Cold-rolling and Recrystallization Texture Formation in Electro-deposited Pure Iron with a Sharp and Homogeneous γ -fiber

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(Received on October 14, 1997; accepted in final form on January 22, 1998)

The formation of the cold-rolling and annealing texture has been investigated in electrolytically deposited pure iron having a sharply developed $\langle 111 \rangle$ //ND fiber texture without any anisotropy along γ -fiber nor any orientation density along other fibers. In no respect of cold reduction, $\{111\}\langle 112 \rangle$ texture is formed after cold-rolling. The same texture still remains after the recrystallization in the 65% cold-rolled sheet, whereas the position of the peak is shifted from $\{111\}\langle 112 \rangle$ towards $\{111\}\langle 110 \rangle$ through recrystallization in the 80% cold-rolled sheet. The nucleation texture seems to be responsible for the difference of recrystallization texture between the 65 and 80% cold-rolled sheets. Additionally, it is thought that the growth of the recrystallized grains also plays an important role. It is considered that the recrystallized nuclei with $\{111\}\langle 112 \rangle$ orientation can hardly grow into the deformed matrix of the 80% cold-rolled sheet because they frequently encounter the deformed grains with nearly the same orientation and thus suffer from a reduced mobility. On the other hand, nuclei with a $\{111\}\langle 110 \rangle$ orientation can easily grow into the deformed $\{111\}\langle 112 \rangle$ grains because of the favorable growth orientation relationship between those. Conversely, in 65% cold-rolled sheet, the nuclei with $\{111\}\langle 112 \rangle$ are considered to grow more easily since the frequency of pinning due to limited mobility of a low angle grain boundary is rather small, as compared to that of the 80% cold-rolled sheet. The same mobility argument as applied to the growth of nuclei seems to hold also for the growth of subgrains, namely, nucleation texture formation.

KEY WORDS: cold-rolling; annealing; recrystallization; texture; microtexture; EBSD; nucleation and growth; pure iron; electrolytically deposited iron.

1. Introduction

It is well known that the annealing texture with $\langle 111 \rangle$ //ND fiber (γ -fiber) develops through cold-rolling and recrystallization in BCC iron with fine grain size.^{1–3)} The intensity level of this γ -fiber is not uniformly distributed in most cases. In fact, it is commonly observed after annealing that the peak position is centered near $\{111\}\langle 112 \rangle$ or $\{111\}\langle 110 \rangle$. The $\{111\}\langle 112 \rangle$ texture tends to form upon annealing of heavily cold-rolled steel sheets, whereas the $\{111\}\langle 110 \rangle$ annealing texture becomes dominant in the steels cold-rolled with less reductions. In comparison to the textures of the as cold-rolled state, the relative importance of those components is reversed. In the cold-rolled sheets, namely, $\{111\}\langle 110 \rangle$ is characteristic for heavy rolling, while the $\{111\}\langle 112 \rangle$ is characteristic for light rolling.^{4,5)} The initial grain boundary has been considered to play an important role not only in the formation of γ -fiber but also in this

change of peak position through annealing. Inagaki⁶⁾ has investigated the recrystallization texture in some polycrystalline irons with sharply developed textures. He has pointed out that the boundary region of a $\{111\}\langle 112 \rangle$ grain in the neighborhood of a $\{111\}\langle 110 \rangle$ grain rotates towards the more stable $\{111\}\langle 110 \rangle$ orientation under influence of severe local strain. Hence, these boundary regions provide the most potential recrystallization nuclei of the latter orientation so that the main peak is shifted from $\{111\}\langle 112 \rangle$ to $\{111\}\langle 110 \rangle$ while recrystallization progresses. Hutchinson⁴⁾ has investigated the recrystallization nucleation at the grain boundaries between (111)[$\bar{1}10$] and (111)[$\bar{1}\bar{1}0$] single crystals. It is reported that $\{111\}\langle 112 \rangle$ grains nucleated at the grain boundary grow into the deformed matrix resulting in $\{111\}\langle 112 \rangle$ texture after full recrystallization.⁴⁾ The more recent investigations^{7–9)} using EBSD (Electron Back-Scattering Pattern), which is sometimes more convenient than TEM or ECP (Electron Channeling

Fig. 1. $\{100\}$ pole figure of the starting material, as electrolytically deposited, measured at the midplane.

Pattern),¹⁰⁾ suggests that in Ti-IF steel γ -fiber orientations nucleate not only at grain boundaries but also at grain interior. Considering recrystallization in the deformed grains consisting mainly of $\{111\}\langle 110 \rangle$ subgrains, it was observed that the nucleating subgrains were the ones showing high local misorientation with the dominant $\{111\}\langle 110 \rangle$ component.⁹⁾ In these senses,^{4,6,9)} it is obvious that the nucleation orientation plays an essential role for the change in the peak position through recrystallization. However, it has not yet been clarified how the nuclei grow into the deformed matrix of the polycrystalline materials. A particular obstacle in such investigation is that one can never recognize the orientation of the deformed grains which were consumed by recrystallizing grains once after the recrystallization has proceeded to some extent and thus it is difficult to know the orientation relationship between the deformed and recrystallized grains. In this respect, it may be interesting to investigate the recrystallization behavior of a material having a sharp initial texture rather macroscopically as Inagaki⁶⁾ has already examined. Electrolytically deposited iron is known to have an extremely sharp $\langle 111 \rangle // \text{ND}$ ring texture without any other fibers such as the α -fiber.¹¹⁾ Furthermore, the electrolytically deposited iron has not yet been examined in detail with respect to the cold-rolling and recrystallization texture formation. The aim of this study is to investigate the mechanism of the orientation change in the peak position along γ -fiber during recrystallization using this specific material, from the point of view not only of nucleation but also of the growth of the nuclei.

2. Experimental Procedure

Electrolytically deposited pure iron produced by Toho Zinc Co., Ltd. was used as a starting material. The texture of this material measured at midplane consists of an extremely sharp and isotropic γ -fiber without any components along other fibers (such as the α -fiber or the ND// $\langle 100 \rangle$ -fiber) as shown in Fig. 1. The surface of the starting materials were removed mechanically since the

Fig. 2. Optical microstructure of transverse section in the starting material, as electrolytically deposited.

Table 1. Chemical composition of specimen. (mass ppm)

C	Si	Mn	P	S	Al	N	O
6	1	1	1	2	2	9	18

surface had somewhat different texture from that of the midplane. An optical micrograph of this material is shown in Fig. 2. It is visible that the microstructure consists of small grains elongated along ND. The chemical composition of this iron, represented in Table 1, shows that this material has rather high purity.

Specimens of 3 mm thickness were cold-rolled with reduction of 65 and 80 %, respectively. The cold-rolled specimens were then isothermally annealed at 400°C for various periods, in order to assess the recrystallization kinetics. Annealing was finished by simple air-cooling to room temperature. The midplane textures of cold-rolled and annealed sheets were determined by the conventional X-ray diffraction method. The EBSD technique was employed to measure the crystal orientations of the recrystallized grains in partially recrystallized specimens.^{9,12)}

3. Results

Recrystallization takes place in both 65 and 80 % cold-rolled specimens even at 400°C. It is considered that the high purity,¹³⁾ the small grain size^{11,14)} and the initial texture of the starting materials¹¹⁾ decrease the recrystallization temperature. The higher cold-rolling reduction clearly makes the recrystallization kinetics faster as represented in Fig. 3. The optical microstructures of cold-rolled and recrystallized specimens are shown in Fig. 4. Considering the grain size after recrystallization, the number of nucleation site in 80 % cold-rolled material is supposed to be a lot more than in the 65 % cold-rolled sheet.

The textures of cold-rolled, partially recrystallized and fully recrystallized materials are shown in Fig. 5. In both 65 and 80 % cold-rolled sheets, a $\{111\}\langle 112 \rangle$ component is observed. It is noteworthy that the cold-rolling reduction of 80 % made the $\{111\}\langle 112 \rangle$ component stronger, although it is widely accepted that $\{111\}\langle 110 \rangle$ is the more stable rolling component rather than $\{111\}\langle 112 \rangle$.^{6,15)} In 65 % cold-rolled sheet, the

Fig. 3. Evolution of the recrystallized volume fraction as a function of annealing time at 400°C in both 65 and 80% cold-rolled sheets.

$\{111\}\langle 112 \rangle$ component remains dominant also after the recrystallization. In 80% cold-rolled sheet, however, the position of the peak is shifted from $\{111\}\langle 112 \rangle$ towards $\{111\}\langle 110 \rangle$ while recrystallization progresses. The results obtained here are summarized in Table 2.

4. Discussion

4.1. Relation between Nucleation and Recrystallization Textures

In order to understand the reason why a $\{111\}\langle 110 \rangle$ texture in 80% cold-rolled specimen is formed after recrystallization while a $\{111\}\langle 112 \rangle$ texture is formed in 65% cold-rolled sheet, the orientations of the recrystallized grains at the early stage of recrystallization (when the recrystallized volume fraction is around 5%) were measured by EBSD. ODF textures based on the EBSD measurements are shown together with the ECC

Fig. 4. Optical microstructure of transverse section in (a) 65% cold-rolled and (b) fully recrystallized sheet, (c) cold-rolled with 80% reduction and (d) fully recrystallized sheet, respectively.

Fig. 5. The change of $\{100\}$ pole figure for 65 and 80% cold-rolled sheets with the progress of recrystallization.

(Electron Channeling Contrast) image in **Fig. 6**. ODF textures were calculated under the assumption that each orientation measured by EBSD is at the center of a Gaussian peak with a spread of 7 degrees. These textures will be called nucleation textures, hereafter. It is obvious from Fig. 6 that a $\{111\}\langle 112\rangle$ nucleation texture is strongly formed in 65 % cold-rolled sheet. In 80 % cold-rolled sheet, however, a $\{111\}\langle 110\rangle$ nucleation texture is observed together with a nearly isotropic distribution along the γ -fiber. Therefore, it seems natural to think that the nucleation texture plays a dominant role in the formation of the recrystallization texture.

4.2. Influence of Growth of Nuclei on Texture Evolution

As mentioned above, the nucleation texture seems to play an important role in the development of recrystallization texture. However, the question still remains why the $\{111\}\langle 112\rangle$ orientation can grow into the matrix only in 65 % cold-rolled sheet although $\{111\}\langle 112\rangle$ density in the nucleation texture of 80 % cold-rolled sheet is as high as that of 60 % cold-rolled sheet. **Figure 7**

Table 2. Summary of texture evolution through cold-rolling and annealing.

Initial Texture	Cold-rolling Red. (%)	Cold-rolling Texture	Nucleation Texture	Recrystallization Texture
Homogeneous	65	$\{111\}\langle 112\rangle$ (weak)	$\{111\}\langle 112\rangle$	$\{111\}\langle 112\rangle$
γ -fiber	80	$\{111\}\langle 112\rangle$ (strong)	$\{111\}\langle 110\rangle$	$\{111\}\langle 110\rangle$

represents the changes in the intensity of $\{111\}\langle 112\rangle$ and $\{111\}\langle 110\rangle$ with the recrystallized fraction, calculated from three dimensional analysis using the Vector method.¹⁶⁾ It is realized from Fig. 7 that the difference of intensity between the $\{111\}\langle 112\rangle$ and $\{111\}\langle 110\rangle$ components in 80 % cold-rolled sheet is larger than in 65 % cold-rolled sheet. Taking this difference of intensity in the deformed matrix into account, the difference of recrystallization texture may become understandable. Namely, in 80 % cold-rolled sheet, the recrystallized nuclei with $\{111\}\langle 112\rangle$ scarcely meet deformed grains having any other orientation than $\{111\}\langle 112\rangle$. It is well known that the boundary between grains having similar orientations hardly move because of its small mobility. Therefore, it is considered that the recrystallized nuclei with $\{111\}\langle 112\rangle$ hardly grow into the deformed matrix in the 80 % cold-rolled sheet. In contrast to the $\{111\}\langle 112\rangle$ nuclei, the nuclei having $\{111\}\langle 110\rangle$ orientations have a much higher probability to encounter the deformed matrix grains with different orientations, such as $\{111\}\langle 112\rangle$ grains which have 30 degrees misorientation about the common $\langle 111\rangle$ axis. As a consequence, the $\{111\}\langle 110\rangle$ texture components are more likely to be stronger during recrystallization as compared to the $\{111\}\langle 112\rangle$ orientations.

In order to clarify the influence of the intensity difference between $\{111\}\langle 112\rangle$ and $\{111\}\langle 110\rangle$ in the deformation textures on the growth of recrystallized nuclei, the following calculation has been done. The model simulating the recrystallization texture which has been developed and applied to the recrystallization

Fig. 6. ECC image and nucleation texture on $\phi_2=45^\circ$ section in Euler space (*i.e.* texture of recrystallized grains after 5% recrystallization fraction) measured by EBSD in (a) 65 % and (b) 80 % cold-rolled sheets. Iso-levels: 0.7–1–1.4–2–2.8–4–5.6–8–11–16.

Fig. 7. Change in the density of $\{111\}\langle 112 \rangle$ and $\{111\}\langle 110 \rangle$ in (a) 65% and (b) 80% cold-rolled sheets with annealing time or recrystallized volume fraction.

textures of several kinds of steel sheets by one of the present authors¹⁷⁻²¹⁾ was used. The basic assumption of this method is that the process of recrystallization can be modelled in two sequential steps: first nucleation and then growth. Throughout the calculations, a discrete set of orientations g_i of volume fractions $f(g_i)$ is used to represent the overall ODF.

The recrystallization texture $f^R(g_i)$ is obtained by allowing the growth probability function $P_G(g_i)$ to operate on the nucleation texture $f^N(g_i)$:

$$f^R(g_i) = c(P_G(g_i))f^N(g_i) \quad (1)$$

where c is the appropriate normalization factor. In the calculation here, $f^N(g_i)$ is given by the nucleation textures measured by EBSP. The growth probability of each nucleus $P_G(g_i)$ is given in the following manner:

$$\omega \geq 15^\circ: P_G(g_i) = 1 \quad (2)$$

$$\omega < 15^\circ: P_G(g_i) = 0 \quad (3)$$

where ω is the misorientation between the nucleus and the deformed grain. The growth probability of nuclei depends on the mobility, which corresponds to the misorientation with the surrounding matrix. Although it is possible to apply a continuous function of the growth probability against misorientation, Eq. (2) is thought to

Fig. 8. Recrystallization texture on $\phi_2 = 45^\circ$ section in Euler space calculated with the measured nucleation texture and the reproduced cold-rolling texture. (a) 65% and (b) 80% cold-rolled sheets. Iso-levels: 0.7-1-1.4-2-2.8-4-5.6-8-11-16.

Fig. 9. Calculated distribution of the orientation density along γ -fiber taken from Fig. 8.

be a close approximation of reality, given the sharp drop in mobility when the misorientation ω falls below 15 degrees.²²⁾ Therefore, for simplification, the growth probability were set as Eqs. (2) and (3). Cold-rolling ODF textures were reproduced based on measured textures. The results of the calculation are presented in Fig. 8, while the intensity distribution along γ -fiber is shown in Fig. 9. It is revealed by comparing Figs. 8 and 9 with Fig. 7 that this model can realistically simulate the recrystallization texture. However, because it is difficult in this calculation to distinguish the influence of nucleation texture from that of the growth probability, a random texture along the γ -fiber was employed as nucleation texture in order to assess the effect of the growth selectivity. The growth probability $P_G(g_i)$ is modelled according to Eqs. (2) and (3). The changes in the intensity along the γ -fiber, which are calculated here, are described in Fig. 10. It is obvious that the $\{111\}\langle 112 \rangle$ texture of 65% cold-rolled sheet is more developed than that of the material with 80% cold reduction. This calculation suggests that the nuclei having $\{111\}\langle 112 \rangle$ orientations in 65% cold-rolled sheet grow easier into the deformed matrix as compared to those in 80% cold-rolled sheet because of the smaller density of its $\{111\}\langle 112 \rangle$ component in the deformed texture. It may be concluded that the growth selectivity of nuclei, which is influenced by the intensity difference between $\{111\}\langle 112 \rangle$ and $\{111\}\langle 110 \rangle$ in the cold-rolling texture,

Fig. 10. Change in the intensity $f(g)$ along the γ -fiber, calculated with a nucleation texture composed of a homogeneous distribution along γ -fiber and reproduced cold-rolling texture.

is important as well as the nucleation texture itself.

4.3. Mechanism for Nucleation Texture Formation

Although it is difficult to understand how the nucleation textures shown in Fig. 6 are formed, the following interpretation might be plausible. Because $\{111\}\langle 112 \rangle$ seems to be the final orientation formed by crystal rotation due to the cold-rolling in this study, it is reasonable to think that some of the deformed grains with $\{111\}\langle 112 \rangle$ should have the largest strain, taking into account the fact that the starting material had sharp and isotropic $\langle 111 \rangle // \text{ND}$ ring texture. Therefore, there must be more potential nuclei in deformed $\{111\}\langle 112 \rangle$ grains than in deformed $\{111\}\langle 110 \rangle$ grains. Thus, with respect to the driving force, a $\{111\}\langle 112 \rangle$ nucleation texture should be formed both in 65 and 80% cold-rolled sheets. However, this speculation is not supported by the present observations. Therefore, the influence of subgrain boundary mobility on nucleation texture formation will be taken into account. Namely, when subgrain growth is considered to be a possible nucleation mechanism, the mobility of subgrain boundary migration, which is mainly determined by the orientation relationship between a subgrain and the surrounding deformed matrix, is supposed to be important for nucleation texture formation. It follows that the nuclei with $\{111\}\langle 112 \rangle$ orientation would be difficult to be formed in 80% cold-rolled specimen because of its strongly developed $\{111\}\langle 112 \rangle$ cold-rolling texture. In contrast, nuclei with $\{111\}\langle 110 \rangle$ are more likely formed because of the weaker intensity of this component in the cold-rolling texture. In 65% cold-rolled sheet, as opposed to the 80% cold-rolled one, it is thought that the deformed grains with $\{111\}\langle 112 \rangle$ can still nucleate because of the higher mobility due to the less strong intensity of $\{111\}\langle 112 \rangle$ in the deformed matrix. In fact the same mobility argument as applied above to the growth of nuclei into deformed matrix also holds for the growth of subgrains into their environment. Further investigations are

required to clarify the mechanism of nucleation texture formation in the future.

5. Conclusion

The formation of the cold-rolling and annealing texture has been investigated in electrolytically deposited pure iron having sharply developed $\langle 111 \rangle // \text{ND}$ ring texture without any anisotropy along γ -fiber nor any other orientations. The conclusions obtained are summarized as follows:

(1) In both 65 and 80% cold-rolled sheets, $\{111\}\langle 112 \rangle$ texture is formed after rolling. However, the density difference between $\{111\}\langle 112 \rangle$ and $\{111\}\langle 110 \rangle$ in 80% cold-rolled material is larger than that in the 65% cold-rolled sheet.

(2) In 65% cold-rolled sheet, $\{111\}\langle 112 \rangle$ orientation still remains dominant after the recrystallization whereas the position of the peak is shifted from $\{111\}\langle 112 \rangle$ towards $\{111\}\langle 110 \rangle$ through recrystallization in 80% cold-rolled sheet.

(3) EBSD measurement indicates that the recrystallized grains at the stage of 5% recrystallized volume fraction have near $\{111\}\langle 112 \rangle$ orientations in the 65% cold-rolled sheet. In 80% cold-rolled sheet, however, $\{111\}\langle 110 \rangle$ nucleation texture is observed together with a homogeneous spread along γ -fiber.

(4) The nucleation texture plays an important role in the formation of recrystallization texture. Nevertheless, it is thought that the growth of the recrystallized grains also affects the texture development. The nuclei with $\{111\}\langle 112 \rangle$ orientation in 65% cold-rolled sheet are easier to consume the deformed matrix than they do in 80% cold-rolled sheet since their frequency to encounter a deformed grain with nearly the same orientation is much smaller in 65% rolled sheet as compared to that of 80% rolled sheet.

(5) A simple calculation was made to confirm the influence of the growth of recrystallized nuclei on the peak position along γ -fiber after recrystallization completes.

(6) For the nucleation texture formation, the mobility as well as driving force is considered important.

Acknowledgments

The authors are indebted to TOHO ZINC Co., Ltd. for their effort to provide the suitable samples for this research work. Helpful discussions with Dr. T. Senuma in Nippon Steel Corporation are gratefully acknowledged.

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