Deformation and Recrystallization of Tensile-deformed or Rolled Fe–3%Si Alloy Single Crystals

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Fe–3%Si alloy single crystal samples with various tensile directions were deformed to strains of 0.25 to 0.65 and subsequently annealed. Necking occurred and work-hardening was very small in all tensile-deformed samples. After annealing, no recrystallized grain was formed. Fe–3%Si alloy single crystal sample of {111}<112> orientation was lightly rolled to 25 % and subsequently annealed. Many recrystallized grains were formed in the rolled sample. The orientation of recrystallized grains formed at the rolled surface was totally different from the orientation of those formed in the interior of the sample. These results are discussed based on the tendency for cross-slip and the dislocation network model for nucleus of recrystallized grain.

KEY WORDS: Fe–3%Si alloy; single crystal; deformation; recrystallization.

1. Introduction

Recrystallization has long been studied because it is virtually the only measure for texture control of metals and alloys in which phase transformation does not occur in appropriate temperature range. Because almost all structural metallic materials are polycrystalline and heavy plastic deformation is applied in their production process, the main stream of recrystallization study has been the evaluation of texture changes associated with large deformation and subsequent annealing.

For the complete understanding of recrystallization, we should examine the dislocation mechanisms of recrystallization through detailed analysis of deformation microstructures, *e.g.* slip morphology and associated local orientation change, because recrystallized grains (RGs) are formed through migration, rearrangement and annihilation of dislocations accumulated in deformed materials. Experimental studies using single crystals and bicrystals as samples are important for unambiguous evaluation of the deformation microstructure.^{1–9)} In addition, relatively small strain is preferred for slip analysis of such samples.

Inoko and coworkers^{5–9)} have been studying the deformation microstructures of tensile-deformed aluminum single crystals and bicrystals, and their initial stage of recrystallization. Recently, they reported interesting results of {001}<112> and {111}<111> oriented aluminum single crystals tensile-deformed at room temperature, which exhibited multiple slip from the initial stage of plastic deformation.^{10–14)} For these single crystals, bands of large orientation gradient such as deformation bands were not formed. Hence, the misorientation in their deformed microstructures was within a range of 5°. Despite such macroscopically uniform deformation, RGs were formed in the {111}<112> single

crystals after annealing.^{10,11)} Many RGs had {111}<111> rotation relationship with the orientation of the deformation matrix. On the other hand, recrystallization did not occur in the {001}<111> single crystal.¹⁰⁾ This difference reflects well their deformation behaviors, especially tendency to cross-slip. Among these single crystals, the work-hardening rate was the largest for the {111}<111> single crystal and the nominal tensile stresses at a strain of 0.22 were 22 MPa, 32 MPa and 71 MPa for the {001}<112>, {112}<112> and {111}<111> single crystals, respectively. In the {001}<111> single crystal, cross-slip with large step height of approximately 100 μm developed, which resulted in a release of strain accumulation at the intersections of slip bands, most likely recrystallization sites for single crystals of multiple slip orientation. On the other hand, cross-slip with large step height was suppressed in the {111}<112> and {112}<112> single crystals.

Although extensive studies have been conducted for texture formations associated with deformation and annealing, relatively smaller numbers of studies have been done for dislocation mechanisms of recrystallization in BCC metals. Okada *et al.*^{15,16)} investigated slip deformation and recrystallization of compressed Fe–30%Cr alloy single crystals. The RGs formed in a shear band had {110}<110> rotation relationship with the orientation of the shear band. In the proceeding paper, we reported that in a tensile-deformed {111}<112> Fe–3%Si alloy single crystal, recrystallization did not occur even in the neck portion of the sample.¹⁷⁾ On the other hand, in a tensile-deformed Fe–3%Si alloy bicrystal containing {111}<112> and {001}<110> grains, slip in the {111}<112> grain changed markedly from that in the single crystal due to the constrain from the grain boundary, and RGs were formed only in the non-uniformly deformed region close to the grain boundary in the {111}<112> grain.

From the experimental results described above, close re-

relationship between deformation and recrystallization is expected in BCC metal single crystals. One major objective of the present study is to investigate deformation and annealing behaviors of single crystals of Fe–3%Si alloy with various tensile orientations. The other objective is to study the recrystallization in uniformly deformed Fe–3%Si alloy single crystal of $\{111\}\langle 112 \rangle$ orientation deformed by light rolling.

2. Experimental Procedure

Single crystal samples were spark-cut from a coarse-grained Fe-3%Si alloy polycrystal prepared by JFE Steel Co. Ltd. The dimension of the gauge portion of the samples for tensile deformation was 4 mm×12 mm×2 mm in width, length and thickness. Four samples were prepared for tensile deformation. The tensile directions of the samples are plotted in a stereographic triangle in **Fig. 1**. Sample No. 4 had the same {111}(112) orientation as the one in the proceeding paper.¹⁷⁾ These samples were tensile-deformed at the strain rate of 5.6×10^{-5} to $1.4 \times 10^{-4} \text{ s}^{-1}$, to strains of 0.25 to 0.65. The dimension of the sample for rolling was 4 mm×46 mm×2 mm in width, length and thickness. The initial orientation of the rolled sample is presented in **Fig. 2**. The normal direction (ND) of the rolled surface and the rolled direction (RD) were [111] and $[2\bar{1}\bar{1}]$, respectively. In order to obtain uniform deformation, relatively light rolling of 25% was applied. The deformed microstructure was examined using a scanning electron microscope (SEM JEOL JSM-6400). Orientation change associated with deformation was determined from electron channeling patterns

Fig. 1. Tensile directions of single crystal samples plotted in a stereographic triangle. The tensile directions of the sample No. 3 and No. 4 are $\langle 123 \rangle$ and $\langle 112 \rangle$, respectively.

Fig. 2. The initial orientation of the rolled sample. The rolling direction (RD) and normal direction (ND) are $[2\bar{1}\bar{1}]$ and $[111]$, respectively. The tangential direction (TD) is $[0\bar{1}1]$.

(ECPs) taken with the SEM.

The annealing was conducted for the tensile-deformed samples at 1173 to 1273 K for 180 to 780 s. The rolled sample was annealed at 873 K for 720 s. After annealing, orientation of RGs was determined from their ECPs. For the rolled sample, the rolled surface was removed by 0.25 mm to observe the formation of RGs in the interior of the sample.

3. Results

3.1. Tensile-deformed Samples

Nominal stress-strain curves for the four tensile-deformed samples are presented in **Fig. 3**. In all samples, necking started from the early stage of plastic deformation. After the neck formation, plastic deformation proceeded mainly in the neck portion. Hence, nominal work-hardening was very small for all samples. The overall appearance of Sample No. 4 with the nominal strain of 0.25 is shown in **Fig. 4**. After annealing, the four samples did not recrystallize. The difficulty in recrystallization for tensile-deformed Fe-3%Si single crystals is discussed in Sec. 4.1.

3.2. Rolled Sample

SEM image of the side surface (TD surface) of the rolled sample is presented in **Fig. 5**. Narrow folds are recognized. The arrangement of the folds is totally different from that of shear bands formed in 60–70% rolled Fe–3%Si alloy single crystals of $\{111\}\{112\}$ orientation.^{1–4)} Since the for-

Fig. 3. Nominal stress–strain curves for the tensile-deformed single crystal samples.

Fig. 4. The shape of the tensile-deformed sample No. 4.

Fig. 5. The TD surface of the rolled sample.

Fig. 6. Typical slip bands in the TD surface of the rolled sample.

mation of folds in the present sample did not affect its recrystallization, we consider that the deformation was almost uniform. A typical SEM image of slip bands is presented in Fig. 6. Slips on $\{110\}$ planes and $\{112\}$ planes were observed. Most slip bands are straight except for Slip 2 which cross-slips between $(1\bar{1}0)$ [or $(\bar{1}01)$] and $(2\bar{1}1)$ [or $(\bar{2}1\bar{1})$] planes.

After annealing, RGs were formed. Recrystallization observed in the TD surface is presented in Fig. 7. Since the arrangement of RGs had no relationship with the position of the folds, we consider that recrystallization in the present sample occurred in uniformly deformed sample. The optical microscopic image of the ND surface along with its schematic is presented in Fig. 8. The orientation of 79 RGs was measured and compared with that of non-recrystallized region, *i.e.* deformed matrix. Out of 79 RGs, 44 RGs had $\langle 110 \rangle$ rotation relationship with the deformed matrix. The rotation axes are classified in Table 1. In the present study, $\langle 112 \rangle$ rotation relationship was not checked because it is

Fig. 7. (a) SEM image of the TD surface of the rolled sample after annealing. Positions of some of the folds are indicated by elliptic marks. (b) A schematic of (a). Grain boundaries around recrystallized grains are drawn.

Fig. 8. (a) Optical micrograph of the ND surface of the rolled sample after annealing. (b) A schematic of (a).

generally found in RGs formed at shear bands in heavily (60 to 70%) rolled Fe-3%Si alloy single crystals. About the half of $\langle 110 \rangle$ -rotated RGs (20 out of 44) were rotated about the TD ($[0\bar{1}1]$) axis. The $\langle 110 \rangle$ pole figure of such RGs is presented in Fig. 9. (In the figure, the orientation of the deformed matrix was determined by averaging the orientation of several points in non-recrystallized region. The misorientation of the deformed matrix was within a range of 5° .)

Table 1. Rotation axis of RGs formed at the rolled surface with respect to the matrix.

[110]	9
[101]	6
[011]	5
[10 $\bar{1}$]	2
[1 $\bar{1}$ 0]	2
[0 $\bar{1}$ 1]	20
Twin orientation	0
Others	35
Total	79

Fig. 9. $\langle 110 \rangle$ pole figure of RGs rotated about the TD ($[0\bar{1}1]$) axis with respect to the deformed matrix. The RGs were formed at the rolled surface.

The sense of rotation is singular, that is, the RGs are rotated counterclockwise about the $[0\bar{1}1]$ axis.

The rolled surface was removed by 0.25 mm to observe recrystallization in the interior of the sample where the direct effect of the roller, *i.e.* friction, is considered to be smaller. The arrangement of RGs is presented in **Fig. 10**, which is different from that at the rolled surface (Fig. 8). The orientation of 104 recrystallized grains were measured and compared with that of the deformed matrix. The results are summarized in **Table 2**. The occurrence of each $\langle 110 \rangle$ rotation relationship is different from that observed at the rolled surface (Table 1). The major difference is the very small number of occurrences of the TD ($[0\bar{1}1]$)-rotated RGs in the interior; 1/104 compared with the high value of 21/79 at the rolled surface. We also note large number of occurrences of $[1\bar{1}0]$ - and $[10\bar{1}]$ -rotation relationship in the interior of the sample, 20/104 and 9/104, respectively. The $\langle 110 \rangle$ pole figures for $[1\bar{1}0]$ - and $[10\bar{1}]$ -rotated RGs are presented in **Figs. 11** and **12**, respectively. The interesting feature is

Fig. 10. RGs formed in the interior of the rolled sample.

Table 2. Rotation axis of RGs formed in the interior with respect to the matrix.

[110]	9
[101]	8
[011]	2
[10 $\bar{1}$]	9
[1 $\bar{1}$ 0]	20
[0 $\bar{1}$ 1]	1
Twin orientation	3
Others	52
Total	104

that the sense of rotation is almost singular, that is, most RGs are rotated counterclockwise about the $[1\bar{1}0]$ -axis or clockwise about the $[10\bar{1}]$ -axis. The singular rotation of RGs was reported in compressed single crystals of Fe–30%Cr alloy.^{15,16} In the compressed Fe–30%Cr single crystals, a shear band was formed and recrystallization occurred only in the shear band. The counterclockwise rotation of RGs with respect to the orientation of the shear band was opposite to the deformation-induced clockwise rotation of the shear band from the initial orientation. Such singular rotation was explained by dislocation network model of RGs.^{15,16} Although it is difficult to apply the same discussion to the present case due to the complexity of rolling deformation, the singular rotation of RGs is noteworthy.

Fig. 11. Orientation of RGs rotated about the $[1\bar{1}0]$ axis with respect to the deformed matrix. The RGs were formed in the interior of the sample.

Fig. 12. Orientation of RGs rotated about the $[10\bar{1}]$ axis with respect to the deformed matrix. The RGs were formed in the interior of the sample.

4. Discussion

4.1. Relationship between Deformation and Recrystallization in Tensile-deformed Single Crystals

For tensile-deformed and subsequently annealed single crystals of aluminum, it is known that recrystallization behavior varies with their tensile direction. Inoko and coworkers¹⁰⁻¹³⁾ have conducted a series of studies for $\langle 001 \rangle$ -, $\langle 112 \rangle$ - and $\langle 111 \rangle$ -oriented aluminum single crystals. When deformation was applied at room temperature (RT), prominent cross-slip as large as $100\ \mu\text{m}$ occurred in the $\langle 001 \rangle$ single crystal. The flow stress saturated at the early stage of plastic deformation; the work-hardening was very small. The slip bands at the surface were coarse and the interior dislocation microstructure was characterized by coarse cells bounded by cell walls with low dislocation density. On the contrary, in the $\langle 111 \rangle$ single crystal, cross-slip with large step height

was suppressed. The slip bands were very fine and the cell size was small, bounded by cell walls with high dislocation density. The work hardening of the $\langle 111 \rangle$ single crystals was very large. At a nominal strain of 0.22, the flow stress of the $\langle 111 \rangle$ single crystal (71 MPa) was about three times as large as that of the $\langle 001 \rangle$ single crystal (22 MPa). The slip morphology of the $\langle 112 \rangle$ single crystal was similar to that of the $\langle 111 \rangle$ crystal with relatively low flow stress (32 MPa at a strain of 0.22). After annealing, very fine RGs were formed in the $\langle 111 \rangle$ single crystal. On the other hand, RGs were not formed in the $\langle 001 \rangle$ single crystal. In the $\langle 112 \rangle$ single crystal, coarse RGs were formed. The number of RGs in the $\langle 112 \rangle$ single crystal was smaller than that in the $\langle 111 \rangle$ single crystal, which suggests lower density of recrystallization sites in the former.

The close relationship between slip behavior and recrystallization was confirmed in $\langle 001 \rangle$ aluminum single crystals deformed in tension at liquid nitrogen temperature (LNT).^{12,14)} The flow stress of the $\langle 001 \rangle$ single crystal deformed at LNT (106 MPa at the strain of 0.22) is about five times as large as the flow stress obtained at RT (22 MPa). Cross-slip with large-step height was suppressed in the LNT crystal. The slip bands were very fine similar to that of $\langle 111 \rangle$ single crystal. After annealing, coarse RGs were formed.

The above results are explained by the dependence of cross-slip on tensile directions for FCC single crystals. It is known that the ratio of the Schmid factor of cross slip system (m_c) to that of primary slip system (m_p) depends on the tensile direction.¹⁸⁾ For the tensile directions on the line connecting the $\langle 112 \rangle$ and $\langle 011 \rangle$ poles in a stereographic triangle, the m_c/m_p ratio is zero. For tensile directions staying in the area close to the $\langle 111 \rangle$ pole, the m_c/m_p ratio takes negative values, that is, cross-slip is suppressed. For tensile directions in the area close to the $\langle 001 \rangle$ pole, the m_c/m_p ratio takes positive values, that is, cross-slip is promoted. The tendency for cross-slip also depends on deformation temperature. When temperature is lowered, cross-slip is suppressed due to lower thermal activation. Here we should note that the above discussion is based only on geometrical consideration. For FCC metals with low stacking fault energy such as copper and nickel, care must be taken for their difficulty in cross-slip even at RT.

For tensile-deformed single crystals of BCC metals such as Fe-3%Si, the m_c/m_p ratio always takes positive values. For tensile direction on the $\langle 112 \rangle$ - $\langle 011 \rangle$ line, the ratio takes the minimum value of 0.5. This suggests very easy cross-slip for any tensile direction of single crystal of BCC metals. Hence, the strain accumulation is considered to be small at the intersections of slip bands, the most likely positions of recrystallization nucleus in tensile-deformed single crystals of multiple-slip orientation. As a result, the work-hardening is very small. Because of small strain accumulation, such crystals are unlikely to recrystallize when annealed.

4.2. Recrystallization Nucleus

For uniformly deformed crystals, we should assume nucleation sites for recrystallization other than deformation bands. Inoko and coworkers^{19,20)} analyzed the deformation at the intersections of slip bands and proposed that such in-

Fig. 13. A model of RG nucleus formed at the intersection of two slip bands. In this figure, shear of two slip bands rotates the nucleus in the clockwise direction.

tersection could be the nucleation sites of RGs. A schematic of their model is presented in Fig. 13. The nucleus of RG is bounded by two sets of dislocation networks introduced by intersecting slips. From geometrical consideration of Fig. 13, it is apparent that the rotation axis of RG is normal to the plane on which two slip directions coexist. Hence, for BCC metals, rotation axis of RG is $\langle 110 \rangle$.

In the present study, the predominant rotation axis of RGs formed at the rolled surface is $[0\bar{1}1]$ (TD). In the interior of the sample, the predominant rotation axes are $[10\bar{1}]$ and $[1\bar{1}0]$. Following the geometrical model of Fig. 12, the slip directions required for the $[0\bar{1}1]$ -rotation are $[\bar{1}11]$ and $[111]$. For $[10\bar{1}]$ - and $[1\bar{1}0]$ -rotations, the corresponding slip directions are $[1\bar{1}1]$ and $[111]$, and $[11\bar{1}]$ and $[111]$, respectively. Here it should be noted that for all rotations slip along the $[111]$ direction is involved. Since slip systems with slip direction parallel to $[111]$ (ND) are minor ones in the present sample, their activation is probably limited in small regions. In addition, such slip systems do not produce steps on the TD surface. Hence, it is virtually impossible to observe their slip bands in SEM images such as Fig. 6. In the above mentioned model, we assume a combination of major slip systems such as those with slip direction parallel to $[\bar{1}11]$, $[1\bar{1}1]$ or $[11\bar{1}]$ and minor ones, i.e. slip along $[111]$ direction for the formation of a RG nucleus. The possibility that such a combination acts as a RG nucleus was proposed for recrystallization in aluminum single crystals tensile-deformed along $\langle 001 \rangle$ direction at LNT and subsequently annealed.^{10,13,14)} In such single crystals, in addition to eight primary slip systems, activation of slip systems with Schmid factor of zero was induced by the local strain accumulation caused by the complete suppression of prominent cross-slip. The formation of RGs rotated about four $\langle 111 \rangle$ axes in both clockwise and counterclockwise directions with respect to the deformed matrix was geometrically explained by the combination of major and minor slip systems. We consider that similar explanation is applicable to the recrystallization of the present sample. The variation of predominant rotation axis of RGs along the thickness of the sample is attributable to different degree of activation of the slip along the $[\bar{1}11]$ direction, that is, the slip along the

$[\bar{1}11]$ direction almost parallel to the RD is active at the rolled surface probably due to friction of the roller, but its activity diminishes in the interior of the sample.

5. Summary

Fe-3%Si alloy single crystal samples with various tensile directions were deformed to nominal strains of 0.25 to 0.65 and subsequently annealed. Neck developed in all samples. Recrystallization did not occur even in the neck portion. This is probably due to the fact that in tensile-deformed BCC metal single crystals, cross-slip easily occurs for all tensile directions and strain accumulation is small.

Fe-3%Si alloy single crystal sample of $\{111\}\langle 112 \rangle$ orientation was lightly rolled to 25%. The sample was almost uniformly deformed without shear band formation. After annealing, many RGs were formed. At the ND surface, a large number of RGs were rotated about the TD axis with respect to the deformed matrix orientation. However, the ratio of TD-rotated RGs decreased in the interior of the sample. This is probably due to the difference in slip activity almost parallel to the RD direction, which was caused by the friction from the roller.

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