

# Grain Boundary Plane Distributions in a Hot Rolled 5A06 Aluminum Alloy\*\*

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To investigate the preferential distribution of grain boundary planes in a hot rolled aluminum alloy, the electron backscattered diffraction approach and five-parameter analysis method are used to characterize the grain boundary plane distributions in a hot rolled 5A06 aluminum alloy, and the results show that the orientations of grain boundary planes are textured. Grain boundary planes favor the {111} orientation at the center of the specimen, and their population is 56% higher than that in a random distribution, whereas grain boundary planes favor the {110} orientation on the surface of the specimen, and their population is 69% higher than that in a random distribution. Moreover, this anisotropic distribution of grain boundary planes occurs at all misorientation angles. The study suggests a new understanding of the pathway to evaluate the crystallographic orientation dependent performances.

## 1. Introduction

As a common thermomechanical practice for metallic materials, hot rolling aims for both realizing the desired products shape and improving the properties through the

modification of metallurgical structures. In hot rolling process, the microstructure of the material changes significantly as a result of the coupled effects of heat and deformation, which strongly affects the final properties of the hot rolled product. The grain boundary plane distribution (GBPD), which quantitatively describes the type and frequency of grain boundary planes, is a major index of the microstructure and is therefore significantly concerned.<sup>[1]</sup> To comprehensively describe the GBPD within a polycrystalline material, the lattice misorientation across a boundary plane can be routinely characterized via electron back-scattered diffraction (EBSD) technique; however, two extra spherical angles are needed to describe the orientation of that boundary plane.<sup>[2]</sup> Recently, the stereological approach named “five-parameter analysis (FPA)” has been developed to determine these spherical angles,<sup>[2]</sup> and the validity of the FPA method has been widely reported in a variety of materials.<sup>[3]</sup>

Al-Mg alloys have excellent environmental and intergranular corrosion (IGC) resistance and are, therefore, ideal for marine applications. Although hot rolling treatment could enhance certain desirable performances of Al-based alloys,<sup>[4]</sup> the GBPDs in the hot rolled structure and the possible connection to properties are yet unknown, and a number of unresolved issues remain. First, is there any texture of grain boundary plane orientations in the hot rolled microstructure? Second, to what extent can the anisotropic distribution of grain boundary planes be achieved after a typical hot rolling procedure? Third, in what way could the spatial distribution of grain boundary planes affect the material performance? Therefore, the major objective of present work is to address

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these questions, and in particular, the FPA method is used to measure the GBPDs that arise during the hot rolling on a selected Al–Mg alloy.

## 2. Method

The Al–Mg alloy specimen (AA-5A06) was prepared by a semi continuous casting method at BJUT, and had nominal components of 5.7 wt% Mg, 0.70 wt% Mn, 0.11 wt% Zr, with additional 0.24 wt% Er, and balanced Al. The ingot was homogenized at 470 °C for 20 h in an air-circulating oven and, afterwards, was cut and milled. The ingot was then hot rolled up to 75% total reduction in thickness with a starting temperature of 450 °C and an ending temperature of 300 °C. The final thickness of the hot rolled plate was 30 mm.

Two portions of the sample were removed and mechanically polished from cross sections namely ND-TD plane: sample 1 (similarly hereinafter) was removed from the center of the specimen, and sample 2 (similarly hereinafter) was removed from the surface of the specimen. The EBSD measurements were performed using a high speed EBSD detector incorporated in a Zeiss Supra 55 scanning electron microscope. To ensure the accuracy of the measurements, the EBSD data were recorded with a step size of 1 μm.

The first step in EBSD data processing was to use a cleanup procedure to correct spurious points in the orientation map due to incorrect indexing. The microstructures of the samples were then indicated by the inverse pole figure (IPF) maps. Subsequently, the hot rolled textures were revealed both by crystal orientation maps (showing the spatial distributions and volume fractions for the ideal fcc rolling components) and the orientation distribution functions (ODFs) (describing the grain orientation textures in the Euler space). The misorientation across the observed boundaries was illustrated by the misorientation angle distribution functions (MDFs).

The observations needed for the FPA analysis are line segments that are extracted from the orientation maps and are associated with the crystal orientations. Using the FPA method, the GBPD,  $\lambda(\Delta g, n)$ , is defined as the relative area of a grain boundary plane with a misorientation,  $\Delta g$ , and boundary plane normal,  $n$ , in units of multiples of a random distribution (MRD). Note that when the GBPD is averaged over all misorientation, the GBPD,  $\lambda(n)$ , represents the distribution of boundary planes in the crystal frame of reference.<sup>[5]</sup> Also note that for crystals with cubic symmetry, the FPA method requires  $5 \times 10^4$  line segments for a complete analysis<sup>[21]</sup> and only needs  $2 \times 10^3$  line segments if the misorientation parameters are not considered. The current work merely concentrates on  $\lambda(n)$  and therefore, such analysis should be accurate with  $2 \times 10^3$  line segments. Actually, each GBPD analysis in this work contains enough line segments and therefore can be regarded as reliable. The GBPDs were calculated using stereological programs developed at Carnegie Mellon University, and the details are described in reference.<sup>[2]</sup>

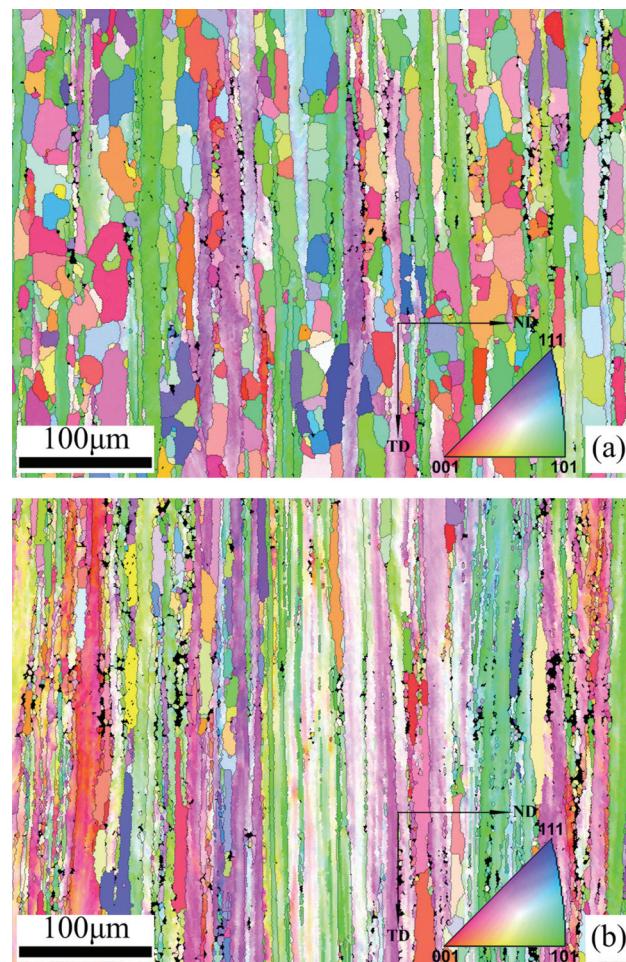
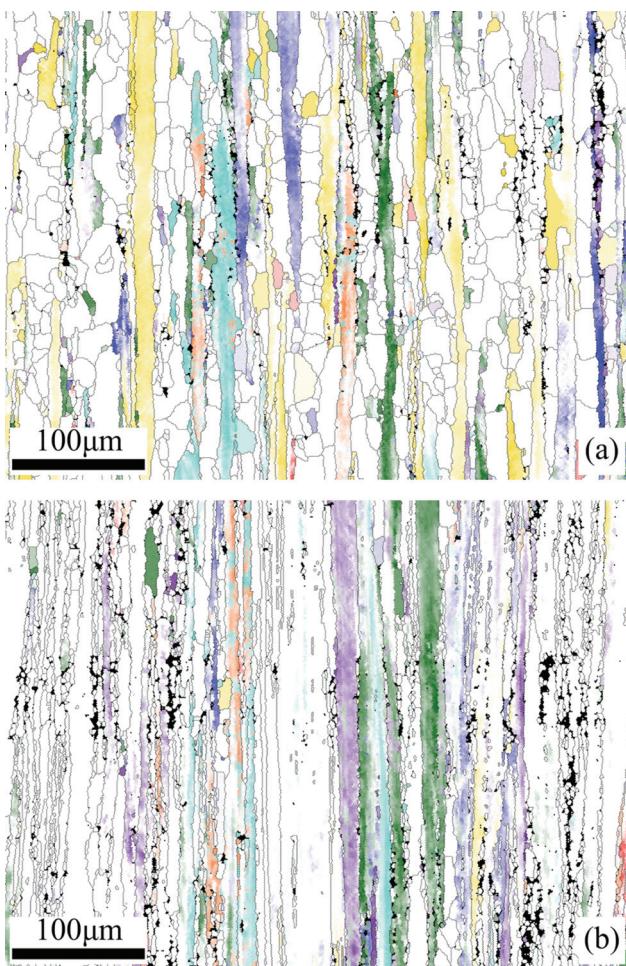


Fig. 1. Inverse pole figure (IPF) maps of the two samples, (a) sample 1, (b) sample 2, with orientations determined by the color coding scheme for cubic system.

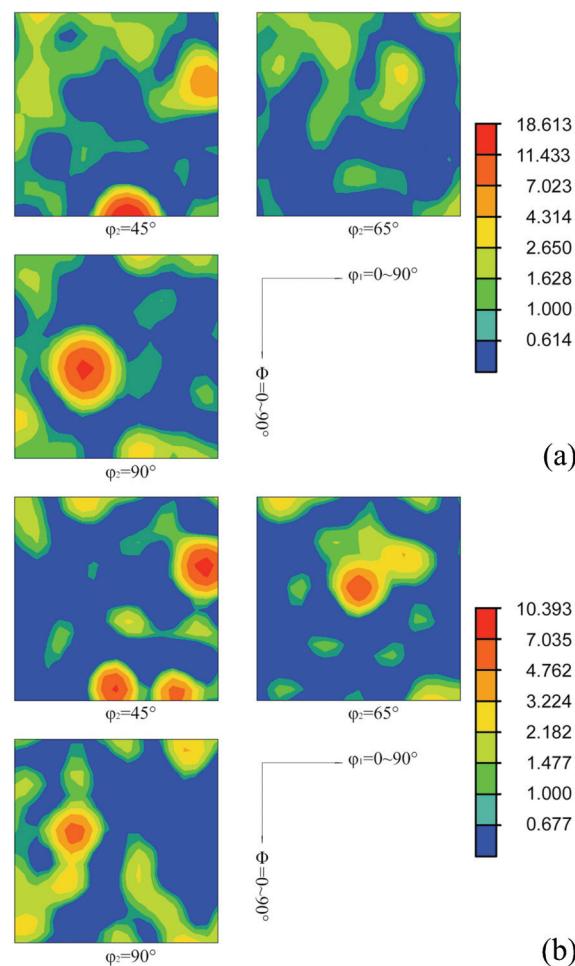
## 3. Results and Discussions

The microstructures of the two samples are depicted by the inverse pole figure (IPF) maps in Figure 1, where the grain color specifies the orientation according to the coloring indicated in the key. The images illustrate that hot rolling has affected the homogeneity of the microstructure, and one can observe that grains align along the rolling direction created a lamellar structure, however, the lamellas were differently distorted at different locations, that is, grains on the surface suffered more deformation and had thinner shapes than those at the center of the ingot, which might bring the inhomogeneous distribution of hot rolled textures.

To analyze the spatial distribution of hot rolled textures in the two samples, crystal orientation maps showing the volume fractions of orientations near the ideal fcc rolling components are presented in Figure 2, in which each grain color specifies the texture component. According to Figure 2, the most prevalent textures are different in the two samples: in sample 1, (110)⟨1–12⟩ Brass, (231)⟨3–46⟩ S3, and (241)⟨1–12⟩ S1 textures occupy 16.9%, 6.9%, and 6.6% volume fractions respectively, whereas in sample 2, (241)⟨1–12⟩ S1, (231)⟨1–24⟩ S2, and (4 4 11)⟨11 11 –8⟩ Taylor textures occupy 10.3%, 8.8%,



**Fig. 2.** Crystal orientation maps showing the volume fractions for the ideal fcc rolling components without considering the orthotropic variants, (a) sample 1, (b) sample 2, with rolling textures indexed by different colors, where orange for Copper, green for S1, purple for S2, blue for S3, cyan for Taylor, yellow for Brass, and red for Goss.



**Fig. 3.** Orientation distribution functions (ODFs) of the two samples plotted in the reduced Euler space, (a) sample 1, (b) sample 2, with units of the contours in MRD.

and 5.8% volume fractions, respectively. The observation demonstrates that textures produced by the hot rolling process are mainly dominated by components belonging to the so-called  $\beta$ -skeleton line,<sup>[6]</sup> and specifically, S1 texture has a higher volume fraction on the surface than the volume fraction at the center, which is consistent with the conventional understanding that the deformation texture generally increases under the higher deformation degree.

The grain orientation textures imparted by the hot rolling process were specified by the ODFs plotted into a reduced Euler space, as illustrated in Figure 3. In the figure, contours in units of MRD represent the distribution densities of the referred textures. It can be observed that the textures are dissimilar at different locations. For example, on the  $\varphi_2 = 45^\circ$  ODF sections, Brass texture has the major peak in sample 1, while in sample 2, the major peak is close to the Copper texture position, and the Brass texture is merely the secondary peak with a relatively weaker spread around. On the  $\varphi_2 = 65^\circ$  and  $\varphi_2 = 90^\circ$  ODF sections, different texture components

can also be observed in the two samples; moreover, the distribution densities of major peaks in the two samples are different.

Both Figure 2 and 3 demonstrate that the crystal orientation textures in the hot rolled sample are not homogeneous, however, these characterizations ignore the distribution of boundary planes on the whole. As a beginning of investigating the distribution of grain boundary planes in the hot rolled structure, the MDFs showing the misorientations across the boundaries were calculated, and the result is illustrated in Figure 4. In the chart, the black line represents the misorientation distribution for an ideally random microstructure,<sup>[7]</sup> with blue and red lines showing the misorientation distributions for the grain boundaries in sample 1 and 2, respectively. It can be seen that the experimental distributions are clearly not random; that is, boundaries with misorientation angles lower than  $15^\circ$  (low angle boundaries, LABs) are remarkably above the random, and boundaries with misorientation angles larger than  $15^\circ$  (high angle boundaries, HABs)

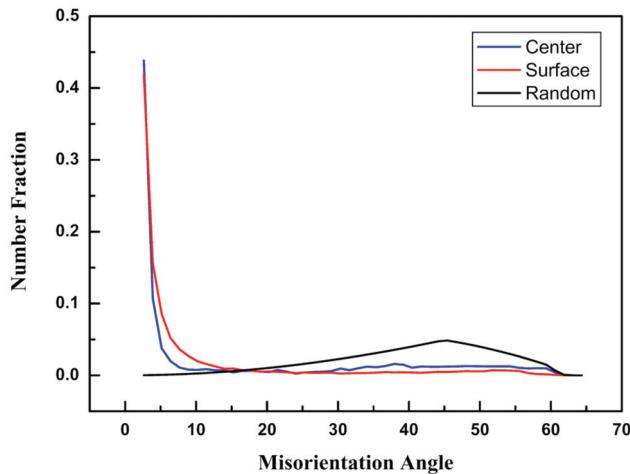


Fig. 4. Grain boundary populations in the two samples and the random distribution as a function of misorientation angle.

are beneath the random. The results clearly reveal the prevalence for LABs in both samples.

For both samples, the GBPDs,  $\lambda(n)$ , which are independent of the misorientation parameters, are plotted in the crystal reference frame in Figure 5 to show the relative areas of grain boundary planes. In Figure 5, values greater than one MRD indicate the total area associated with a specific type of plane is larger than the area that would be expected in a random distribution, and values less than one MRD are associated with specific planes whose total areas are less than the area that would be expected in a random distribution. For the entire boundaries in sample 1, the most frequently observed grain boundary plane orientation is {111} with a relative area 56% higher than that in the random distribution; the smaller peak for {110} planes is 8% higher than that in the random distribution (see Figure 5a). For the entire boundaries in sample 2 however, the maximum of the distribution is at {110} positions and the relative area is 69% higher than that in the random distribution; the smaller peak for {111} planes is 9% lower than that in the random distribution (see Figure 5b). Note that the {100} orientations are the minimum points in the GBPDs of both samples. In materials undergoing normal grain growth (not the case here), it is known that the population of grain boundary planes is generally inversely correlated to the grain boundary energy.<sup>[3]</sup> In aluminum alloys, the {111} surface has the lowest energy, {110} has the highest, and {100} is intermediate, and the total anisotropy is within 15%.<sup>[8]</sup> Another literature<sup>[9]</sup> also reports that boundaries based on {111} planes in fcc metals exhibit unusually low energies. Measurements of the GBPD of commercially pure Al are consistent with these ideas and show a maximum at the {111} orientation.<sup>[10]</sup> Considering this, the observed GBPDs in current work may suggest the anisotropy of the crystallographic orientation dependent performances.

Recent research indicates that there is significant anisotropy in the GBPDs at fixed lattice misorientations,<sup>[11]</sup> so we should examine the GBPD in subsets defined by ranges of the

misorientation angle. According to their populations illustrated in Figure 4, boundaries in both samples are divided into two sections: those with misorientation angles lower than 15° (LABs) and those above 15° (HABs). Note that during data cleanup procedure, a minimum misorientation angle of 5° was used to define a boundary, which makes the partition of LABs and HABs meaningful for GBPD analyzing. The GBPDs from these subsets are also shown in Figure 5. The GBPDs from sample 1 indicate that although a high preference for {111} planes can be observed in both LABs and HABs, the frequency of occurrence varies from 2.3 MRD (see Figure 5c) to 1.5 MRD (see Figure 5e). Particularly, in Figure 5c, contours around {111} position approach to {122} planes that deviate about 16° from {111} planes. Although the phenomenon is not very obvious, it possibly implies that LABs at center position of the ingot have multiple occurring planes, and some of these occurring planes might correspond to the substructure created by the hot deformation.<sup>[12]</sup> The results from sample 2 demonstrate that for all misorientations, the {110} orientations are preferred and a smaller maximum is found at {111}. The frequency of occurrence of the {110} plane decreases from 2.4 MRD (see Figure 5d) to 1.4 MRD (see Figure 5f). Overall, these results demonstrate that the GBPDs for LABs, HABs, and all boundaries, are qualitatively identical, and moreover, the preferred planes for all misorientations are the same: in sample 1 the preferred plane is {111}, and in sample 2 it is {110}, however, the spreads about these preferred planes are variant, and the spread about the preferred plane is greater for HABs in both samples.

Till now, it has been demonstrated that different low index crystallographic planes are preferred at different locations within the ingot. What's more, there is some indication that in the middle of the ingot, the population of boundary planes is inversely correlated to the boundary energy. In this sense, the heterogeneity of the boundary planes can substantially help to predict the impact of boundary plane orientation texture on material performance. The preponderance of high-energy {110} planes at the surface, as a result of hot rolling, could suggest degraded performance for intergranular corrosion. These {110} planes will be more reactive, and the surface should corrode faster. The diminished corrosion resistance at the surface will introduce surface cracks which will lead to poor performance overall if the sample is under load (especially tensile load, and even worse if it is under fatigue loading). Thus the results seem to suggest that the hot rolling should not improve performance, but rather diminish it. Therefore, the retention of a high percentage of low energy boundaries offers an important approach for performance improvement, and possible routes may include surface texture modification that produces LABs on low energy planes, and grain refinement by oriented growth control during annealing. Current work also shows that during hot rolling process, the surface of the slab typically experienced a higher deformation strain than that at the center, and grains on the surface therefore have overall higher stored energies for recrystallization than those at the center. This may also

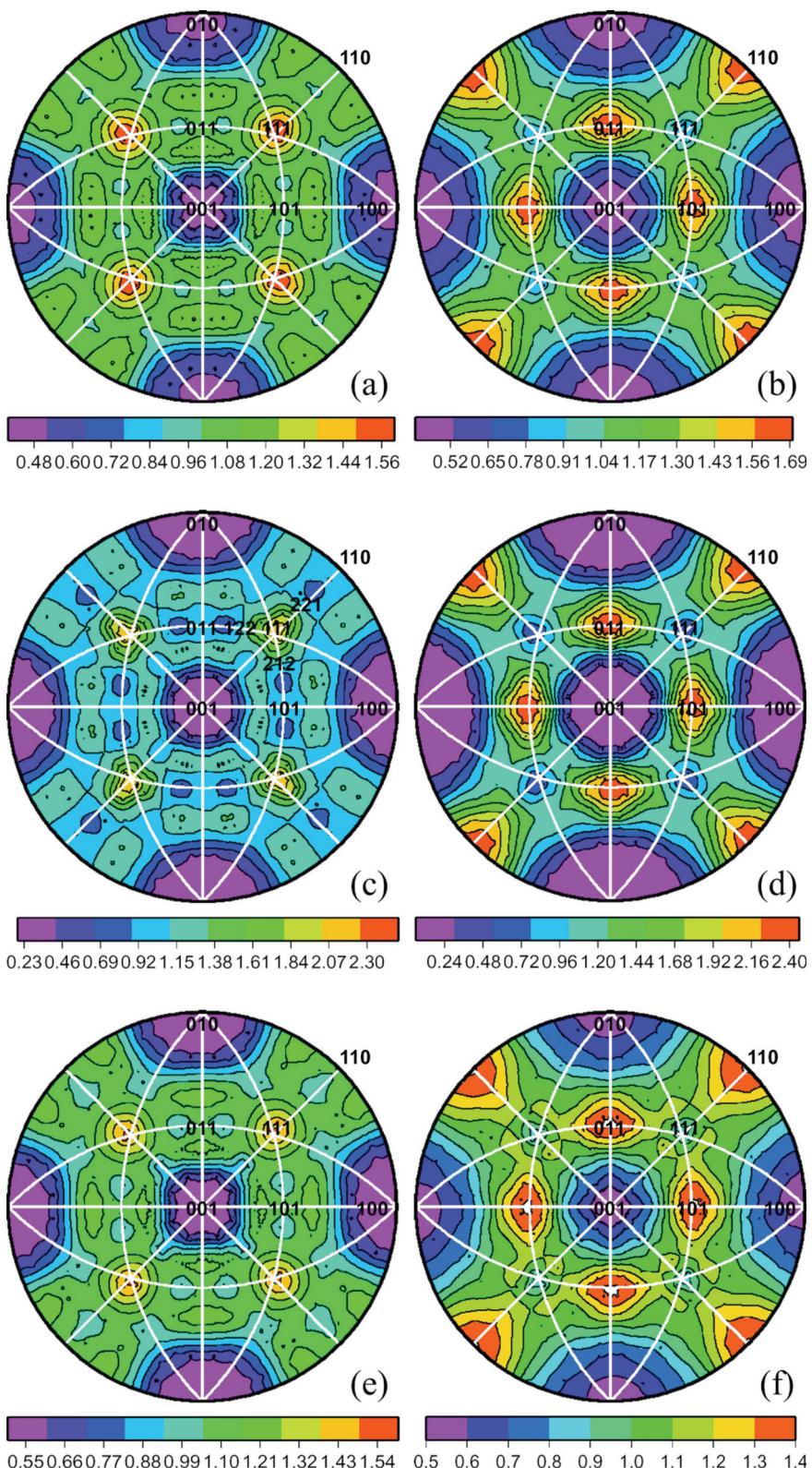


Fig. 5. The misorientation averaged distribution of boundary planes plotted in stereographic projection along [001] direction, for all boundaries in sample 1 (a) and sample 2 (b), for boundaries with misorientation angles lower than 15° in sample 1 (c) and sample 2 (d), and for boundaries with misorientation angles higher than 15° in sample 1 (e) and sample 2 (f). The units of the contours are in MRD.

influence the grain boundaries, which take higher energy orientations that include slip directions along the direction of the shear deformation. Meantime, the stored energy is the driving force for recrystallization during annealing, and it is claimed that recrystallization in fcc metals and alloys is mainly controlled by low stored energy nucleation.<sup>[13]</sup> Hence, the anisotropic GBPDs observed in this work could also present beneficial information to infer the growth rate of grain boundaries during annealing.

#### 4. Conclusions

Studies of the grain boundary plane distributions in a hot rolled aluminum alloy specimen lead to the following conclusions: hot rolling promotes the inhomogeneous GBPDs in the microstructure. Five-parameter analysis finds the preferential distribution of grain boundary planes. Boundary planes situated at the center and surface positions favor {111} and {110} orientations respectively, and such anisotropic distribution of grain boundary planes occurs at all misorientation angles. The spatially heterogeneous distribution of the grain boundary planes can influence the intergranular corrosion resistance as well as crystallographic evolution during subsequent annealing. Therefore, current observation suggests a new understanding of the pathway to improve the material performances that depend on the crystallographic orientation.

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