Nucleation of a \{111\} Recrystallized Grain at the Grain Boundary of Cold Rolled Polycrystalline Iron*

By Hirosuke Inagaki**

Using transmission electron microscopy and selected area electron diffraction, nucleation of \{111\} recrystallized grains at grain boundaries was studied in an Fe–0.02% C alloy cold rolled by 75% and isothermally annealed at 773 K. It turned out that the grain boundary region of a \{111\}<uvw> grain neighbouring a \{111\}<110> grain rotated during rolling about the \{111\}//ND axis into the \{111\}<110> orientation, so that both stress and strain compatibilities might be satisfied simultaneously. Contrary to the modified Taylor theory proposed recently by some authors, the compatibility strains were accommodated not homogeneously by the entire grain but within a limited distance from the grain boundary. From the highly strained \{111\}<110> region thus created in the grain boundary region of the \{111\}<uvw> grain, nucleation of the \{111\}<110> recrystallized grain occurred preferentially. In the case of a \{111\}<110> grain neighbouring a \{100\}<011> grain, the grain boundary region rotated during rolling about the \{111\}//ND axis toward the \{111\}<112> orientation. From this region, \{111\} recrystallized grains considerably remote from the \{111\}<110> orientation were found to be preferentially formed.

(Received September 24, 1986)

Keywords: grain boundary, polycrystalline iron, recrystallization, electron microscopy, \{111\} texture

I. Introduction

It has long been known that, in the recrystallization of cold rolled polycrystalline iron, nucleation of a \{111\} recrystallized grain takes place preferentially in the grain boundary regions\(^{(1)-(4)}\). This has been attributed to the heavy local deformation introduced into these regions during rolling. Since both the development of the inhomogeneous strain and orientation changes induced in these regions are strongly dependent on the orientations of two grains neighbouring each other at the boundary, nucleation of a \{111\} recrystallized grain is expected to occur only at some grain boundaries. However, such a possibility has not been clarified as yet.

Deformation and recrystallization in the grain boundary region have already been studied in detail by using bi-crystal specimens or polycrystalline specimens in which coarse grains penetrate the whole plate thickness\(^{(4)-(9)}\). However, since the constraints of the neighbouring grain in these cases are only two-dimensional, conclusions obtained from these investigations cannot be applied directly to fine grained specimens, in which constraints of the neighbouring grain is not only three-dimensional but also much stronger. In fact, it has been found recently that the development of the \{111\} recrystallization texture is strongly dependent on the initial grain sizes\(^{(4)}\). A remarkable development of this texture has been observed, only if the initial grain size is smaller than 65 μm. This result suggests that the formation of a \{111\} recrystallized grain cannot be clarified without choosing a fine grained specimen as the starting material.

The orientation change introduced near the grain boundary region has been studied most commonly by the etch pit technique\(^{(1)}\)(\(^{(2)}\))\(^{(4)}\)(\(^{(10)}\)). In all cases, however, no precise determination of the orientation has been made, and only approximate orientations have been inferred from the appearance of etch pits. For the precise determination of the orientation, it is generally desirable to develop etch pits larger than 10 μm. This, in turn, implies that the

* This paper was originally published in Japanese in J. Japan Inst. Metals, 50 (1986), 250.
** Technical Research Center, Nippon Kokan K.K., Minamiwatarida-chou 1-1, Kawasaki-ku, Kawasaki 210, Japan.
orientation changes having occurred within the distance of 10 µm cannot be resolved by this method. In the grain boundary region, an appreciable orientation change can occur within a much shorter distance. It is also evident that this method is not effective for a fine grained specimen. As another disadvantage of this method, it should be pointed out that, according to this method, the orientation changes cannot be directly correlated with the internal dislocation structure. These problems can be completely eliminated by using transmission electron microscopy and selected area electron diffraction.

Taking these problems in the previous investigations into account, an attempt was made in the present investigation to clarify the mechanism which was responsible for the formation of the {111} recrystallization nuclei at grain boundaries. Using transmission electron microscopy and selected area electron diffraction, inhomogeneous deformation introduced into the grain boundary regions was studied on a specimen having the initial grain size of 40 µm. Local orientation changes induced in these regions were determined for various combinations of neighbouring orientations at the grain boundary. They were further correlated with the orientations of the recrystallized grains formed in these regions after annealing. From these results, it was tried to find out the grain boundary which provided recrystallization nuclei of the {111} orientations.

II. Experimental Procedure

The starting material was 150-kg ingot of a high purity Fe-0.02% C-0.15% Mn alloy melted in a vacuum furnace. Contents of impurities were N=0.0015%, S=0.001%, P=0.002% and O=0.0087%. This ingot was hot rolled to a thickness of 30 mm. After reheating at 1523 K for 1 h, it was further hot rolled to a thickness of 3 mm. The grain size obtained after the hot rolling was 40 µm. After pickling, the hot band was cold rolled to a 75% reduction in thickness. Coupons having the dimensions of 15 mm × 20 mm × 0.75 mm were cut from the cold rolled sheet and isothermally annealed in a salt bath held at 773 K. From these specimens, thin foils were prepared with the standard chemical and electrolytic polishing technique. The microstructures developed by cold rolling and annealing in the grain boundary region were observed by an electron microscope (JSM-200CX) operated at 200 kV. The orientations were determined by means of selected area electron diffraction. The orientations are expressed as (HKL)[UVW], where (HKL) and [UVW] represent the rolling plane and the rolling direction, respectively. However, the [UVW] became often such a high order of indices that they were too complex to be readily understood. For this reason, the orientations were expressed in the form (HKL)[RST]+α, where [RST] represents a simple low index direction nearest to the [UVW] direction and lying in the (HKL) plane. The α represents an angle of rotation about the [HKL] axis, which is needed to bring the (HKL)[RST] orientation into the (HKL)[UVW] orientation. The sign of α was defined as being positive for the clockwise rotation.

III. Results

From 97 grain boundaries studied, it was found that {111} recrystallized grains were formed only on some restricted boundaries, which were always associated with a {111} <uvw> deformed grain. On some of typical examples, the inhomogeneous deformation and recrystallization behaviour observed in a region near these grain boundaries will be described below.

1. Grain boundary between (111)[110] and (111)<uvw> deformed grains

Figure 1 represents the local inhomogeneous deformation observed in the grain boundary region between (111)[uvw] and (111)[110] deformed grains after 75% rolling reduction. While the orientation of the right-hand side grain (Grain 1) was uniform showing the (111) [110] orientation, the orientation of the left-hand side grain (Grain 2) varied considerably. Although the (111) plane was kept always parallel to the rolling plane, the angle α between the rolling direction and the [110] direction changed continuously as the grain bound-
Nucleation of a \{111\} Recrystallized Grain at the Grain Boundary of Cold Rolled Polycrystalline Iron

ary (G.B.) was approached. The values of \( \alpha \) were \( 9^\circ, 7^\circ \) and \( 2^\circ \) at the distances of 9, 5 and 2 \( \mu m \) from the grain boundary, respectively. This indicates that, as the grain boundary was approached, the \( \{111\}[uvw] \) grain rotated about the \([111]/ND\) axis, arriving finally at the \( \{111\}[1\bar{1}0] \) orientation of the neighbouring grain. Also, the dislocation density increased considerably as the grain boundary was approached. These results clearly shows that a highly strained \( \{111\}[1\bar{1}0] \) region is formed in the grain boundary region of the \( \{111\}\langle uwv \rangle \) grain. Figure 2 shows recrystallized grains formed on the grain boundary between \( \{111\\langle 011 \rangle \) and \( \langle 100\rangle[011] \) deformed grains after annealing of 540 S at 773 K. Orientations of the deformed structure determined along the line AB in Fig. 2 at 2 \( \mu m \) interval from the grain boundary are shown in Fig. 3. Grain A was stable showing always the \( \{111\\langle 110 \rangle \) orientation. Grain B, on the other hand, showed rotations about the \( \{111\}//ND \) axis as the grain boundary was approached, and at the grain boundary, it showed the same \( \{111\\langle 1\bar{1}0 \rangle \) orientation as the neighbouring deformed grain. Thus, the same result as that given in Fig. 1 has been confirmed in the case of Fig. 2. In Fig. 2, recrystallized grains formed on the grain boundary grew to the sizes of several microns. it should be noted that most of them had their \( \{111\) plane parallel to the rolling plane. Although the angle \( \alpha \) varied between \( 3^\circ \) and \( 25^\circ \), half of the recrystallized grains had orientations very near to the \( \{111\\langle 1\bar{1}0 \rangle \) orientation.

2. Grain boundary between \( \{111\\langle 01\bar{1} \rangle \) and \( \langle 100\rangle[01\bar{1}] \) deformed grain

Figure 4 shows the microstructures and orientation changes observed along the grain boundary.
boundary between these two grains. Diffraction spots obtained from the grain boundary region of the (111)[01\perp] grain showed often considerable arcs, although their centers were always located approximately at the (111)[01\perp] orientation. This indicates that, in order to accommodate the compatibilities at the grain boundary, considerable clockwise and counter-clockwise rotations about the [1111//ND axis occurred within a short distance. On the other hand, (100)[01\perp] grain was unstable in the grain boundary region and showed a maximum rotation of 15° about the [100]//ND axis. Corresponding to this orientation change, the dislocation densities in these regions were considerably high. Figure 5 shows the recrystallized grains formed along the grain boundary between the (001)[1\overline{1}0] and (111) [1\overline{1}0] deformed grains. Orientations measured along the line CD at a 2 \mu m interval from the grain boundary are shown in Fig. 6. Although the (111)[1\overline{1}0] grain was considerably stable, diffraction patterns obtained from the grain boundary region of this grain showed considerable arcs. Although the (001)[1\overline{1}0] grain was stable within the grain, it was unstable in the boundary regions. In the regions within 4 \mu m from the grain boundary, this grain was fragmented into regions of several different orientations. Orientations of the recrystallized grains formed along the grain boundary is illustrated in Figs. 7 and 8. In both cases, they were not random and most of the recrystallized grains had either (111) or (100) plane parallel to the rolling plane. In the case of Fig. 7, the angle \alpha of the (111) recrystallized grains measured from the (111)[1\overline{1}0] orientation showed a wide spread of 49°. In the case of Fig. 8, the recrystallized grains having the near-(001) [1\overline{1}0] orientation (Grains A and B) were nucleated on the grain boundary. On the side of the (111)[1\overline{1}0] deformed grain, the (111) [1\overline{1}0] recrystallized grain was observed only on the grain boundary (Grain D). With increasing

Fig. 2 Preferential nucleation of {111} recrystallized grains which occurred along the boundary (G.B.) between (111)[1\overline{1}0] and (111)[uvw] deformed matrices. Observations were made on the specimen annealed 540 s at 773 K after 75% cold rolling.
distance from the grain boundary, the orientation of the recrystallized grains tended to deviate from (111)[110], and the size of the recrystallized grain became larger. From the above results, it may be concluded that, from the grain boundary region of the (111)[110] grain neighbouring the (100)[011] grain, (111) recrystallized grains considerably remote from the (111)[110] orientation are preferentially formed.

3. Triple point of grain boundaries

Figure 9 shows the microstructure observed at the triple point of the grain boundaries at which (111)[011], (100)[011] and (311)[011] deformed grains meet. Selected area electron diffraction patterns determined at the positions indicated by the numbers 1 to 13 in Fig. 9 are shown in Fig. 10. Although the (100)[011] grain was stable in its interior, it was unstable in the boundary region neighbouring the (111) [011] grain. In this region, it rotated by about 10° about the [100]//ND axis. In the neighbourhood of the triple point, it rotated into the (137)[310]+25° orientation.

In comparison to this, the (311)[011] grain was much more stable. In the regions neighbouring the (111)[011] grain, no rotation was found, whereas at the triple point it rotated into the (110)[011]+8° orientation.

Compared with these orientations, the (111) [011] orientation was much more stable and the dislocation density was highest. At the boundary with the (100)[011] grain, however, it rotated by 6° about the [111]//ND axis. At the triple point, it showed a large arc in its diffraction patterns. In all cases, however, the (111) plane was kept always parallel to the rolling plane.

IV. Discussion

1. Rotation about the [111]//ND axis

In the previous paper(3), it has already been inferred from pole figure measurements that the grain boundary region of a {111}<uvw> grain rotates about the [111]//ND axis during rolling. This rotation was observed most prominently in a specimen having the {111}<112>
Fig. 4  Inhomogeneous deformation and local orientation changes which are introduced by 75% cold rolling into the boundary region between (100)[011] and (111)[011] grains.
initial texture. As a result, a strong [111] // ND fiber rolling texture was developed. Since a pronounced [111]<110> recrystallization texture was obtained after annealing these specimens, it was concluded that the [111]<110> recrystallization nuclei originated in the [111] // ND fiber rolling texture. These observations further suggested that the rotation about the [111] // ND axis occurred as a result of the interaction between grains having [111]<uvw> type orientations. It is expected that, if two grains having [111]<uvw> type orientations are neighbouring each other at the grain boundary, they rotate readily into their common stable end orientation, [111]<110>, since both stress and strain compatibilities are satisfied simultaneously. Among the [111]<uvw> type orientations, the strongest interaction would occur between the [111]<110> and [111]<112> orientations, because the orientation difference is largest. In this combination, the [111]<110> orientation is much more stable than [111]<112>, so that the grain boundary region of the [111]<112> grain, which aims at the [111]<110> orientation, suffers from the severest strain. Through this mechanism, a highly strained region having the [111]<110> orientation would be formed in the grain boundary region of the [111]<112> grain. These highly strained [111]<110> regions would provide the [111]<110> recrystallization nuclei during annealing.

The example discussed above represents an extreme case. If one assumes more generally the combination of [111]<uvw> type orientations, the orientation of the highly strained region might be near to [111]<110>, but not strictly agree with this, or the width of the [111]<110> region formed might be rather limited. This is especially true in the present case in which the rolling reduction adopted was only 75%. The possibility of the rotation
about the [111]//ND axis, which was thus proposed in the previous paper\(^3\), was confirmed in the present investigation by using transmission electron microscopy. To show this rotation about the [111]//ND axis more quantitatively, the results obtained in this investigation are summarized in Fig. 11. In this figure, the angle of the rotation about the [111] axis measured from the (111)[110] orientation is plotted against the distance from the grain boundary. It is evident that the (111)[110] grain was stable everywhere. On the other hand, the (111)[uvw] grain showed its rotation about the [111]//ND axis. The angle of rotation varied linearly with the distance from the grain boundary, and at the boundary of this grain, a 2 µm wide (111)[110] region was formed. Similar results have been reported in the case of Cu subjected to 50% cold rolling\(^{11}\).

Although the orientations of the two neighbouring grains have not been reported, it was found that the difference in orientation between these grains decreased linearly, as the grain boundary was approached. The orientation difference, which amounted to 5° at a position of 5 µm from the grain boundary, decreased to zero at the boundary. The amount of this orientation change coincides quantitatively well with the results illustrated in Fig. 11. From the above results, it might be concluded that the compatibilities at the grain boundary are important factors controlling the crystal rotation in the grain boundary region. Furthermore, these results suggest strongly that compatibility conditions are only gradually accommodated, as the grain boundary was approached. This possibility is also supported by the fact that the dislocation density increased gradually, as the grain boundary was approached. The deformation behaviour is markedly different from that assumed in the modified Taylor theory recently proposed by Öztürk and Davis\(^{12}\) and Raphnel and Van Houtte\(^{13}\).

As long as the homogeneous deformation is concerned the rotation about the [111]//ND axis is difficult to be explained. Recently, Abe, Kokabu, Hayashi and Hayami\(^{14}\) have proposed that the local multiple slip induced in the grain boundary region is responsible for the

---

Fig. 6 Selected area electron diffraction patterns obtained at 2 µm interval along the line CD in Fig. 5.
Fig. 7 Orientations of recrystallized grains shown in Fig. 5.

Fig. 8 Orientations of recrystallized grains shown in Fig. 5.
formation of the \{111\} recrystallized grain. They have demonstrated the condition which leads to this local deformation. However, the stress system adopted by them is not a tri- or bi-axial one that should be assumed in the rolling deformation, but a uniaxial one. Furthermore, although they have assumed that single slip is the deformation mode in the grain interior, it would be more reasonable to assume that the multiple slip is operative. This is especially true for the \{111\}<110>, \{111\}<112> and \{100\}<011> orientations, which have higher symmetry. From the discussion described above, it might be more appropriate to

Fig. 9  Inhomogeneous deformation observed at the regions near the triple point of the grain boundaries after 75% cold rolling.
conclude that the driving force for the rotation about the [111]//ND axis is the compatibility conditions which should be satisfied only in the grain boundary region.

In contrast to the case described above, the rotation about the [111]//ND axis observed in the grain boundary region of the \{111\}\langle110\rangle grain neighbouring the \{100\}\langle011\rangle grain was quite different in nature. Although the mean orientation was \{111\}\langle110\rangle, both clockwise and counter-clockwise rotations about the [111]//ND axis were found to occur con-
siderably within a very small region. Since the \( \{111\} \langle 110 \rangle \) orientation was quite stable, compatible strain in the grain boundary region of this grain should be accommodated in this manner.

2. Orientation of the recrystallization nuclei formed in the grain boundary region

The results described above suggest strongly that the orientation of the recrystallized grain formed in the grain boundary region is strongly related with the orientation of the deformed structure in which it was nucleated. This leads to a non-random nucleation shown in Figs. 2, 7 and 8.

In the case of the \( \{111\}[uvw] \) deformed grain neighbouring the \( \{111\}[110] \) deformed grain, it was found that most of the recrystallized grain formed along the grain boundary had (111) planes parallel to the rolling plane. Half of them had orientations which were very near to \( \{111\}[110] \) orientations of the deformed structure lying along the grain boundary. Some of the recrystallized grains showed, however, considerable deviations from the \( \{111\}[110] \) orientation, although their (111) planes were parallel to the rolling plane. Such deviation might be attributed to various local curvatures existing along the grain boundary. Also the grain boundary orientation might influence the orientations of the recrystallized grain. These factors which have not been taken into account in the present investigation accounts for the fact that nucleation of the recrystallized grain does not occur homogeneously along the grain boundary\(^{(14)}\).

In contrast to the case described above, \( \{111\} \langle 110 \rangle \) recrystallized grains were very difficult to nucleate from the grain boundary between \( \{111\} \langle 110 \rangle \) and \( \{100\} \langle 011 \rangle \) deformed grains. Instead, \( \{111\} \) recrystallized grains, whose orientations deviating significantly from the \( \{111\} \langle 110 \rangle \) orientation were nucleated in the grain boundary region of the \( \{111\} \langle 110 \rangle \) grain. As it has been already pointed out in the above, the presence of arcs in the diffraction patterns suggests strongly that in this region the rotation about the \( [111]/ND \) axis toward the \( \{111\} \langle 112 \rangle \) orientation has taken place. From the regions which have suffered from the largest rotation, \( \{111\} \) recrystallized grains deviating significantly from the \( \{111\} \langle 110 \rangle \) orientation may have

![Fig. 11](image-url) Rotation about \([111]/ND\) axis which was observed in the boundary region between \(\{111\}\) \([110]\) and \(\{111\}[uvw]\) deformed matrices. Angle of rotation measured from \(\{111\}[110]\) is indicated as a function of the distance from grain boundary.
been nucleated.

V. Conclusion

(1) Using transmission electron microscopy and selected area electron diffraction, it was confirmed that during rolling the grain boundary region of a \{111\}<uvw> grain neighbouring a \{111\}<110> grain rotates about the [111]/ND axis, forming a highly strained \{111\}<110> region. It was found that the \{111\}<110> recrystallization nuclei are preferentially formed in this region.

(2) The driving force for the rotation about the [111]/ND axis is the stress and strain compatibilities which should be satisfied in the grain boundary region. In contrast to various recent proposals which have assumed Taylor-type homogeneous deformation, these compatibility conditions are satisfied only within a restricted distance from the grain boundary.

(3) \{111\} recrystallization nuclei considerably deviating from the \{111\}<110> orientation are formed in the grain boundary region of the \{111\}<110> deformed grain.

REFERENCES

(1) M. Matsuo, S. Hayami and S. Nagashima: Adv. in X-ray Analysis, Plenum Press, 14 (1971), 214.
(2) M. Matsuo, S. Hayami and S. Nagashima: Proc. Int. Conf. on the Science and Technology of Iron and Steel II, Suppl. to Trans. ISIJ, 11 (1971), 867.
(3) H. Inagaki: Trans. ISIJ, 24 (1984), 266.
(4) M. Abe, K. Kokabu, M. Hayashi and S. Hayami: Trans. JIM, 23 (1983), 718.
(5) R. E. Hooke and J. P. Hirth: Acta Met., 15 (1957), 535.
(6) R. G. Aspden: Trans. Met. Soc. AIME, 2 (1959), 986.
(7) G. L. Ferran, R. D. Doherty and R. W. Cahn: Acta Met., 19 (1971), 1019.
(8) H. W. F. Heller, C. A. Verbraak and B. H. Koster: Acta Met., 32 (1984), 1395.
(9) A. Skalli, R. Fortunier, R. Fillit and J. H. Driver: Acta Met., 33 (1985), 997.
(10) H. Inagaki and T. Suda: Texture 1 (1972), 129.
(11) A. R. Jones: Grain Boundary Structure and Kinetics, ASM, Ohio, (1980), p. 739.
(12) T. Öztürk and G. J. Davis: Metal Science, 18 (1984), 531.
(13) J. L. Raphael and P. Van Houtte: Acta Met., 33 (1985), 1481.
(14) P. Faivre and R. D. Doherty: J. Mat. Sci., 14 (1979), 897.