientation gradients and low and high angle boundaries were formed during straining. Furthermore, isolated ultrafine grains were found in the matrix of the deformation bands. The morphology and crystallography of the ultrafine grains indicate that they are caused by slip system partitioning, i.e. local activity of a highly stressed slip system that is different from the active slip system in the surrounding crystal volume.

KEY WORDS: silicon steel; bicrystal; deformation band; ultrafine grain; slip system partitioning; EBSD.

1. Introduction

Grain refinement is a promising means to increase the strength of steels without sacrificing toughness, thus intensively studied in the last decade.1–5) Principally there are two strategies to obtain ultrafine grained steels, either advanced thermomechanical processing6–9) or severe plastic deformation10–14). For both methods, however, large plastic strain of 1–4 are required for the formation of ultrafine grained steels with grain sizes below 1 μm. Key factors to understand grain refinement in steel are phase transformation, recovery, and recrystallisation. However, since large straining is involved, it is also a key issue to understand the formation of strain-induced boundaries during deformation. Thus, this paper outlines the characteristics of various types of strain-induced boundaries and discusses the formation of ultrafine grains by slip system partitioning.

2. Experimental Procedure

An Fe–2.5mass%Si bicrystal was grown using the Bridgeman method, and a rectangular compression test specimen was cut with a height of 6.2 mm and an area of about 6·6 mm². The initial crystal orientations correspond to a γ fiber orientation and the cube orientation, respectively, with respect to terminology used for rolling, i.e. a (111) crystal direction was parallel to the compression direction for crystal A, and a {001} crystal direction was parallel to the compression direction for crystal B (Fig. 1). The grain boundary was parallel to the compression direction, parallel to a {111} crystal plane in crystal A, and parallel to a {001} plane in crystal B. The grain boundary misorientation was 56° (123). According to Taylor theory both component crystals are mechanically stable in compression, i.e. crystals A and B do not change their crystal orientations during straining.

The compression test was performed at ambient temperature with a constant head speed of 1 mm/min, corresponding to strain rates increasing from 3·10⁻³ to 6·10⁻³ s⁻¹. The specimen was deformed to a final height reduction of 59%, which corresponds to a strain of 0.9. After deformation the specimen was cut parallel to the compression direction and perpendicular to the initial grain boundary plane. On this cut section, the deformation microstructure was investigated by means of electron backscatter diffraction (EBSD) in a high-resolution field emission gun scanning electron microscope (JEOL JSM7000F). Crystal orientation analysis was performed with TSL OIM software. The measured areas and step sizes were varied over several orders of magnitude in order to analyse the microstructure in various length scales, since a hierarchical approach is considered to be important to understand the microstructure development and the underlying mechanisms.

3. Results

3.1. Microstructure Features of Deformed Bicrystal

Fig. 1. Initial orientations of crystals A and B of Fe–3%Si bicrystal. 001 pole figure. CD: compression direction.

Crystals A and B of the deformed bicrystal have different characteristic microstructure features (Fig. 2(a)). In
crystal A, the most striking features are microshear bands\(^{15,16}\) that extend from the center of the specimen close to the grain boundary to the edge of the specimen. The shear related to the microshear bands is indicated by steps at the grain boundary. Furthermore, a deformation band\(^{17,18}\) was formed in crystal A at the grain boundary, which was not analysed in detail in this study. In crystal B, deformation bands with a characteristic wedge-shape developed in the vicinity of the grain boundary (Figs. 2(a), 2(b)). In order to understand the dependence of the formation of these microstructure features on the presence of the neighbouring grain corresponding single crystal experiments were per-

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**Fig. 2.** Microstructure of bicrystal specimen deformed to a strain of 0.9. EBSD data. Color coding according to inset inverse pole figure with respect to the sample direction as indicated by an arrow. (a) View of entire bicrystal. CD: compression direction; SB: microshear bands; DB: deformation bands; F: deformation regions caused by friction with the piston. EBSD step size: 4 \(\mu m\). (b) Deformation band in crystal B. Inset square: area shown in Fig. 2(c). EBSD step size: 1 \(\mu m\). (c) Strain-induced boundaries types 1–3. In addition to the crystal orientation, the EBSD pattern quality is displayed in grey scale. Step size: 0.2 \(\mu m\). Inset square: area shown in Fig. 4(a).

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**Fig. 4.** (a) Detail of deformation band shown, which is shown in Fig. 2(c), with isolated ultrafine grains in the matrix (boundary type 2; indicated by arrows) and a band structure with strain-induced low and high angle boundaries and high orientation gradients (boundary type 3). EBSD orientation data and pattern quality. Step size: 30 nm. (b) Detail of Fig. 4(a) displaying isolated ultrafine grains (highlighted in red). Black lines: boundaries with misorientation >15°. (c) 011 pole figure for the orientation data shown in Fig. 4(b). The red data points correspond to the grains highlighted in red. The rotation axis (star mark) is a (110) crystal axis, which is perpendicular to the compression direction. (d) Detail of Fig. 4(a) showing the band structure. Black lines: boundaries with misorientation >15°. (e) 011 pole figure for the data marked by a polygon in Fig. 4(d), i.e. for the interior of a band. The rotation axis (star mark) is a (110) crystal axis, which is approximately perpendicular to the compression direction.
formed. It was found that the microshear bands in crystal A also formed in a single crystal with the same crystal orientation. Thus, they are not caused by the presence of the neighbouring crystal. In contrast, the wedge-shaped deformation bands in crystal B were not found in the corresponding single crystal. Hence, these deformation bands are caused by the grain boundary constraint. The single crystal experiments and the microstructure evolution of the bicrystals and single crystals during straining will be discussed in more detail in a forthcoming paper. In the following we focus on the formation of microstructure features in the interior of the deformation bands in crystal B, i.e. isolated ultrafine grains in the matrix as well as strain-induced boundaries and orientation gradients in band structures.

3.2. Characteristics of Deformation Bands

In crystal B of the bicrystal specimen, four wedge-shaped deformation bands were formed during straining in the vicinity of the grain boundary (Fig. 2(a)). Their sizes in the direction of the long axis range from 150 to 800 μm, and from 20 to 110 μm in the direction of the shorter axis. The deformation bands are inclined 30°–45° with respect to the compression direction. They are separated from the surrounding crystal volume by a high angle boundaries (strain-induced boundary type 1) with a misorientation of about 45°. The rotation axis is close to a (112) crystal axis and almost perpendicular to the compression direction (Fig. 3). These boundaries are partly smooth, but in other parts they form jags with a length of 10–20 μm (Fig. 2(c)). The material in the deformation bands rotated away from the initial orientation up to 60°, whereas in the surrounding region the crystal rotated at maximum approximately 20° from the initial crystal orientation.

In the interior of the deformation bands two types of strain-induced boundaries can be distinguished (Fig. 2(c) and Fig. 4(a)). Isolated ultrafine grains were formed in the matrix of the deformation bands (boundary type 2). Furthermore, characteristic bands structures with orientation gradients and strain-induced low and high angle boundaries (boundary type 3) were observed in the interior of the deformation bands.

3.3. Isolated Ultrafine Grains in Deformation Bands

In the matrix of the deformation bands, isolated regions smaller than 1 μm that are enclosed by high, and partly low, angle boundaries (boundary type 2) were formed during deformation, i.e. these are new strain-induced ultrafine grains (Figs. 4(a), 4(b) and Fig. 5). The material inside these grains rotated up to approximately 20° about a (011) axis with respect to the surrounding matrix (Fig. 4(c)). The ultrafine grains are elongated (aspect ratio of about 3) with the long side inclined to the horizontal by 2°–9° (Fig. 6). In the long direction of the grains, small orientation gradients are found of up to 8°/μm in some cases, whereas in the short direction the crystal orientation is almost constant (Fig. 5). Furthermore, the long sides of the grains are parallel to the trace of a (112) crystal plane with respect to the matrix orientation (Fig. 6). Linear structures in the surrounding matrix, which can be recognised on the EBSD pattern quality map, are parallel to the trace of a (110) crystal plane and inclined to the horizontal by 19°–30° (Fig. 6).

3.4. Bands with Strain-induced Boundaries and Orientation Gradients

In the interior of the deformation bands, bands structures with a width of several micrometer formed during straining (Figs. 4(a), 4(d)). The rotation axis of these band regions with respect to the matrix region in the wedge-shaped deformation bands is not very well defined, but roughly about an (001) crystal axis that is inclined about 35° to the com-
pression direction. The interior of these bands is characterised by high orientation gradients and low and high angle boundaries, in some places enclosing crystal regions with sizes smaller than 1 μm (Figs. 4(a), 4(d)). The crystal rotations inside these bands are up to 50° around a (011) crystal axis (Fig. 4(e)).

4. Discussion

4.1. Wedge-shaped Deformation Bands in Crystal B

The high angle boundaries separating the deformation bands from the surrounding crystal gradually developed from a transition band,17,18) which was initially situated between the deformation band and the surrounding material. Transition bands continuously accumulated the misorientation over a wide region. If they become narrower to finally form a sharp boundary with a discontinuous orientation change, the resulting boundary might be positioned anywhere inside the initial transition band, and thus at some places jags are formed (Fig. 7).

4.2. Formation Mechanism of Isolated Ultrafine Grains

In order to clarify the formation mechanism of the isolated ultrafine grains in the deformation bands, the resolved shear stresses were considered for all possible slip systems. Schmid factors were calculated for 12 {110}111 and 12 {112}111 slip systems. The calculation was performed for the (32, 99, 344) crystal orientation in the matrix of the deformation band assuming a vertical loading direction. It was found that two slip systems with the same slip direction have high Schmid factors, i.e. 0.46 for (121)[111], and 0.45 for (110)[111], whereas all others slip systems have Schmid factors below 0.35 (Fig. 8(a)). The angle between the trace of these slip systems in the cut section and the horizontal are 7° for (121)[111] and 27° o (110)[111] (Fig. 8(b)). The inclination of 7° for (121)[111] corresponds to the experimentally observed inclination angle of 2°–9° of the boundaries of the isolated ultrafine grains. Whereas the inclination angle of 27° for (110)[111] corresponds to the inclination of 19°–30° of the linear features in the matrix surrounding the isolated ultrafine grains. Thus, it can be supposed that, even though two slip systems have a high resolved shear stress, they are not active in the same crystal volumes, but in the matrix dislocation glide only occurs on one of them, i.e. (110)[111] (Fig. 8(c)). Whereas the other highly stressed slip systems, i.e. (121)[111], might be only locally active in sub-micrometer-sized and distant regions causing the formation of isolated ultrafine grains (Fig. 8(c)).

The described partitioning of slip system activity is considered to be a possible cause for the formation of ultrafine grains. However, more detailed TEM observations are necessary in order to prove the proposed mechanism. In the particular case of this study the two slip systems with a high resolved shear stress have the same slip direction. Further research is needed to draw general conclusions about the dependence of slip system partitioning on the crystal orientation and on the number and crystallographic relationship of highly stressed slip systems.

Note that the Schmid factors were calculated assuming that the loading direction is vertical. However, we discussed that the deformation bands are caused by a stress field that was modified due to the grain boundary constraint in the bicrystal specimen. Consequently, the principal stress direction is supposed to have change from the vertical loading direction. This discrepancy can be solved if we assume that in the initial stage of deformation the stress field is changed in the vicinity of the grain boundary and the deformation bands are formed. In a later stage of deformation the influence of the neighbouring grain becomes negligible and the assumption of a vertical loading direction is valid again. Thus, resulting in the good agreement of calculated active slip systems and experimentally observed microstructure.

Fig. 7. Scheme of formation of a jagged boundary from a transition band.

Fig. 8. (a) Schmid factors for the crystal orientation in the matrix of a deformation band. Two slip systems, i.e. (110)[111] and (121)[111] have a high resolved shear stress, while it is low for all others. (b) Stereographic projection displaying the orientation of the two slip systems with high Schmid factors. The slip direction is the same for both. The lines show the trace of the slip planes, respectively, in the cut section. (c) Local activity of (121)[111] causing the formation of ultrafine grains.
4.3. Formation of Bands inside Deformation Bands

The morphology and microstructure of the bands in the interior of the deformation bands with high orientation gradients and strain-induced boundaries indicates that these bands are microshear bands. However, in the observed cut section indications for shear strain cannot be observed. But it is possible that the direction of shear is perpendicular to the cut section and thus shear is not identifiable in the investigated section. The lattice rotation inside the bands about an axis that is almost parallel to the cut section (Fig. 4(e)) also indicates that shear strain would be perpendicular to the cut section. Furthermore, the crystallography of the rotation axis of the crystal rotations inside the bands indicates that a limited number of active slip systems caused the formation of these bands with strain-induced boundaries.

An open question is the formation mechanism of these bands. Further clarification with respect to this question is expected from numerical simulations, which are, however, beyond the scope of this paper.

5. Summary and Conclusions

An Fe–3%Si bicrystal with two mechanically stable component crystals was deformed at ambient temperature to a strain of 0.9. After deformation the microstructure was analysed by EBSD. Three types of strain-induced boundaries related to deformation bands were found in crystal B (Table 1).

Wedge-shaped deformation bands in crystal B are caused by the grain boundary constraint. After a strain of 0.9, the deformation bands are separated from the surrounding material by a high angle boundaries with misorientations of about 45° around a (112) crystal axis.

In the interior of the deformation bands, bands with high orientation gradients and low and high angle boundaries formed during deformation. In some places, these boundaries enclose small crystal regions indicating the formation of ultrafine grains with a size smaller than 1 μm. The crystal rotations inside these bands are up to 50° around a (011) crystal axis. These bands might be microshear bands.

Moreover, isolated ultrafine grains with sizes smaller than 1 μm were formed in the matrix of the deformation bands, presumably in a later stage of their evolution. These grains mostly have high angle boundaries with 20° (011) misorientation. We propose that these ultrafine grains are formed due to slip system partitioning of two (or more) highly stressed slip systems. One highly stressed slip system is only locally active in regions smaller 1 μm, whereas another slip system with a high Schmid factor is activated in the surrounding matrix.

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REFERENCES

1) M. Niikura, M. Fujioka, Y. Adachi, A. Matsukura, T. Yokota, Y. Shirota and Y. Hagiwara: J Mater. Process. Technol., 117 (2001), 341.
2) M. Niikura, Y. Hagiwara, K. Nagai, K. Tsuzaki and S. Takaki: Proc. Int. Symp. on Ultrafine Grained Steels, The Iron and Steel Institute of Japan, Tokyo, (2001), 26.
3) P. Hodgson, T. Maki, S. Takaki and T. Tsukiyama: Mater. Trans., 45 (2004), 2150.
4) R. Song, D. Ponge, D. Raabe, J. G. Speer and D. K. Matlock: Mater. Sci. Eng. A, 444 (2006), 1.
5) T. Maki: Proc. Int. Symp. on Ultrafine Grained Steels, (2003), CD.
6) Y. Adachi, T. Tomida and S. Hinotani: Tetsu-to-Hagané, 85 (1999), 620.
7) Y. Adachi, T. Tomida and S. Hinotani: Tetsu-to-Hagané, 85 (1999), 691.
8) R. Uejj, N. Tsuji, Y. Minamino and Y. Koizumi: Acta Mater., 50 (2002), 4177.
9) Y. Adachi, M. Wakita, H. Beladi and P. D. Hodgson: Acta Mater., 55 (2007), 4925.
10) N. Tsuji, Y. Saito, H. Utsunomiya and S. Tanigawa: Scr. Mater., 40 (1999), 795.
11) Y. Saito, H. Utsunomiya, N. Tsuji and T. Sakai: Acta Mater., 47 (1999), 579.
12) A. Belyakov, K. Tsuzaki, H. Miura and T. Sakai: Acta Mater., 51 (2003), 847.
13) S. Takaki, T. Tsukiyama, K. Nakashima, H. Hidaka, K. Kawasaki and Y. Futamura: Met. Mater. Int., 10 (2004), 533.
14) S. V. S. N. Murty, S. Torizuka, K. Nagai, N. Koseki and Y. Kogo: Scr. Mater., 52 (2005), 713.
15) J. Hutchinson: Scr. Metall., 18 (1984), 421.
16) S. Harren, H. Deve and R. Asaro: Acta Metall., 36 (1988), 2435.
17) C. S. Barrett: Trans. Metall. Soc. AIME, 135 (1939), 296.
18) F. J. Humphreys and M. Hatherly: Recrystallization and Related Annealing Phenomena, Pergamon Press, Oxford, (1995), 11.