Microstructure and texture through thickness of ultralow carbon IF steel sheet severely deformed by accumulative roll-bonding

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Abstract

Ultralow carbon interstitial free (IF) steel was severely deformed up to a strain of 5.6 by the Accumulative Roll-bonding (ARB) process at 773 K. Crystallographic analysis by electron back-scattering diffraction (EBSD) technique in a field-emission type scanning electron microscope (FE-SEM) was carried out for the ARB processed IF steel throughout thickness of the sheet. Microstructural parameters, such as grain size, grain boundary misorientation and crystal orientation, through thickness of the ARB processed specimen were quantitatively clarified by the EBSD analysis. The ARB processed material was homogeneously filled with the lamellar or pancake-shaped ultrafine grains whose mean grain thickness were about 200–300 nm. More than 80% of the boundaries surrounding the ultrafine grains were high-angle grain boundaries. The ARB processed sheet had unique and complex textural distribution through thickness. The region near the thickness center has the conventional but quite weak rolling texture mainly composed of (110)\textit{\textit{h}}//RD and (111)\textit{\textit{h}}//ND. On the other hand, the surface region had the sharp shear texture, ND//k\textit{\textit{110}}l. Such a textural distribution is due to the redundant shear strain induced by high-friction between the sheet and roll during rolling. The correspondence between the textural and microstructural distribution and the shear strain distribution throughout thickness of the sheet was discussed.

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1. Introduction

Severe plastic deformation (SPD) \cite{1–8} is one of the most effective ways to produce ultrafine grained metallic materials with the mean grain size smaller than 1 \textmu m. The ultrafine grained materials are expected to improve various properties, such as strength, toughness and corrosion resistance, etc. Saito et al. \cite{9} have invented a SPD process using rolling, named accumulative roll-bonding (ARB), which has been the only SPD process applicable for large bulky sheet materials. In the previous studies, it has been clarified that the ARB process makes it possible to obtain the ultrafine grained structures in various kinds of structural metallic materials, such as aluminium alloys \cite{10–13}, copper alloy \cite{14}, ferritic steels \cite{15–17} and austenitic steels \cite{18,19}. In addition, the crystallographic analysis by Kikuchi-line technique in transmission electron microscopy (TEM) has proved that most boundaries surrounding the ultrafine grains in the ARB processed materials are high-angle boundaries \cite{12,16,17}. On the other hand, studies on texture of the ARB processed materials have been limited \cite{10,20–23}. Because the ARB has been usually carried out under the rolling condition without lubrication, a large amount of redundant shear strain is introduced in the subsurface regions of the sheets \cite{20}. The distribution of such shear strain must be complicated through thickness of the sheets, since cutting, stacking and roll-bonding are repeated in the ARB \cite{25}. Thus, when investigating the texture of the ARB processed materials, textural distribution through thickness of the sheet should be taken into account. In addition, the complicated change through thickness is expected not only for the texture but also for the microstructure. Therefore, quite careful measurements in large areas through thickness are needed for crystallographic and microstructural analysis of the ARB.
processed sheets. Recently, Tsuji et al. [21] have reported that electron back-scattering diffraction (EBSD) technique [26] in a field-emission type scanning electron microscope (FE-SEM) is a powerful method to crystallographically characterize the ultrafine grained microstructures fabricated by the SPD process. The EBSD technique makes it possible to get the crystallographic information including various kinds of microstructural parameters, such as grain size, boundary misorientation, microscopic texture, etc., in large areas. The purpose of this study is to clarify both the microstructural and textural distribution through thickness of the ultralow carbon interstitial free (IF) steel severely deformed by the ARB process by the use of the FE-SEM/EBSD technique. The change in the texture and microstructure through thickness will be discussed in consideration of the redundant shear strain distribution.

2. Experimental

Ti-added ultralow carbon IF steel sheet was used in this study. The chemical composition of the material is shown in Table 1. The material showed the fully-recrystallized microstructure filled with the equiaxed ferrite grains having the mean grain size of 20 μm. The starting sheet for the ARB process was 1 mm thick, 30 mm wide and 300 mm long. After degreasing and wire-brushing the surface, two pieces of the sheet were stacked to be 2 mm thick and spot-welded at the front and back ends. The stacked sheets were held for 600 s in an electric furnace set at 773 K under an air atmosphere and immediately roll-bonded by 50% reduction in thickness (equivalent strain; ε = 0.8) in one pass. A two-high mill having the rolls 310 mm in diameter was used for the roll-bonding. The roll-bonding was carried out without lubrication at a roll speed of 17.5 m min⁻¹ (equivalent strain rate of 19 s⁻¹). The roll-bonded sheet was immediately water-cooled. The rolled sheet was cut to be nearly initial dimensions again and subjected to the next ARB cycle. This procedure was repeated up to 7 cycles (ε = 5.6).

Thin foils parallel to the longitudinal section of the ARB processed sheet were prepared by twin-jet electropolishing for TEM observation and the FE-SEM/EBSD measurement. The electropolishing was carried out in a 100 ml HClO₄ + 900 ml CH₃COOH solution at 285 K at a voltage of 17.5 m/min⁻¹ (equivalent strain rate of 19 s⁻¹). The rolled sheet was cut to be nearly initial dimensions again and subjected to the next ARB cycle. This procedure was repeated up to 7 cycles (ε = 5.6).

3. Results

3.1. TEM observation

Fig. 1 shows the TEM microstructure and corresponding selected area diffraction (SAD) pattern of the specimen ARB processed by 7 cycles (ε = 5.6) at 773 K. The microstructure was observed from the transverse direction (TD) near the thickness center of the specimen. The ultrafine grains elongated along the rolling direction (RD) were homogeneously observed through the large area in the thin foil. The mean grain thickness and length, dₜ and dₙ, of the ultrafine grains were 210 and 930 nm, respectively. This microstructure is a typical one observed in the materials severely deformed by the ARB process [12,13,16–19]. In the previous study [12,13], it has been known that the grain shapes in the severely ARB processed materials are lamellar or elongated ones when observed from TD and relatively equiaxed ones when observed from the normal direction (ND) of the sheets. That is, three-dimensional morphology

| C   | N   | Si  | Mn  | P   | Cu  | Ni  | Ti  | Fe  |
|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| 0.002 | 0.003 | 0.01 | 0.17 | 0.012 | 0.01 | 0.02 | 0.072 | bal. |

Fig. 1. TEM microstructure and corresponding SAD pattern of the IF steel ARB processed by 7 cycles (ε = 5.6) at 773 K. Observed from TD.
of the ultrafine grains is an elongated pancake. In the present case, the severely deformed material was homogenously filled with the pancake-shaped ultrafine grains. The SAD pattern obtained by the use of an aperture 1.6 μm in diameter is not spotty but ring-like, indicating that boundaries between the adjacent grains have large misorientation.

3.2. EBSD measurement

The microstructural characteristics, as shown in Fig. 1, of the material severely deformed by the ARB process are similar to those reported in the previous studies. However, the microstructural distribution through thickness of the ARB materials has not been clarified yet, since the TEM observation is applicable only for limited areas. As will be explained in details in the later section, it is expected that the ARB processed materials have a complicated change in texture and microstructure through thickness due to the redundant shear strain introduced during roll-bonding. In order to clarify the microstructural and textural distribution through thickness of the ARB sheet, the EBSD measurement was carried out throughout the thickness of the IF steel sheet in the present study. Fig. 2 shows the SEM micrograph from the surface to the center in the 7-cycle ARB processed IF steel. The regions represented by 10 × 30 μm white rectangles were subjected to the EBSD measurement in FE-SEM. The 19 areas were totally measured to cover the whole thickness from surface to center. The rectangle areas shown in the figure were selected in order to prevent the ARB interfaces severely etched by electro-polishing. The EBSD measurement was carried out at a step size of 50 nm.

The EBSD measurement, grain boundary misorientation maps (GB maps, hereafter), ND orientation maps (ND maps) and RD orientation maps (RD maps) were constructed. Figs. 3 and 4, for example, show the GB maps (a), ND maps (b), and RD maps (c) obtained from the subsurface and the thickness center of the ARB sheet, corresponding the top and the bottom rectangle areas in Fig. 2, respectively. When constructing these orientation imaging maps, the measured points whose confidence index (CI) values were less than 0.1 were removed and painted black in order to raise the reliability of the analysis. In the GB maps, the green lines indicate the high-angle grain boundaries whose misorientations were larger than 15°, while the red lines indicate the low-angle boundaries whose misorientations were between 2° and 15°. The boundaries less than 2° of misorientation angle were cut off in consideration of the errors in the EBSD measurement. The colors in the ND maps and the RD maps represent the crystallographic orientations parallel to ND and RD, respectively. The correspondence between the color and the crystallographic orientation is indicated in the stereographic triangle. In Figs. 3 and 4, the elongated ultrafine grains were clearly reconstructed. Both the thickness center and the surface region were homogeneously filled with the elongated ultrafine grains, and almost all the boundaries are high-angle boundaries drawn by green lines, as shown in Figs. 3a and 4a. However, in the surface region the grain thickness is slightly larger and the grain length is smaller than those in the thickness center. That means the ultrafine grains in the surface region are somewhat equiaxed compared with those in the center region. The difference in texture between the center and surface is clear from the color maps (Figs. 3a,b and 4a,b). In the thickness center, the texture seems relatively random because various colors are observed in Fig. 3b and c. In the surface region, in contrast, the ND map had a great number of green-colored grains, and the RD map was mostly colored either blue or red. This means that the surface region mainly has the \( \{100\}//ND \) texture including \( \{110\}\langle001\rangle \) orientations, which is known as shear texture in rolled bcc metals [28]. Figs. 5 and 6 show the \( \{001\} \) and \( \{011\} \) pole figures constructed from the EBSD data shown in Figs. 3 and 4, respectively. As shown in Fig. 5, the center region of the sheet has the weak \( \{111\}\langle ND \rangle \) and \( \{110\}\langle RD \rangle \) orientations which are typical rolling textures of ferritic

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1 The CI value is the parameter in the OIM analysis that indicates the reliability of the measured point. In general, the points having the CI value larger than 0.1 are considered to be measured accurately [27].

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![Fig. 2. SEM micrograph of the IF steel ARB processed by 7cycles (e = 5.6) at 773 K. The regions represented by white rectangles were subjected to the EBSD measurement in FE-SEM.](image-url)
However, the intensity of the texture is weak, and it can be concluded that the center region does not have a strong texture. This is somewhat surprising because this specimen was severely deformed up to a total strain of 5.6, which corresponds to 99.2% rolling reduction. Such a weak texture coincides well with the random color distribution in Fig. 3. On the other hand, the surface region of the sheet has the relatively sharp (110)//ND texture around {110}<001>, so-called Goss orientation. The pole figures shown in Fig. 6 are typical ones formed in the region undergoing severe shear deformation in rolled bcc metals [28].

3.3. Distribution of microstructural parameters through thickness

The EBSD measurements were carried out in every area represented in Fig. 2, and the same analysis as Figs. 3–6 was done for all the measured areas. In order to clarify the microstructural distribution through thickness of the ARB processed sheet, the microstructural parameters, i.e. the mean grain size (mean grain thickness and length), the mean misorientation and the fraction of high-angle grain boundaries, analyzed from the EBSD data were plotted as a function of the thickness location in Figs. 7 and 8.
Here, the normalized position of the sheet, $t/t_0$, where $t_0$ is the total thickness of the sheet and $t$ is the distance from the thickness center, was used to represent the thickness location. Therefore, $t/t_0 = 0$ and $t/t_0 = 0.5$ correspond to the thickness center and the surface of the sheet, respectively. The broken lines in the figure indicate the positions of the roll-bonded interfaces formed in the 4th, 5th and 6th cycle of the ARB. The positions of the interfaces were determined from Fig. 2. Fig. 7 shows the mean grain size distribution through thickness of the sheet. Here, mean grain thickness and length were measured from the GB maps, like Figs. 3a and 4a, by the use of two types of definition. One is the mean grain thickness and length, $d_{\text{ALL}}^t$ and $d_{\text{ALL}}^l$, determined from the whole grain boundaries including both high-angle and low-angle boundaries. The other is those taking account of only high-angle boundaries, $d_{\text{HAGB}}^t$ and $d_{\text{HAGB}}^l$. The ARB processed specimen has a relatively homogeneous distribution in both mean grain thickness and length through thickness of the sheet. However, strictly speaking, mean grain thickness near the surface region is slightly larger than that near the center, and the length near the surface is smaller than that near the center region. Such a slight change in grain size distribution was qualitatively mentioned in Figs. 3 and 4. There is no
great difference between the $d_{\text{ALL}}^t$ determined from the whole grain boundaries and the $d_{\text{HAGB}}^t$ determined only from the high-angle boundaries throughout the thickness of the sheet. This means that almost all the boundaries parallel to RD was high-angle boundaries. On the other hand, the $d_{\text{HAGB}}^l$ is somewhat larger than the $d_{\text{ALL}}^l$, which indicates that the boundaries parallel to ND have a larger fraction of low-angle boundaries than those parallel to RD. The $d_{\text{ALL}}^t$ and $d_{\text{ALL}}^l$ near the center region were 230 and 1360 nm, respectively. By the way, the mean grain sizes near the center region of the sheet, which were determined from the TEM microstructure (Fig. 1) are $d_l = 210$ nm and $d_l = 930$ nm. The $d_{\text{ALL}}^t$ and $d_{\text{ALL}}^l$ near the center are slightly larger than those measured from the TEM. This is reasonable because the boundaries with misorientations smaller than 2 degrees were cut in the EBSD analysis, which can be observed in TEM. Anyway, roughly speaking, the ultrafine grain size is uniform throughout the thickness of the IF steel sheet ARB processed by 7 cycles at 773 K.

Fig. 8 shows the distribution of the fraction of high-angle grain boundaries and the misorientation of the boundaries through thickness, which were calculated from each EBSD datum. The fraction of high-angle boundaries tends to decrease gradually from the thickness center to the surface. As a result, the mean misorientation also decreases gradually from about 36° at the center to 33° at the surface. The fraction of high-angle boundaries and the mean misorientation tend to have slightly large values near some of the bonded interfaces. Anyway, the as-ARB processed material had the large fraction of high-angle boundaries from 80 to 90% throughout the thickness of the sheet. From Figs. 7 and 8, it can be concluded that the pancake-shaped ultrafine grains in the ARB processed IF steel are uniform throughout the thickness from viewpoints of both sizes and crystallographic misorientation.

### 3.4. Distribution of texture through thickness

As was shown in Figs. 3–6, the ARB processed specimen has large difference in texture between the center and surface. The difference in texture is due to the redundant
shear strain introduced during the ARB process. In general, it has been well-known that the rolled bcc metals show the (110)//RD texture and (111)//ND texture, which are called α-fiber and γ-fiber, respectively. The center region of the ARB sample showed α-fiber and γ-fiber, although they are very weak (Figs. 3 and 5). On the other hand, the shear deformed region, such as the surface region in hot-rolled sheets [28], has the sharp (110)//ND texture (Figs. 4 and 6). The shear strain distribution in the ARB processed materials is expected to be quite complicated [25]. Thus, in order to clarify the textural distribution though thickness, the area fractions of the grains belonging to these three types of the fiber texture, i.e. (110)//RD ± 10°, (111)//ND ± 10° and (110)//ND ± 10°, were analyzed from the EBSD data and plotted in Fig. 9. The positions of the roll-bonded interfaces after fourth ARB cycle are also indicated in the figure. Roughly speaking, the shear strain is expected to show the maximum values around the bonded interfaces [25].

The actual texture distribution through thickness of the sheet in Fig. 9 is quite complicated. In the thickness center region, there is no strong fiber texture. This means the texture of the center region is relatively random, as shown in Figs. 3 and 5. The α-fiber texture gradually increases from the center to surface, and shows the peak at the position of about 0.15. Then, the α-fiber decreases up to 0.25 thickness location, increases again, shows another peak, and then decreases up to the surface. The γ-fiber shows the similar profile to that of the α-fiber, although the fraction level of the γ-fiber is fairly smaller than that of the α-fiber. Finally, the fraction of the rolling textures decrease to a few percent at the subsurface region. The (110)//ND, which is a kind of shear texture, shows complicated distribution from the center up to around 0.35 thickness location. The intensity of the shear texture in these regions is not so strong, and there are weak peaks in its distribution some of which coincide with the bonded interface. However, in the subsurface region, strong shear texture can be recognized. This result agrees with Figs. 4 and 6. Consequently, it was clarified in the present study that the 7-cycle ARB processed IF steel has a unique and complex texture distribution through thickness of the sheet.

4. Discussion

4.1. Estimation of shear strain distribution through thickness

In this study, the distribution in microstructure and texture was clarified through thickness of the ARB processed IF steel. One of the most interesting results obtained is that the severely ARB processed IF steel has a characteristic texture distribution through thickness of the sheet. This is due to the redundant shear deformation caused by the high-friction rolling. In general, the ARB process has been carried out without any lubricant in order to achieve quick grain refinement [20,25]. As a result, large amount of redundant shear strain owing to large friction between the rolls and the materials is introduced in the surface regions, so that the microstructure and texture in the surface regions are different from those in the thickness center [20,21]. In addition, characteristically in the ARB process, half of the severely deformed region near the surface comes to the center by cutting and stacking in the next ARB cycle. Because this procedure is repeated again and again during the ARB process, the redundant shear strain distribution through thickness of the sheet becomes very complicated, as reported by Lee et al. [25]. It is expected, therefore, that the ARB processed material has the complicated distribution in both microstructure and texture through thickness.

Lee et al. [25] have measured the redundant shear strain distribution through thickness in the 1-cycle ARB processed commercial purity aluminium and predicted the shear strain distribution after several ARB cycles on the basis of the measurement. Fig. 10 illustrates the distribution of the shear strain through thickness after 1, 2 and 7 cycles of the ARB, assuming that the shear strain similar to that reported by Lee et al. is induced in the case of IF steel. Arrows indicate the position of the roll-bonded interfaces. After 1 cycle, the distribution through thickness shows a parabolic curve like Fig. 10(a). In the second cycle, the roll-bonded sheet is cut into two, stacked to be the initial dimension and then roll-bonded again. That is, half of the surface having large shear strain comes to the center in the next cycle. As a result, another shear strain peak appears at center of the sheet after 2 cycles (Fig. 10b). After repeating this procedure, the shear strain distribution is expected to be very complicated as shown in Fig. 10c.

4.2. Relationship between texture and shear strain

Here, the correspondence between the predicted distribution in Fig. 10c and the actual texture distribution through thickness in Fig. 9 is discussed. The subsurface region is...
expected to show the maximum shear strain, as is illustrated in Fig. 10, because the surface has been surface throughout the ARB process and the shear strain has been accumulated. Consequently, the strong shear texture in the surface, which was actually observed in the present investigation, makes sense. However, the center region of the 7-cycle ARB processed material had no strong texture. This is somewhat surprising because the center in the 7-cycle specimen had been the surface till the 6th cycle, where large shear deformation had been accumulated. This result suggests that the shear texture is easily destroyed in the subsequent rolling (plane-strain) deformation. The similar behaviors that the sharp texture developed by severely shear deformation could be easily broken down during the following rolling have been reported in the ARB processed aluminium alloys [23,24]. However, it was quantitatively shown in the present investigation that the center region from 0 to 0.1 thickness location still maintained relatively large intensity of shear texture (Fig. 9). The small peak of shear texture at the sixth bonded interface in Fig. 9 is probably a trail of this remaining shear texture. However, at the fourth and fifth interfaces, peak of shear texture was no longer observed in Fig. 9. On the other hand, the maximum peak of the rolling texture is located around 0.15 thickness location in Fig. 9. This corresponds well with the location of the lowest shear strain expected in Fig. 10c. Further, the small peaks of the α-fiber around 0.2 and 0.28 thickness locations in Fig. 9 also correspond with the positions having the minimum shear strains in Fig. 10c. It can be concluded from these considerations that the texture distribution is roughly understood from the shear strain distribution in the ARB materials though it is not simple but fairly complicated.

4.3. Effect of shear strain on microstructural evolution during ARB

Another significant information obtained from this study is the microstructural parameters such as the grain size, the boundary misorientation and the fraction of high-angle grain boundaries, through thickness of the sheet. In the previous studies, the microstructure of the ultrafine grained materials fabricated by the ARB process have been characterized mainly by the use of TEM observation. As far as the previous studies are concerned, it has been shown that the severely ARB materials are ‘homogeneously’ filled with the lamellar or pancake-shaped ultrafine grains. However, there has been some doubt whether the ARB processed materials certainly have the homogenous microstructural distribution ‘throughout’ thickness of the sheet. The microstructure in a macroscopic scale has not been clear, because the TEM observation is applicable only for limited areas in the specimen.

However, the present study clearly confirmed that the ultrafine grained structure of the ARB processed material is fairly uniform throughout the thickness, although, strictly speaking, the distribution in grain size, fraction of high-angle boundaries and mean misorientation has slight differences between the center and the surface. This is somewhat surprising because the shear strain distribution accumulated during the ARB is quite complicated as shown Fig. 10. This is probably because many (seven) cycles of the ARB were already applied to the present specimen and the effect of shear strain on the microstructural evolution was equalized in a sense.

By the way, Huang et al. [30] recently pointed out that the microstructural evolution in the ARB material is much quicker than that in the conventionally rolled material. That is, even at the same equivalent strain, the interval of the lamellar boundaries parallel to RD is smaller and the misorientation of the boundaries is larger in the ARB material than those in the conventionally rolled material. It has been clarified in the parallel study [31] that the formation mechanism of the ultrafine grained structure in the ARB process is essentially the ultrafine grain subdivi-

![Fig. 10. Schematic illustration showing the distribution of the redundant shear strain through thickness of sheet severely ARB processed by (a) 1cycle, (b) 2cycles and (c) 7cycles.](image-url)
sion, that is, dividing initial grains to submicrometer-sized regions by severe plastic deformation. The microstructural evolution by grain subdivision must be affected by many factors: strain, strain rate, strain path, deformation temperature and metallurgical factors within the materials.

One of the possible reasons for the quick grain subdivision in the ARB is the redundant shear strain considered in the present study. The substantial plastic strain in the ARB material is much larger than that evaluated only from the reduction in thickness by rolling, because the shear strain shown in Fig. 10 must be taken into account. The unique point in the ARB process is that the shear strain distributes complicatedly through thickness (Fig. 10). The local strain rate as well as the resulted temperature increase would be also different locally depending on the thickness location reflecting the shear strain distribution. These factors might also affect the microstructural evolution. Furthermore, the textural effect can greatly affect the microstructural evolution during the ARB. As was clearly shown, the history of texture development is quite complex depending on the thickness location. This means that the strain path complicatedly differs depending on the positions and the ARB cycle. The transition of texture obviously indicates that the difference in the strain path activates the different slip patterns (combination and/or amount of the operated slip systems) complicatedly depending on both the thickness location and the number of cycles, which is probable to accelerate the microstructural evolution by grain subdivision. It was confirmed, anyway, that concerning the microstructural parameters except for texture the difference due to the shear strain distribution was equalized and the fairly homogeneous structure formed after many (seven) cycles of the ARB. In order to clarify the mechanism of microstructural evolution during the ARB process discussed above, it is necessary to investigate the low-cycle ARB processed, that is, relatively low-strained specimens taking the shear strain distribution into consideration.

The distribution in microstructural and textural parameters through thickness of the ARB processed IF steel sheet having ultrafine grained structure was clarified in any case. These quantitative parameters should be used in the future study to correlate the unique mechanical properties of the ultrafine grained materials [12,13] with their microstructures.

5. Conclusions

Ultralow carbon IF steel was deformed up to a strain of 5.6 by the ARB process at 773 K. The distribution of the microstructure and the texture of the severely ARB processed material was quantitatively clarified by the FE-SEM/EBSD analysis. The main results obtained are shown below.

(i) The ultralow carbon IF steel severely strained by the ARB was filled with the pancake-shaped ultrafine grains with the mean grain thickness of 200–300 nm. More than 80% of the boundaries surrounding the ultrafine grains were high-angle boundaries. The grain size, the fraction of high angle grain boundaries and the mean misorientation were quite uniform throughout the thickness of the ARB sheet.

(ii) The severely ARB processed material had a unique and complicated distribution of texture through thickness. The texture distribution was roughly understood from the shear strain distribution in the ARB materials. At the surface where shear strain has been accumulated, strong shear texture develops. The shear texture, however, can be easily destroyed in subsequent plain-strain deformation. On the other hand, rolling texture develops at the locations where the shear strain is expected to show minimum values.

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