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Cube orientation in hot rolled high purity aluminum plate ①

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Abstract: X-ray diffraction and orientation mapping in EBSD measurement were applied to obtain information of deformation and recrystallization with the emphasis on the cube orientation in hot rolled high purity aluminum plates. It is shown that cube orientations are retained to a large extent during hot rolling. Some deformed cube grains are found to have experienced large extent of recovery according to their Kikuchi band contrasts. The deformed cube-oriented grains in hot rolled plates are in an unfavorite growth condition with respect to their neighboring grain orientations for the subsequent annealing. The reasons for the phenomena observed, as well as the influence of hot rolling process on subsequent cold rolling and final annealing were discussed.

Key words: high purity Al; cube texture; hot rolling; electron back-scattered diffraction; orientation mapping
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1 INTRODUCTION

It is well known that, in the production of beverage cans made of Al-alloys of 3×××or 5××× series, it is necessary to adjust suitably processing parameters of hot rolling, cold rolling and subsequent annealing so as to obtain a best combination of cube texture and deformation textures and to eliminate earings. In contrast, during the production of high voltage electrolytic capacitors, the hot rolling, cold rolling and annealing should be controlled to obtain the cube texture as strong as possible. The microstructure and texture evolution should be different in the two procedures due to the difference in composition and impurity contents. Recrystallization of high purity Al does not occur easily, either dynamically or statically during hot rolling passes, owing to strong recovery. Although detailed studies on cube texture formation were performed in AlMnMg alloy or commercial pure Al[1-3] and modellings of recrystallization were successful[4,5], there are few investigations on the cube orientation in hot rolled high purity Al and its influence on the subsequent processing. A throughout understanding on hot deformed microstructure as well as texture and their influences on subsequent processing is crucial because hot processing is one of the key factors influencing the cube texture intensity[6]. X-ray diffraction and EBSD (Electron Back-Scattered Diffraction) analyses indicated that the cube texture is always retained to some extent after hot rolling in Al alloys[1-3]. X-ray diffraction and, particularly, the orientation mapping in EBSD technique are used in this paper to reveal the structural and orientational features of hot rolling and subsequent recrystallization with the emphasis on cube-oriented grains. By making use of the acquired grain morphologies, misorientations, the Kikuchi band contrasts, it is also possible to obtain some knowledge about the extent of recovery and recrystallization so as to further understand the formation of cube texture.

2 EXPERIMENTAL

The investigated hot rolled high purity Al has the composition as follows: Fe $7\times10^{-6}\sim10\times10^{-6}$, Si $\leq10\times10^{-6}$, Cu $44\times10^{-6}\sim50\times10^{-6}$, Mg $\leq13\times10^{-6}$, Al > 99.99%. The thickness of slab is 200 mm. The rough rolling temperature is 570 ℃ and the finishing temperature is 271 ℃. The final thickness is 6.8 mm. The reduction is 96.6% in total. Samples were annealed in salt bad oven at 200 ℃, 300 ℃ and 500 ℃. Macro-texture was measured on the mid-thickness of samples using a Siemens-D5000 diffraction goniometer. Orientation mapping was conducted through an EBSD system[7] mounted on a LEO-1450 SEM. Samples for EBSD measurements were electrolytically polished and the measurements were performed on longitudinal sections made of RD-ND (rolling direction-normal direction). Orientation deviation was demonstrated by grey grade. The intensity lines in pole figures are 1, 2, 4, 8, 16, 32 × random respectively.

3 RESULTS AND ANALYSIS

Fig. 1 shows the macro-textures of hot-rolled samples

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as well as those annealed subsequently at 300 ℃ for 30 min and at 500 ℃ for 10 min. It is evident that after hot rolling a weak texture with cube component {100}〈001〉 and the TD-rotated cube component retained together with deformation texture components C{112}〈111〉, S{123}〈634〉, B{110}〈112〉(Fig. 1(a)). The difference of hot rolled samples from that of cold rolled plate/foils lies in the relative large percentage of cube grains retained after hot deformation. An annealing at 300 ℃ (Fig. 1(b)) gives rise to a slight increase of texture intensity, but the hot rolling texture is still retained. No preference is shown for cube texture. Orientation mapping indicates (shown later in Fig. 5) that recrystallization did take place and equal-axial grains were formed. The anneal at 500 ℃ (Fig. 1(c)) led to an increase of texture intensity, but no cube texture was found. Although the volume of cube grains in hot rolled plates was higher than that in cold rolled foils, no strong cube or R texture was produced after annealing.

Fig. 2 shows an example of orientation mapping. The dark color stands for TD-rotated cube orientation (see the left part of Fig. 2(a)). Color gradient corresponds to the deviation to idea cube orientation. It is seen that cube subgrains may be in different forms in the left two deformed grains. Whereas some of them are located near

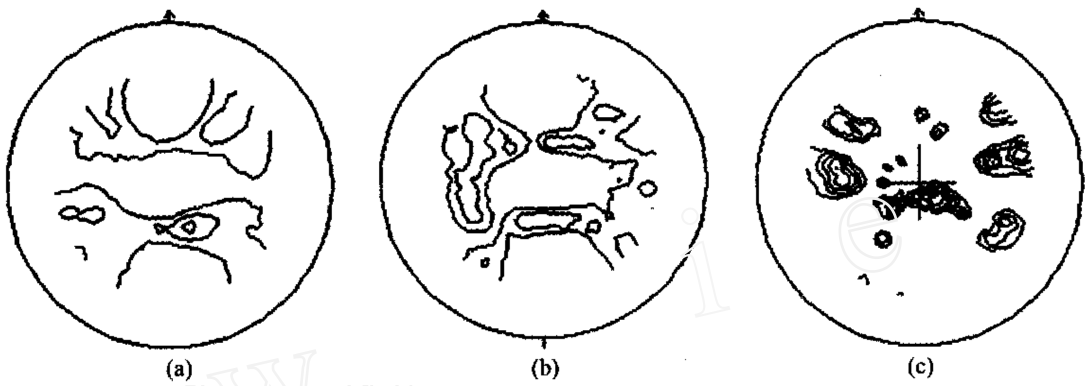
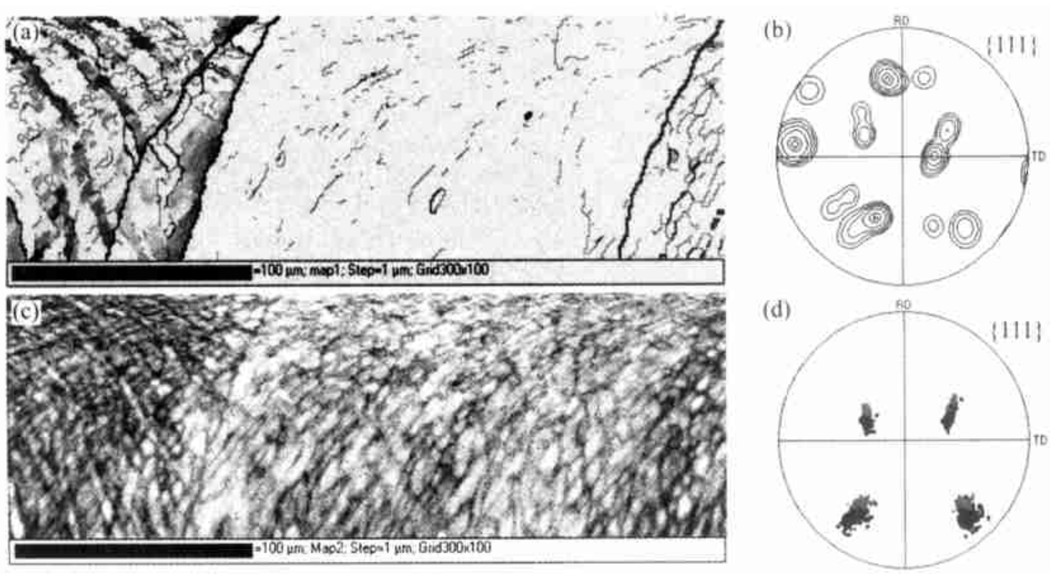


Fig. 1 Macro-textures of hot rolled and annealed samples, {111} pole figure, ↑ = RD, → = TD
(a) —Hot rolled; (b) —300 ℃, 30 min; (c) —500 ℃, 10 min



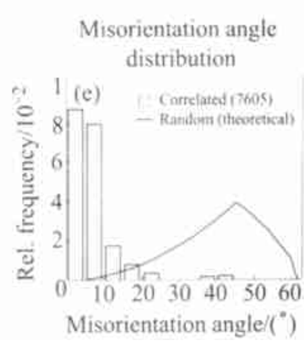


Fig. 2 Orientation mapping in hot rolled plate
(a) —Orientation mapping (dark: cube orientation), ↑ = RD, → = ND;
(b) —Distribution of Kikuchi band quality (darker color refers to more severe distortion in lattice);
(c) —Orientations in whole region;
(d) —Local orientations within left grain in (a);
(e) —Distribution of misorientation

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the grain boundaries, others are present in band form in an angle with rolling orientation. The map of Kikuchi band quality (Fig. 2(c)) reveals the morphology of subgrain microstructure. In this case the subgrains in the left part are smaller than those in the right deformed grains. No typical deformation orientation is found in this region (Fig. 2(b)). According to the local orientation presented in Fig. 2(d), it is clear that most part of the left grain in Fig. 2(a) experienced a rotation of TD-rotated cube orientation around TD, which is often seen in hot deformed Al or in the early stage of cold deformation[8]. In addition, more subgrain boundaries (thin lines represent subgrain boundaries of more than 5° and 10°) are found in the left deformed grains than in the right grains. Fig. 2 (e) demonstrates the misorientation distribution in the whole region. Low angle grain boundaries take the most part as is the case in cold rolled Al.

Fig. 3 is another example of orientation mapping. Grey color refers to cube oriented regions. In this case cube subgrains are parallel to RD. The left part of Fig. 3 (a) originates from a cube grain before deformation. After hot rolling some parts rotated to other orientations, whereas most part of it survived the rolling. The cube-oriented region has a width of more than 100 μm, being far thicker than a cube band observed in cold-rolled sample[9]. The distribution of Kikuchi band quality reveals a more strongly recovered region, i.e. light color in cube oriented region in comparison to the dark color of non-cube oriented region. Fig. 3 (c) indicates that no typical deformation orientation like C- or S-orientation was found in this region. Misorientation distribution shows (Fig. 3 (d)) a similar behavior as that of microstructures with strong deformation textures. The high percentage of low angle grain boundaries refers to abundant subgrain boundaries. Low angle grain boundaries can raise the stability of grain boundaries and therefore resist dynamic recrystallisation. It is noted that, since several cube-oriented regions are present within an original cube grain after hot rolling, several cube bands may be resulted during the subsequent cold rolling. In other words, a cube grain in hot rolled plate may lead to not just one, but several cube-oriented transition bands[10] in a cold rolled foil. Fig. 2 illustrates also the possibility of several cube bands within a deformed grain in spite of large deviation to idea cube orientation.

Fig. 4 shows an orientation mapping in a hot rolled sample after an anneal at 200 ℃ for 10 min. Firstly, a trace of S-shear band situated at an angle of about 35° to the rolling direction can be seen at the right part of Fig. 4 (a). New high angle grain boundaries have been formed at this place as potential nucleation site. Secondly, several recrystallized grains formed as their Kikuchi band qualities are higher than the surroundings (Fig. 4(b) small, white color). The orientations of these grains are nearly random (Fig. 4(e)). Some parts of elongated deformed grains are rotated to some extents to the S/R-orientations (dark color). Notice that most parts of cube grains have rotated to other orientations including S orientation, but still some of them are cube oriented (black color). They do not possess the feature of cube bands observed in cold rolled Al-Mn alloy[9]. Fig. 4 (a) indicates furthermore that S orientation can be produced from cube initial orientation. In Fig. 4(c) cube orientation can still be detected in scattered pole figure, while a strong B orientation can be seen in the pole figure

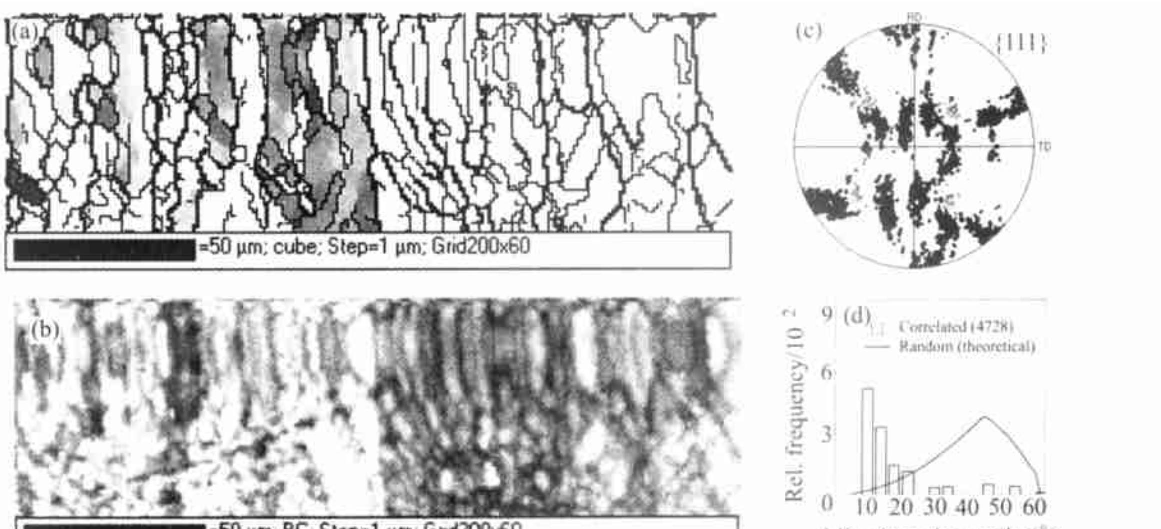

Fig. 3 Cube structure in hot rolled plate

(a) —Orientation mapping; (b) —Distribution of Kikuchi band quality;

(c) —{111} pole figure; (d) —Misorientation distribution

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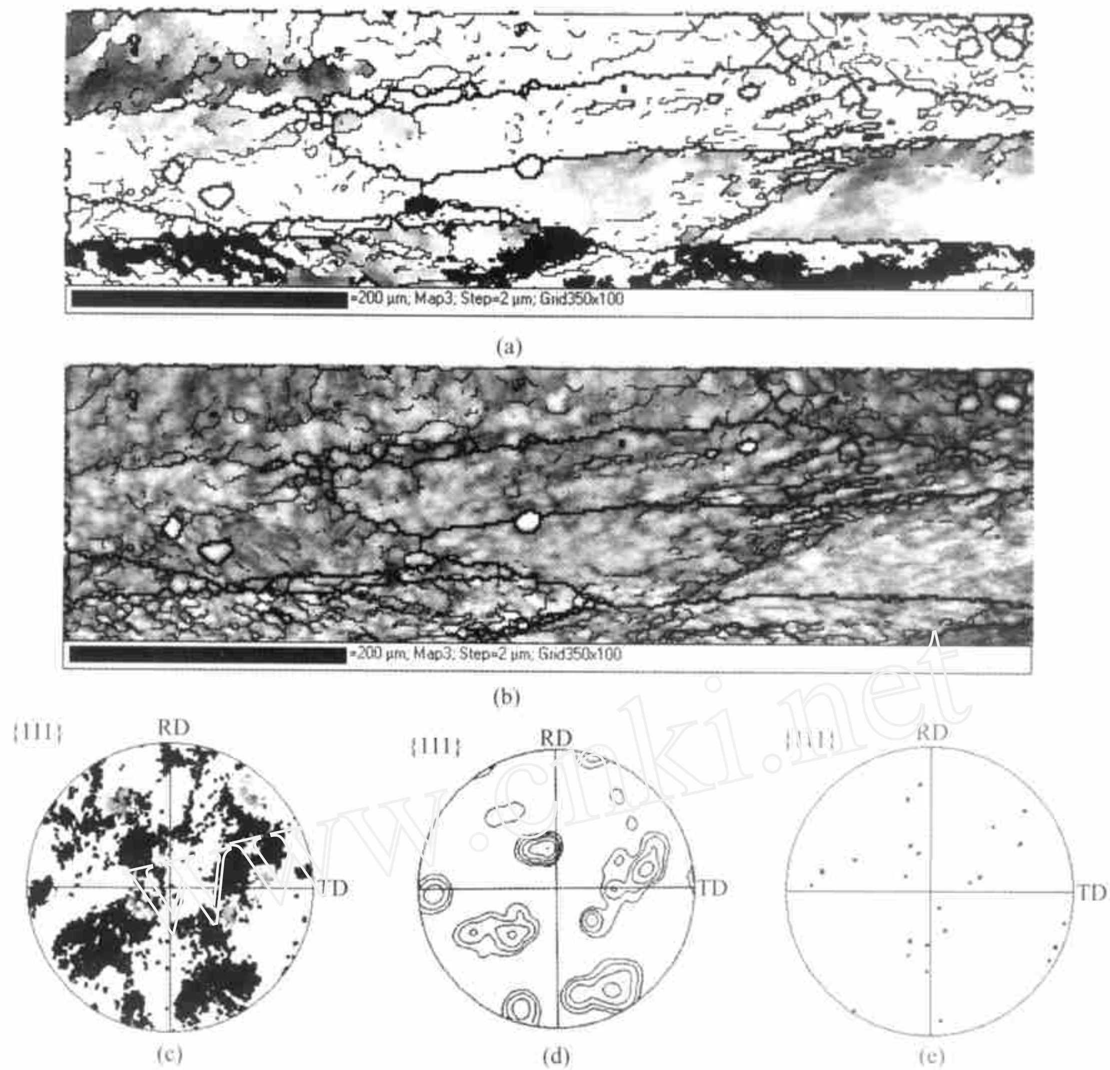

Fig. 4 Orientation mapping in hot rolled plate annealed at 200 ℃ for 10 min
(a) —Orientation mapping, ↑= ND, →= RD;
(b) —Distribution of Kikuchi band quality; (c) —{111} pole figure in scattered form;
(d) —{111} pole figure in iso-intensity line; (e) —Orientations of recrystallized grains

with iso-intensity lines (Fig. 4(d)).

Fig. 5 shows an orientation mapping on a sample hot rolled and subsequently annealed at 300 ℃ for 10 min. The area mapped is 2 mm ×1.7 mm = 3.4 mm^2. The sample was recrystallized completely and its grains are equiaxial. However, subgrain boundaries can still be seen inside some grains (plotted as thin lines). Some of them are cube oriented. The grain sizes are not uniform with large ones being 1 mm in diameter. Misorientation distribution (Fig. 5(d)) reveals a large percentage of high angle grain boundaries, which shows the features of structures with weak texture components.

Fig. 6 shows an orientation mapping on a sample annealed at 500 ℃ for 10 s. Similarly, recrystallisation is finished, but some subgrain boundaries exist mainly in elongated grains (see black thin lines in Fig. 6(a)). The mapped region has an area of 2 mm ×0.6 mm = 1.2 mm^2, but only two of grains are cube oriented. Moreover, cube grains do not show any preference in nucleation as well as in growth. Kikuchi band quality provides clear microstructure profile as shown in Fig. 6(c). The misorientation distribution in Fig. 6(c) indicates two peaks which are different from the single peak in low angle grain boundary region in cold rolled samples or in annealed samples with strong cube texture[11].

4 DISCUSSION

Maurice et al[8] have observed in a plane strain compression test on high purity Al of 99.996%, that above 300 ℃ cube orientation is stable during deformation because the slip systems are changed from {111}〈110〉 at

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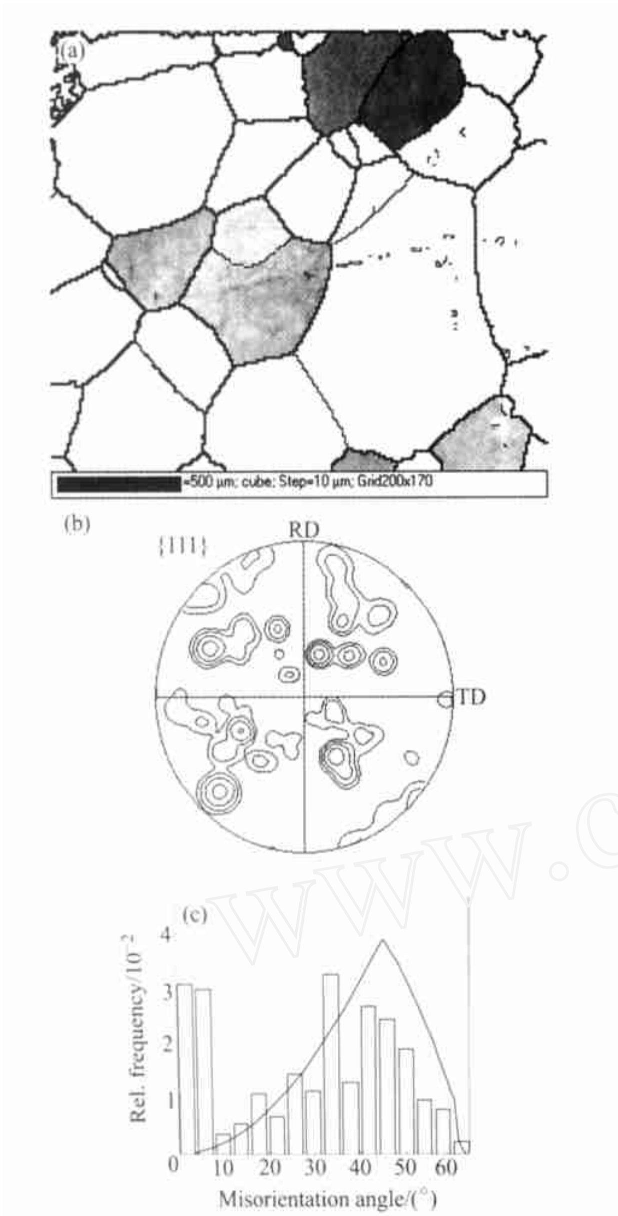

Fig. 5 Orientation mapping on sample hot rolled and then annealed at 300 ℃ for 10 min

(a) —Orientation mapping ↑= RD , →= ND ;
(b) —{111} pole figure ;
(c) —Misorientation distribution

room temperature to {110}〈110〉 at high temperatures. The critical temperature of the transition in slip system depends on strain rate. A higher strain rate corresponds to a higher critical temperature[1,2]. The hot rolling temperatures in this work decreased from stable region of cube orientation to unstable one (570～270 ℃) , the basic features of cube grains are in agreement with those of Maurice et al[8]. According to the 6 orientation mappings on hot rolled samples , as the hot rolling temperature declined to 271 ℃ (plate thickness is 6. 8 mm with a total reduction of 96 %) , cube oriented regions are still rather thick. As cast Al slabs normally contain cube grains[12] , this leads to elongated cube grains in hot rolled plates. If the cube-oriented parts in a large initial cube grain were present as layered form with respect to non-cube parts , it would indicate that the cube orientation is not stable as a whole. On the other hand there are maybe several cube bands formed , serving as nucleation sites during subsequent annealing.

The hot rolling is processed in a temperature-decrease mode. Although it is difficult to know the microstructure evolution at high temperature , it is less possible for the occurrence of dynamic recrystallisation due to the strong recovery reducing the driving force for recrystallisation. On the other hand , the existence of plenty of B-orientation provides large resistance to recrystallisation[13]. According to observation it is clear that in addition to the above mentioned factors the abundant low angle grain boundaries (Figs. 2(e) , 3(d)) reveal the resistance to dynamic recrystallisation. Results of X-ray diffraction (Fig. 1(a)) indicates that many cube grains survive after hot rolling. Orientation mapping illustrates , however , that the cube-oriented regions after hot rolling are not effective nucleation sites for recrystallisation , because they are not frequently connected with S-oriented regions and do not correspond to the condition for quick movement of grain boundaries[10]. It is well known that the cold rolling with subsequent annealing can bring about strong cube texture. Although the cube structure in hot rolled plates can not lead to strong cube texture by dynamic recrystallisation or static recrystallisation afterwards , it can set up a better basis for the formation of high cube texture after cold rolling and subsequent annealing.

The higher percentage of cube volume in hot rolled plates than in cold rolled foils could not give rise to stronger cube texture (Figs. 1 ,5 ,6) by annealing and this indicates that the volume of cube grains is not directly proportional to that in annealed samples. An important factor is the presence of S-oriented deformed grains surrounding them. Since the S orientation is the final stable orientation of cube orientation during rolling[14] , a suitable cold rolling for setting-up their neighborhood is one of the key factors for enhancing cube intensity.

Fig. 4 and Fig. 5 indicate that if the hot rolling temperature is so high that static recrystallisation can occur during cooling , therefore no strong cube texture will be expected and the idea cold rolling texture and annealing texture afterwards will be influenced. So , the finishing temperature of hot rolling can not be held too high and enough thickness of hot rolled plates should be kept for cold rolling. On the contrary , if a heavy cold rolling is performed directly after casting without an appropriate hot rolling , the cube bands in cold rolled sheets as cube

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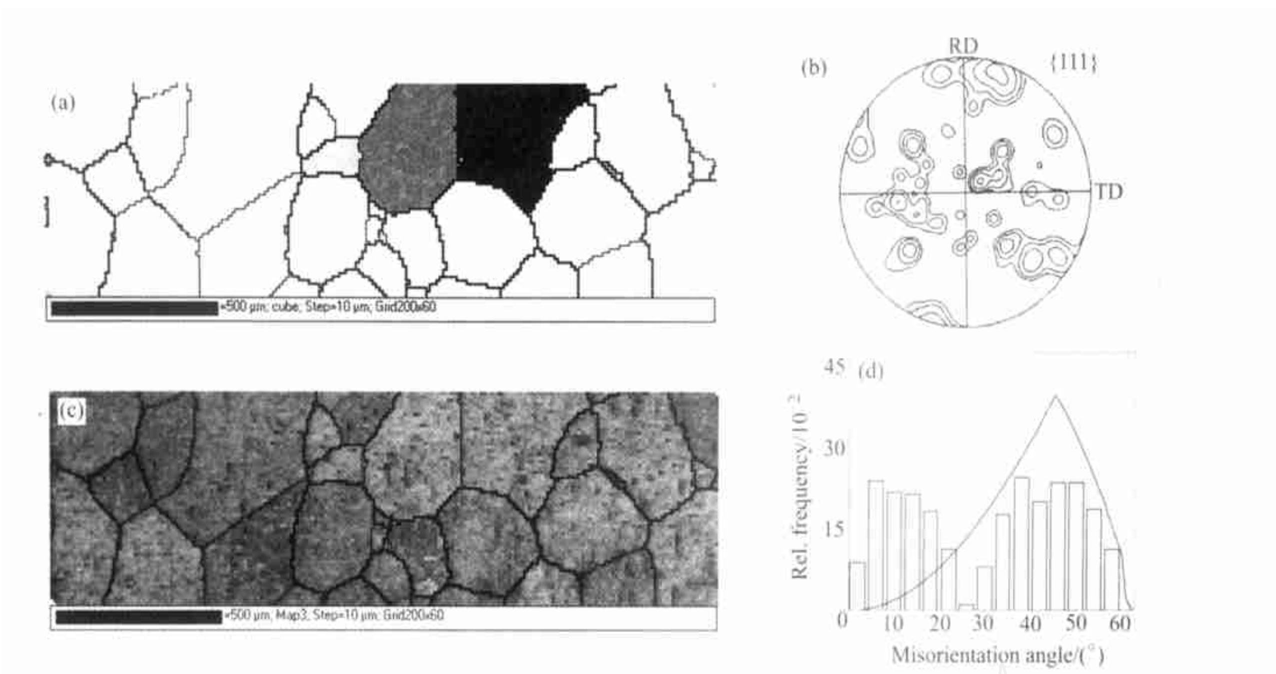

Fig. 6 Orientation mapping on sample annealed at 500 °C for 10 s

(a) —Orientation mapping (grey: cube orientation) ↑= RD, →= ND; (b) —{111} pole figure;

(c) —Distribution of Kikuchi band quality; (d) —Misorientation distribution

seeds for recrystallisation will be strongly reduced, because the cube orientation is unstable at low deforming temperature. This will lead to a weak cube texture in final products. The study on the effect of hot rolling temperature on cube texture in high voltage electrolytic Al capacitors[6] indicates that in a low hot rolling temperature range the finishing temperature only shifts to the suitable intermediate annealing temperature and has less influence on maximal cube texture intensity. Therefore, one can expect that only a suitable combination of hot rolling, cold rolling and annealing can produce a high percentage of cube grains over 90 %.

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