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Influence of Cold Rolled Deformation Degree and Heating Rates on Crystallite Dimension and Recrystallization Fraction of Aluminum Plates

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Abstract: In order to study the microstructure evolution rule of pure aluminum plates during different cold-rolled (CR) deformation degrees and annealing processes, samples with aCR deformation of 50~85%, heating rates of 60~100 °C/min and annealing at the target temperature of 350~500 °C were investigated. The microstructure, crystallite dimension and grain boundary characteristics were characterized by the methods of polarizing microscope (PM) and electron backscattered diffraction (EBSD). The results showed that the crystallite dimension of the initial state was 102 μm and ends up completely broken with an increase in the CR deformation degree. When the CR deformation increases to 85%, the deformed micro-bands were very small, with a band spacing of 5~10 μm. At this time, the grain distortion is more serious, there are more high-density grain defects, such as dislocations, and there is a high deformation of the storage energy, which is the energy preparation for the subsequent finished products to withstand the annealing process. The recrystallization fraction was higher with an increase in annealing temperature. After completed recrystallization, the grains showed an equiaxed shape. Orientation imaging and misorientation angle analysis showed that the red-oriented grains of the (001) plane, which had preferred nucleation, recrystallization and rapid grain growth. Final grains of the completed recrystallization are relatively coarse. Under the same deformation, the average crystallite dimension of the recrystallized grains decreases with an increase in annealing heating rate.

Keywords: cold rolling; annealing; heating rates; crystallite dimension; recrystallization fraction

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1. Introduction

Aluminum alloy is a kind of nonferrous metal structural material with alight weight, high specific strength, high electrical conductivity and good ductility. It is the best material to replace traditional iron and steel in the field of light weight, which is widely used in automotive, aerospace, shipping and electronic products. In order to further improve the usage range and performance requirements of aluminum alloy, along with the microstructure evolution and property changes during deformation, recovery and recrystallization is receiving more and more attention and research by scholars in the field of aluminum alloy [1–3], through studies into the regulation and control on the microstructure, and the performance of aluminum alloy is being adapted to the market demand, such as using more and more packaging and the electronic electrical industry using thinner foils acting as a raw materials.

Many scholars have conducted some in-depth studies on the microstructure evolution and properties tested in aluminum alloy. For example, the microstructure and texture evolution of cold-rolled AA1050 aluminum alloy with large strain was studied [4], as well as the corresponding variables, texture types and large angle interface. Li et al. [5] studied the effect of CR deformation on the microstructure and mechanical properties of 2519 aluminum alloy and mainly discussed the relationship between dispersion phase, dislocation and alloy strengthening. Zhao et al. [6] studied the effects of cold rolled rate and annealing process on the microstructure evolution of 5754 aluminum alloy used in automobiles and found that there were differences in crystallite dimension, texture and microstructure along the plate thickness. The influence of quenching cooling rate on the mechanical properties of 6082 aluminum alloy was studied by Wang et al. [7], who found that the quenching sensitive temperature range of 6082 aluminum alloy is 220~425°C, which provides a theoretical basis for regulating the precipitation of particles. These studies are based on the analysis and exploration of the mechanism during CR deformation, annealing and recrystallized grain growth in high purity aluminum [8–12], and providing technical support for the use of aluminum alloy in automotive, aerospace and other fields.

In this paper, annealing experiments with different CR deformation and controlled heating rates were carried out for high-purity aluminum plates. The purpose of this study is to research the changes in microstructure, grain boundary characteristics and texture during the CR process. In order to reveal the annealing characteristics of high-purity aluminum foil, the foundation is laid for the subsequent corrosion formation and cubic texture occupation of high voltage anode electrolytic capacitor aluminum foils.

Microstructure evolution rule was provided for basic research to the preparation of high-voltage anode capacitor aluminum foil products, and hopefully create a high-quality aluminum foil for capacitors and provide technical support for the market.

2. Experiment

In this study, the experimental material is a three-layered hydro electrolysis hot-rolled high-purity aluminum plate provided by Southwest Aluminum (Group) Co., Ltd., (Chongqing, China) The purity is 99.99%. Table 1 lists the chemical composition of the high-purity aluminum plate, which was detected using a PDA-6000 photoelectric spectrometer. Its high purity only contains trace impurity elements, and these impurity elements all exist in the aluminum matrix in the form of a solid solution, so its influence on recrystallization and grain growth can be ignored.

Table 1. Chemical composition of high-purity aluminum plates (mass content in ppm).

Chemical Elements	Al	Si	Fe	Cu	Mn	Mg	Ni	Zn	Ti
Mass content	Bal.	7	10	12	2	16	1	3	1

The process route of cold rolling and heat treatment in the research process is shown in Figure 1. A hot-rolled, high-purity aluminum plate with an original thickness of 7.6 mm was cold rolled to deformation degrees of 50%, 70% and 85%, respectively. Each cold deformed aluminum plate was heated from room temperature to the target temperature (350~500 °C) at different heating rates to finish annealing. The recrystallization volume fraction, crystallite dimension and grain boundary characteristics were studied by adjusting the heating rate (V1 > V2 > V3 > V4, where V_1 represents 100 °C/min, V_2 represents 60 °C/min, V_3 represents 40 °C/min, V_4 represents 5 °C/min). The annealed samples were anodic coated and electropolished and the microstructure was observed by PM and analyzed by EBSD. The composition of the electropolishing solution is a mixture of perchloric acid and alcohol, and liquid nitrogen was added to cool it to a low temperature. The ratio was $HClO_4$:C_2H_5OH = 1:9. For electrolytic polishing when the positive electrode of the power supply is connected to the sample and the negative electrode of the power supply is connected to the stainless steel, the standard parameters are as follows: voltage is 18 V, current is 0.10~0.25 A and time is 30~45 s. In these studies, the anodic coated liquid is composed of 5 mL HBF_4 and 200 mL H_2O, and the voltage is 12 V, current is 0.01~0.10 A, time is 15~30 s, and the principle of multiple laminating with a small current is used to better achieve the laminating effect, and which clearly show the grain boundary characteristics.

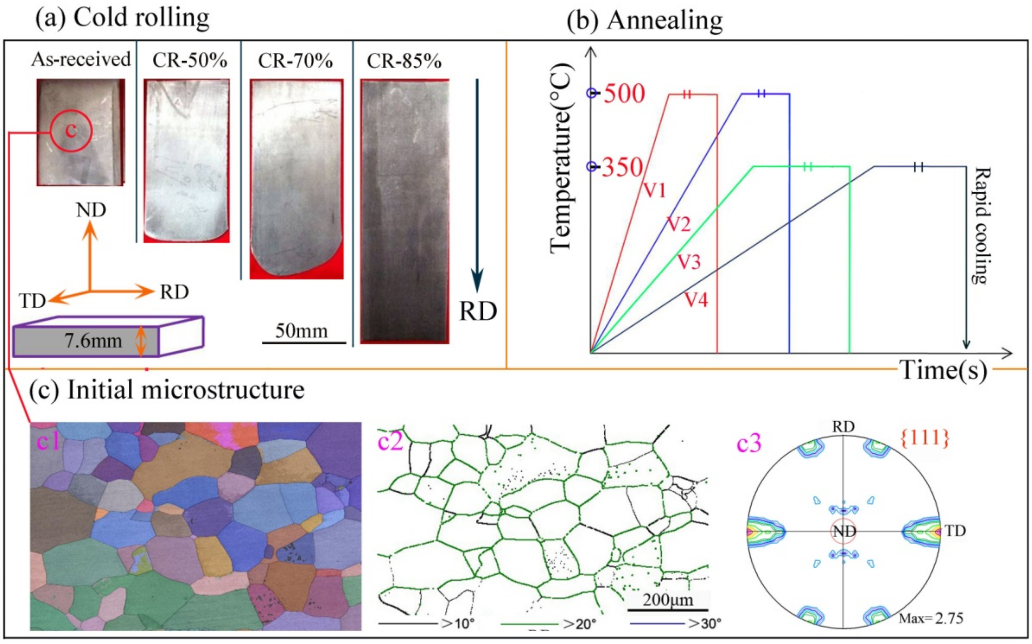

Figure 1. Study process and sample observation of (**a**) cold rolling; (**b**) annealing; (**c**) initial microstructure of hot-rolled aluminum plate.

3. Results and Discussions

3.1. Initial Microstructure of Aluminum Plate

The initial aluminum plate is hot-rolled, high-purity aluminum, and its microstructure, grain boundary characteristics and {111} polar diagram are shown in Figure 1c. The results show that the hot-rolled, high-purity aluminum plate has equiaxed recrystallized grain with an average grain size of 102 μm. The grain boundary characteristic diagram shows that the grain boundary angle is generally greater than 20°, which reveals that there are large angle grain boundaries and indicates the aluminum foil has undergone complete recrystallization, and there is a large angle grain boundary between adjacent grains. The {111} polar figure shows that the main texture of the aluminum plate is recrystallization texture, which mainly concentrates on the basal plane and has relatively high strength.

The hot-rolled, high-purity aluminum plate is used to prepare the material and structure for the subsequent cold rolled deformation and annealing process experiment controlled by different heating rates. It is hoped that the recrystallization behavior and the cubic texture occupancy in aluminum foil can be controlled by the CR deformation degree and heating rates with better microstructure so as to adjust the performance of the final product.

3.2. Cold Rolled Microstructure of Aluminum Plate

The high-purity aluminum plate is subjected to CR deformation of 50%, 70% and 85%, as shown in Figure 2. The initial plate is in the hot rolled state with equiaxed and completely recrystallized grains (Figure 2a). With the increase in CR deformation, the equiaxed grains were flattened and elongated gradually, and a certain deformation was produced along the rolling direction. When the CR deformation increases to 50%, the grains were flattened and showed long strips, and the deformation band spacing along the rolling direction was wide (Figure 2b), including a band space of 45 μm. When the cold rolled deformation increased to 70%, the grain deformation degree was more serious, and all grains were crushed and deformed in a slender strip (Figure 2c); the band spacing was 23 μm. Meanwhile, the cold rolling defects, such as dislocations, became gradually more obvious. When the deformation of cold rolling continued to increase to 85%, the number of deformed microbands increased to a visible quantity, the band spacing became smaller, and the grains were completely broken and disordered.

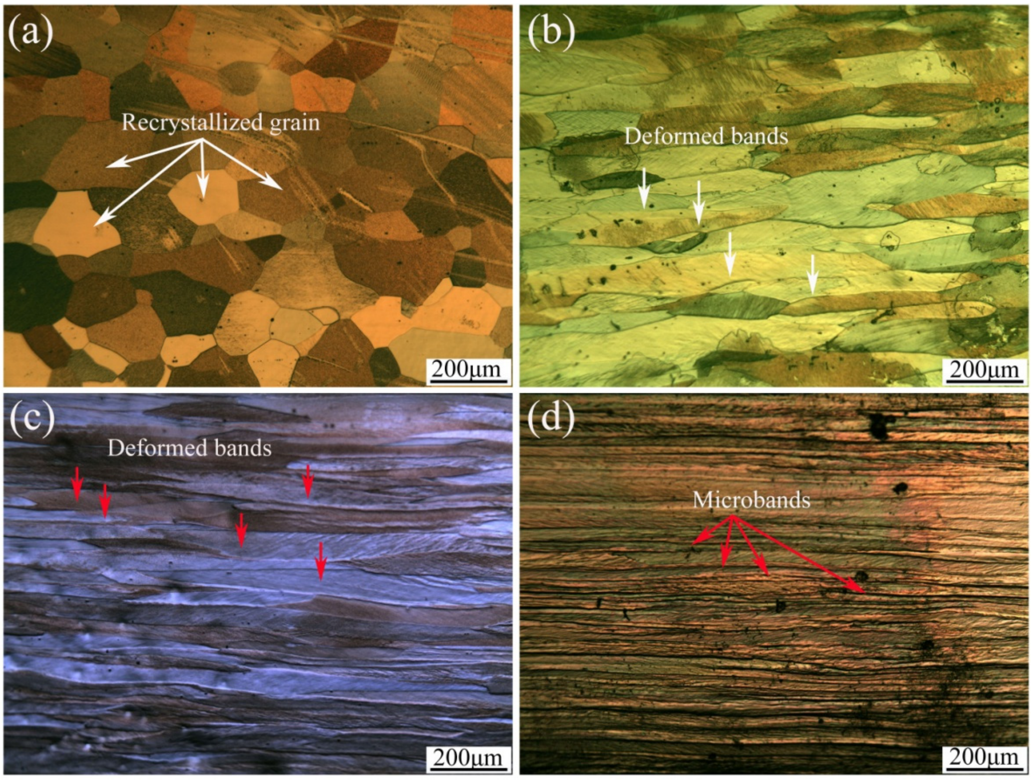

Figure 2. Polarized microstructuresof (**a**) as-received; (**b**) CR-50%; (**c**) CR-70%; (**d**) CR-85%.

Grain size, microstructure and hardening of high-purity aluminum plates during the cold rolling process were studied. It can be seen that with an increase in CR deformation degree, the grains are flattened, elongated, or even broken, and dislocations, subgrains and other defects also gradually appear. Figure 3 shows the grain orientation images and grain boundary structures of aluminum plates with different cold rolled shape variables.

Grain orientation image and grain boundary structure diagrams show that with the increase in CR deformation, grain orientation gradually shifts from (001) plane texture to (101) and (111) plane texture, and the proportion of low-angle grain boundaries (LABs) 0~10° becomes larger (shown in Figure 3b–d). This shows that the misorientation angles between adjacent grains are LABs after the grains are flattened and crushed. When the CR deformation increases to a higher degree, the main texture type is the (101) plane rolling texture, the grains are completely broken, and the grain boundary structure shows that most of the grain boundaries are LABs, and the misorientation angle is distributed between 0~20°.

CR deformation is a process in which the deformation storage energy increases. The required energy and driving force are provided for the recovery and recrystallization of subsequent heat treatment in aluminum plates. The surface texture evolution from (001) to (101) and (111) shows that the grain orientation changes along with the rolling process, and the texture strength and type of the CR plate can be changed by heat treatment.

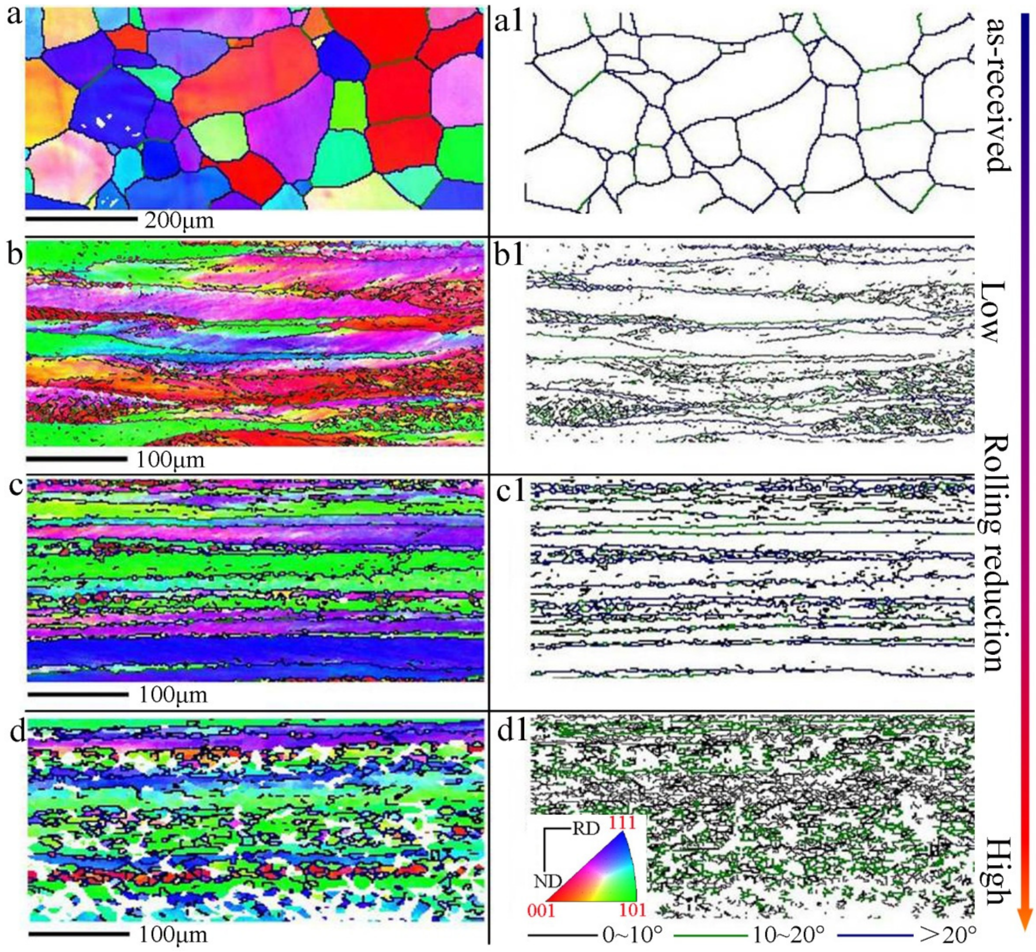

Figure 3. Orientation image microscope and boundary structure with different cold reductions of (**a**,**a1**) as-received; (**b**,**b1**) CR-70%; (**c**,**c1**) CR-85%; (**d**,**d1**) CR-98%.

3.3. Annealed Microstructure of Aluminum Plates

The finished product annealing experiments of high-purity aluminum with different CR deformation degrees of 50%, 70% and 85% were carried out at different heating rates. The annealing heating rates range from 60 °C/min to 100 °C/min, and the annealing target temperatures are 350~500 °C. After reaching the target temperature, the substance is directly cooled to a normal temperature without heat preservation, and the cooling rate is close to 1000 °C/s, so as to retain the current microstructure of the aluminum plate after annealing.

The electronic channel contrast (ECC) image of the CR-50% sample is not significantly different before and after annealing (shown in Figure 4a,b). The main reason for this is that the CR deformation degree is low, and the deformation storage energy is small, which is not sufficient to provide the driving force for recovery and recrystallization after the annealing process, although the annealing heating rate is different between the two samples. As can be seen in Figure 4c,d, the microstructure of the CR-70% sample is significantly different before and after annealing, which is due to the large amount of deformation and different annealed heating rates, so the microstructure changes are relatively obvious. On the one hand, as the annealing target temperature is 350 °C, most of the grains are in a state of recovery, and only a small proportion of the grains have undergone recrystallization. When the deformation degree increases to 80%, recrystallization occurs at a heating rate of 100 °C/min, which is thanks to a large amount of CR deformation and the high annealing heating rate, so that it provides a sufficient driving force for recrystallization for most of the grains.

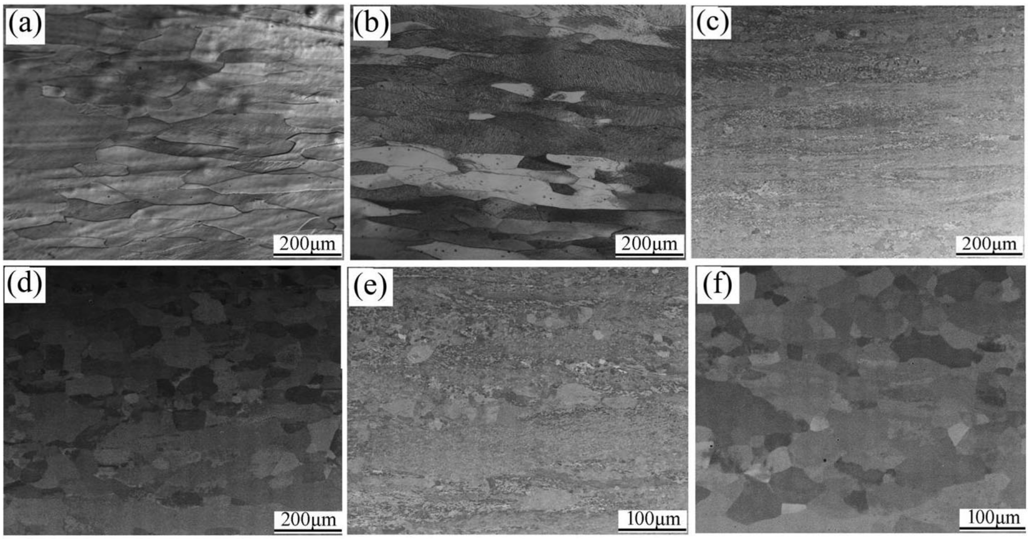

Figure 4. ECC image of 350 °C (**a**) CR-50%, V = 60 °C/min; (**b**) CR-50%, V = 100 °C/min; (**c**) CR-70%, V = 60 °C/min; (**d**) CR-70%, V = 100 °C/min; (**e**) CR-85%, V = 60 °C/min; (**f**) CR-85%, V = 100 °C/min.

In order to further investigate the effects of the CR deformation degree and the annealed heating rate on the recrystallization volume fraction and crystallite dimension, the annealing target temperature was increased to 500 °C (shown in Figure 5). When the annealing temperature is increased, the microstructure of the same cold rolled deformation varies greatly. The main reason for this is that high annealing temperature can reduce the recrystallization starting temperature, so that the grains can continue to grow after complete recrystallization, and the microstructure of some grains becomes relatively coarse due to excess driving force. As shown in Figure 4, a close observation is shown in Figure 5, which shows that the annealed temperature plays a major role in the same CR deformation or heating rate.

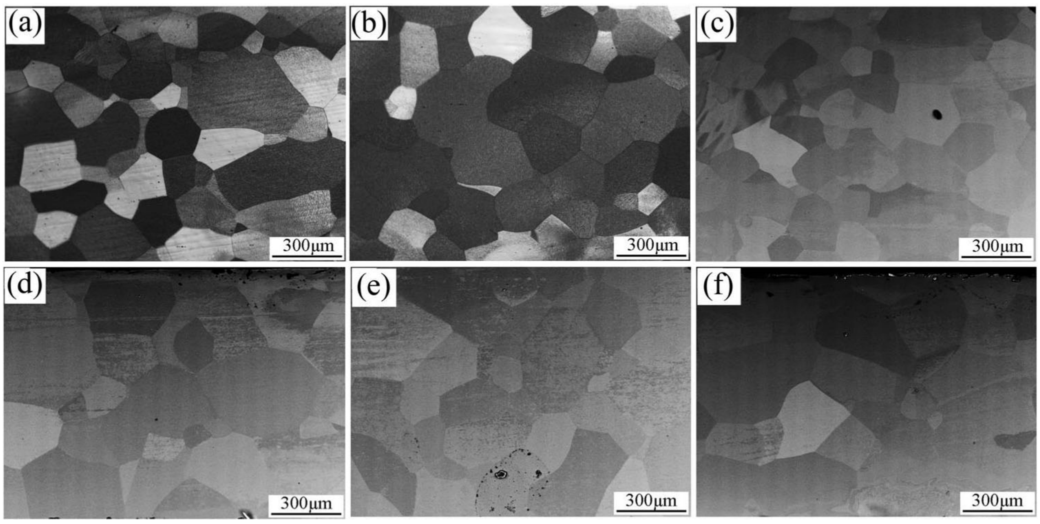

Figure 5. ECC image of 500 °C (**a**) CR-50%, V = 60 °C/min; (**b**) CR-50%, V = 100 °C/min; (**c**) CR-70%, V = 60 °C/min; (**d**) CR-70%, V = 100 °C/min; (**e**) CR-85%, V = 60 °C/min; (**f**) CR-85%, V = 100 °C/min.

The heating rate also plays a very important role in the annealing process because the high heating rate makes the aluminum plate too late to recover and directly enters the recrystallization stage. Most of the deformation storage energy is used as a driving force for direct recrystallization and complete grain growth; compared with conventional recrystallization theory, this recrystallization behavior is considered to be a rapid softening mechanism [10,12].

This method is controlled by the heating rate in the rapid annealing process, which is due to the short nucleation incubation period, where most of the deformation storage energy is used for pre-existing cores that can grow rapidly after annealing. In addition, the deformed substructure is consumed, and recrystallization and grain growth subsequently occur. Therefore, according to the observations in Figure 5, it can be seen that some grains have undergone complete recrystallization and then grow faster and larger in the crystallite dimension aspect.

3.4. Micro-Orientation Analysis of Aluminum Plate

The main purpose of grain orientation analysis is to obtain the texture characteristics of the micro area in aluminum plates, and to analyze the texture type of CR aluminum plates, along with recovery, recrystallization and grain growth during the annealing process. In Figure 6, the aluminum plates are subjected to CR deformation degrees of 70% and 85% and annealed at a heating rate of 60 °C/min with an annealing target temperature of 350 °C, and orientation imaging and misorientation angle analysis were conducted. It can be seen that the CR-70% aluminum plate underwent partial recrystallization and is still in a state of recovery locally, with an obvious gradient of microstructure state (Figure 6a). When the length of Line 1 is 60 μm in the neighboring grain space, the grain boundary gradient at A, B and C is obvious, which indicates that the grain boundary gradient is obviously caused by the change in grain boundary type.

According to the analysis of the misorientation angle in Figure 6e, most of the grains are still in a state of recovery, and most of the grains are broken, while a few grains have undergone recrystallization, which is shown in Figure 6a. The proportion of LABs was 87.2%. In Figure 6b, most of the grains have obviously recrystallized and the grain boundary is clearly in the organized space; the LAB proportion of 62.9% and the average misorientation angle of 16.2° are calculated using software.

As the annealing temperature increased to 500 °C (as shown in Figure 7), and, in turn, it was found that the recrystallization fraction increases compared with Figure 6, the grain misorientation angle becomes smaller, which indicates that the recrystallization texture of the same type and the grain orientation consistency increase after complete recrystallization, as can be seen from Figure 8b.There are more grains with (001) plane orientation [12–15], and the grains with this orientation are more obvious after recrystallization. Therefore, in the annealing process of aluminum plates, the micro-orientation of grains has a certain rule. In the case of severe deformation, the evolution process of the micro-texture of aluminum foil follows. After the recrystallization is completed, most of the cubic orientation is the main texture orientation [16–18] in aluminum plates.

3.5. Annealed Crystallite Dimension of Aluminum Plate

The distribution of crystallite dimension is shown in Figure 8. Through the comparison of the grain size distribution annealed at 350 °C and 500 °C, it was found that the average grain size was larger when the annealed temperature is 500 °C, whether at a heating rate of 60 °C/min or 100 °C/min, and it mainly depends on the annealed target temperature. For the same CR deformation, the higher the temperature, the greater the driving force for recrystallization. After the completion of recrystallization, the grain will continue to grow and will see secondary growth, where the grain will become coarser. This has long been studied by some scholars [14,17] in the annealing process of high purity aluminum or its alloys. After annealing at 500 °C, the grain even exceeds 300 μm and grows up to the millimeter level by grain-size statistics.

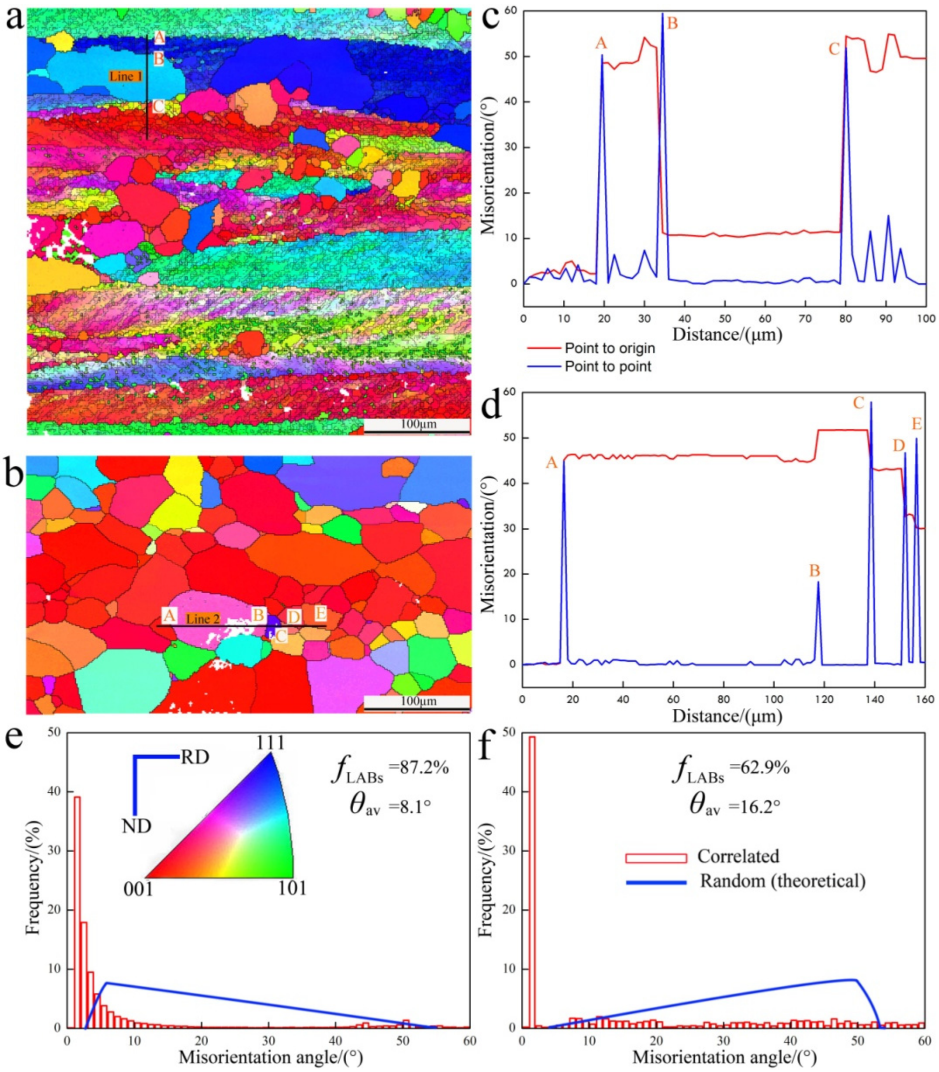

Figure 6. Orientation image and misorientation angle distribution with different heating rates of annealing (**a**) CR-70%, V = 60 °C/min, T = 350 °C; (**b**) CR-85%, V = 60 °C/min, T = 350 °C; (**c**,**d**) misorientation angle corresponding to (**a**,**b**); (**e**,**f**) misorientation angle corresponding to (**a**,**b**).

3.6. Effect of Temperature and Heating Rate on Recrystallization Fraction

According to EBSD IPF maps, which is reconstructed from the reverse pole diagram, it can be clearly seen that the grain size of aluminum foil was relatively large when annealing for 30 s, and the grain size of (001)-oriented grains has an advantage in Figure 9c. As the annealing time extends to 60 s, abnormal growth of individual grains appears immediately (as shown in Figure 9d, red grains). The abnormal grain growth mechanism and driving force of individual grains was further studied and discussed in the subsequent section, and the recrystallization mechanism and formation process of cubic texture were discussed, in which the grain growth was mainly studied in detail by experiments.

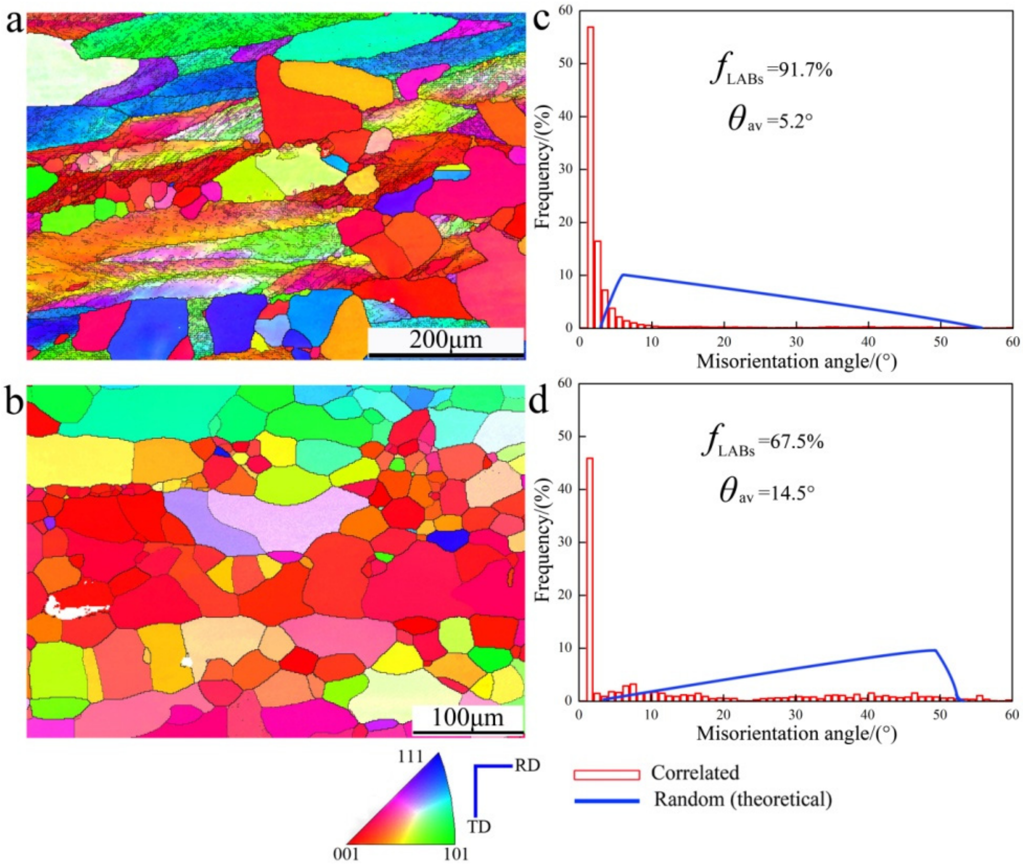

Figure 7. EBSD IPF maps of (**a**) CR-50%, T = 500 °C, t = 120 s; (**b**) CR-85%, T = 500 °C, t = 60 s; (**c**,**d**) Misorientation angle corresponding to (**a**,**b**).

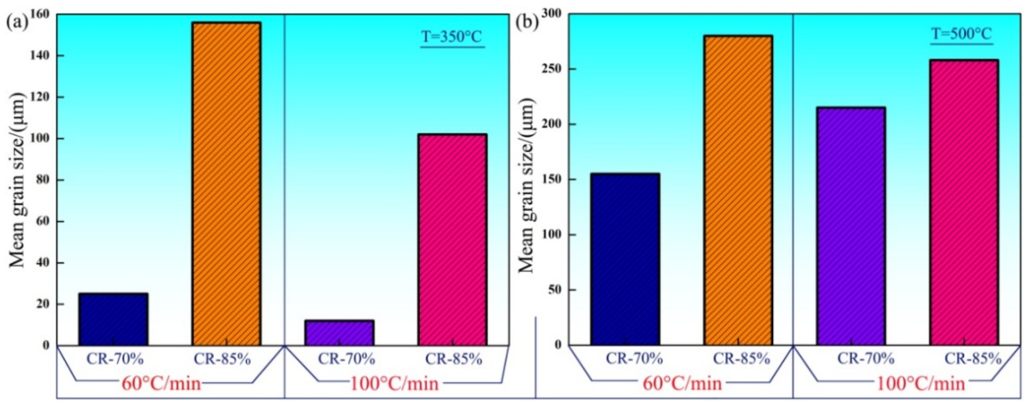

Figure 8. The mean grain size distribution of different heating rate annealing (**a**) T = 350 °C; (**b**) T = 500 °C.

By changing the heating rate to control the finished product, annealing has been gradually applied to the production of the finished aluminum plate or its thin foil. When the heating rate reaches a certain value, it can not only meet the product quality required by the annealed aluminum foil, but it also saves production costs, by shortening the production cycle. Therefore, the selection of an appropriate heating rate is critical in industrial production, and the heating rate will directly affect the degree of recrystallization and the content of cubic texture in an aluminum plate or its thin foil.

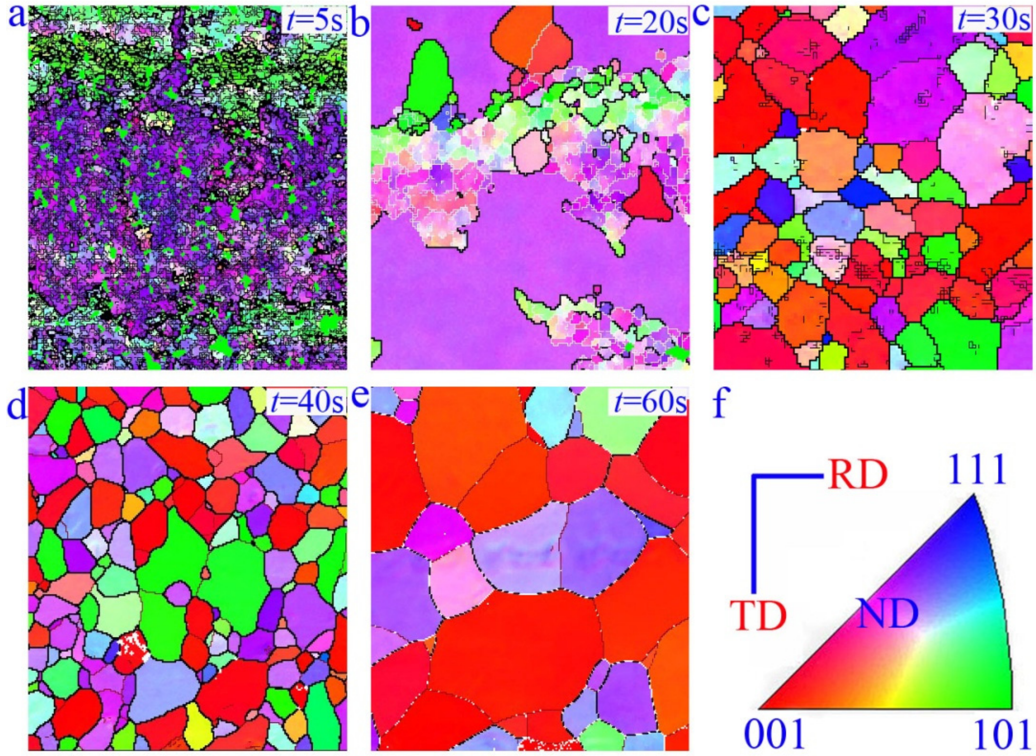

Figure 9. EBSD IPF maps of cold rolled CR-85% at 500 °C annealing for (**a**) 5 s; (**b**) 20 s; (**c**) 30 s; (**d**) 40 s; (**e**) 60 s; (**f**) color code.

In Figure 10, we can see that under the critical heating rate, the recrystallization starting temperature (T_{start}) begins to rise with the increase in heating rate increasing. Above the critical heating rate, the initial temperature of recrystallization decreases with the increase in heating rate because recovery is inhibited. The specific influencing mechanism is as follows: under the critical heating rate, the recrystallization starting temperature increase in the increasing of the heating rate (shown in Figure 10a). This is because the large heating rate means atoms have no time to diffuse and atomic migration lags in the annealing process, which is not conducive to recrystallization, so the recrystallization start temperature rises relatively. When the critical heating rate is exceeded, the atoms are activated, and the driving force of recrystallization increases so that the recrystallization temperature begins to drop.

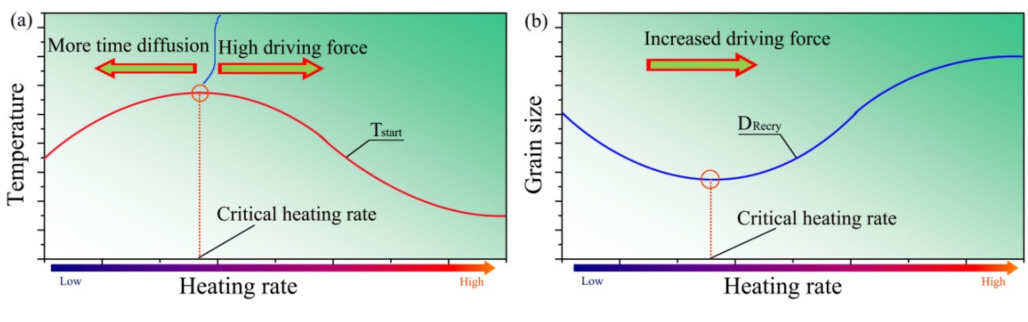

Figure 10. Schematic illustration for the suggested influence of heating rate on (**a**) the initiation of recrystallization; (**b**) recrystallized grain size.

The effect of heating rate on the recrystallized crystallite dimension (D_{Recry}) is shown as under the critical heating rate, the crystallite dimension gradually decreases with the increase in heating rate. This is because the large heating rate leads to a shorter annealing time and insufficient recrystallization and leads to fine grain sizes, as shown in Figure 10b. Above the critical heating rate, with the heating rate increasing, the crystallite dimension increases gradually, which is because the sample is too late to recover, and explained by super rapid softening mechanism [19,20], leads directly into the recrystallization stage. Further, the deformation energy storage and driving force increase, the recrystallized grains grow quickly, which shows that the final size is also course.

In the experimental scheme designed in this paper to obtain a uniform microstructure and high occupancy cubic texture, the selection of heating rate combined with the actual production situation of the factory is below the range of the critical heating rate. Therefore, in order to obtain a relatively small crystallite dimension, it is necessary to confirm an appropriate heating rate so as to improve the quality and energy storage of high voltage anode aluminum foil to a certain extent.

4. Conclusions

In summary, microstructure, grain boundary characteristics, crystallite dimension and recrystallization fraction evolution rule during CR deformation, and annealing processes were investigated by the PM and EBSD devices. Several conclusions can be drawn as follows:

(1) Grain distortion and deformation storage energy are mainly attributed to the CR deformation degree, and after a serious CR deformation, no complete grains can be observed. When the CR deformation degree increases to 85% and 98%, the deformed microbands are very small, with a band spacing of about 5~10 μm. At this time, most of the grains will have broken, the grain boundary is not clear, dislocation packing, and other defects become serious with the increase in dislocation density, and the distortion energy generated by the lattice distortion is also increasing.

(2) As the heating rate increases to 100 °C/min, the sample cannot recover quickly and directly enters the recrystallization stage, which is due to the high heating rate. Most of the deformation storage energy as the driving force is directly used for recrystallization and grain growth. In the rapid annealing process, it is controlled by the heating rate due to the short nucleation and incubation period, and most of the deformation storage energy is used for pre-existing cores that can grow rapidly and consume the deformed substructures. Finally, the recrystallization is completed, and the grains grow subsequently.

(3) Orientation imaging and misorientation angle analysis show that the red oriented grains of the (001) plane are preferred to nucleate and grow the grains. After the recrystallization process is completed, abnormal grain growth behavior appears, and the complete recrystallized grain structure is not uniform; some of the grains are relatively coarse, which is caused by the different annealing heating rates and recrystallization driving forces.

Author Contributions: Y.W.: writing the original manuscript, carrying out the formal analysis and obtaining the project funding. L.R.: assisting in revising the manuscript and editing. J.D.: performing the EBSD characterization. C.C.: carrying out software employment and calculation. All authors have read and agreed to the published version of the manuscript.

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