

Development of cube texture in multistage annealing of high purity aluminum foils^①

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Abstract: The development of cube texture ($\{001\} \langle 100 \rangle$) in high purity (99.99%) aluminum foils in multistage annealing was investigated by ODF and EBSD. It is found that a multistage annealing process can strengthen cube texture markedly, and that each stage of the multistage annealing plays an important role in nucleation and growth of the cube orientation grains. The cube orientation grain nucleates preferably at 180 °C because of its low activation energy of nucleation, the cube nuclei grow favorably at 400 °C because of the anisotropy of $40^\circ \langle 111 \rangle$ growth, and the cube texture is further strengthened at 550 °C and becomes predominant because of favorable bigger grain sizes.

Key words: high purity aluminum; multistage annealing; cube texture

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1 INTRODUCTION

The cube texture is important to high purity aluminum foils for high voltage capacitors because it can enlarge effective surface of aluminum foils and increase capacitance through channel etching. In order to increase the volume fraction of the cube component, a great deal of researches on the formation mechanism of cube texture have been done, and many methods were proposed, such as impurity controlling^[1,2], directional solidification^[3], preheating^[4], inhomogeneous rolling^[5], vacuum annealing^[6] and so on.

In this paper a multistage annealing process is proposed for strengthening the cube component. The development of the grain orientations in high purity aluminum foils in multistage annealing is investigated, so that the contribution of each stage annealing to cube texture can be determined obviously.

2 EXPERIMENTAL

High purity aluminum, with main impurities' chemical composition of Fe 7×10^{-6} – 10×10^{-6} , Si 7×10^{-6} – 10×10^{-6} and Cu 30×10^{-6} – 40×10^{-6} , was melted in an electric furnace. The ingots were preheated at 610 °C, scalped and hot rolled. The slabs were further cold rolled to the foils of 0.104 mm in thickness. The foils were vacuum annealed for 2 h in three groups: 1) at different temperatures 300, 400, 550 and 610 °C; 2) at different first-stage an-

nealing temperatures 180 °C, 2 h+ 550 °C, 2 h, 230 °C, 2 h+ 550 °C, 2 h; and 3) 180 °C, 2 h+ 400 °C, 2 h+ 550 °C, 2 h each and every. The textures of all the samples were determined by four incomplete pole figures ($5^\circ \leq \alpha \leq 85^\circ$) (111), (200), (220) and (113), and the orientation distribution functions (ODF) $f(g)$ were calculated according to the series expansion method^[11] with $l_{\max} = 22$. And all of them were ghost corrected by Gauss-type scattering functions according to the method of Luecke et al.^[12]. And microscopic texture investigations of the sample annealed at 180 °C were carried out by means of the TSL EBSD system attached to a H-3400 scanning electron microscope.

3 RESULTS

Fig. 1 shows the ODF of the samples annealed at different temperatures. It is showed that even though annealed at 300 °C for 6 h, the sample was not completely recrystallized and its rolling texture components (β -fiber texture) were retained. With enhanced temperature, the cube component increased (Fig. 2), while the R component (57° , 29° , 63°) $\{124\} \langle 211 \rangle$ decreased. Because it is not difficult for random components to nucleate and grow at higher temperature, the frequency of the formation of new random grains increases with annealing temperature. However, because of the higher growth rate of the cube component, it is much stronger and become predominant at high temperature.

Figs. 3 and 4 show the development of textures

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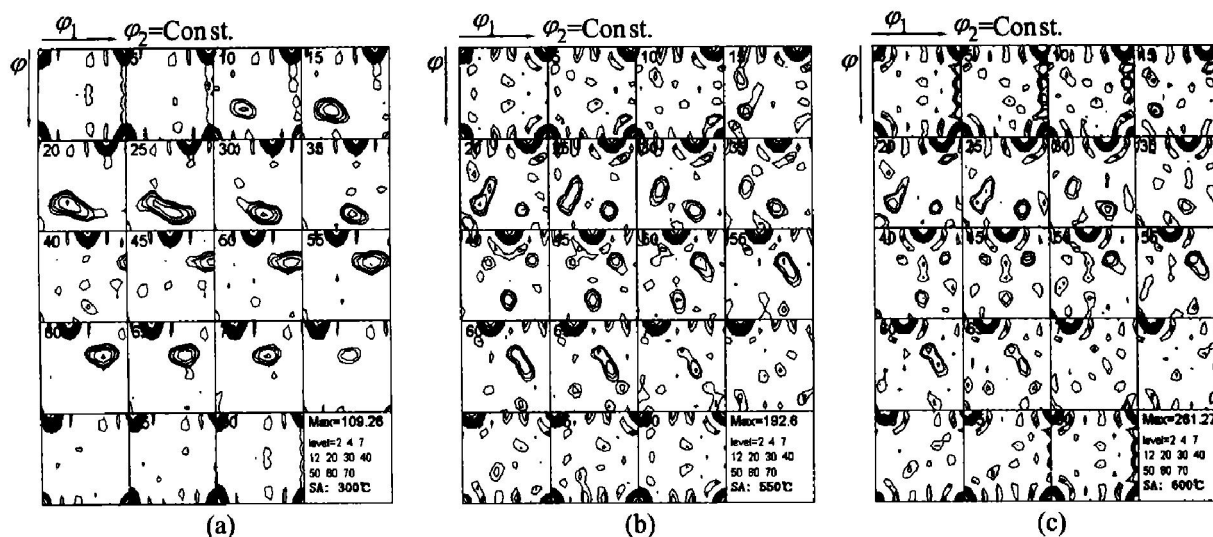


Fig. 1 ODFs of samples annealed at different temperatures for 2 h
(a) $-300\text{ }^{\circ}\text{C}$; (b) $-550\text{ }^{\circ}\text{C}$; (c) $-610\text{ }^{\circ}\text{C}$

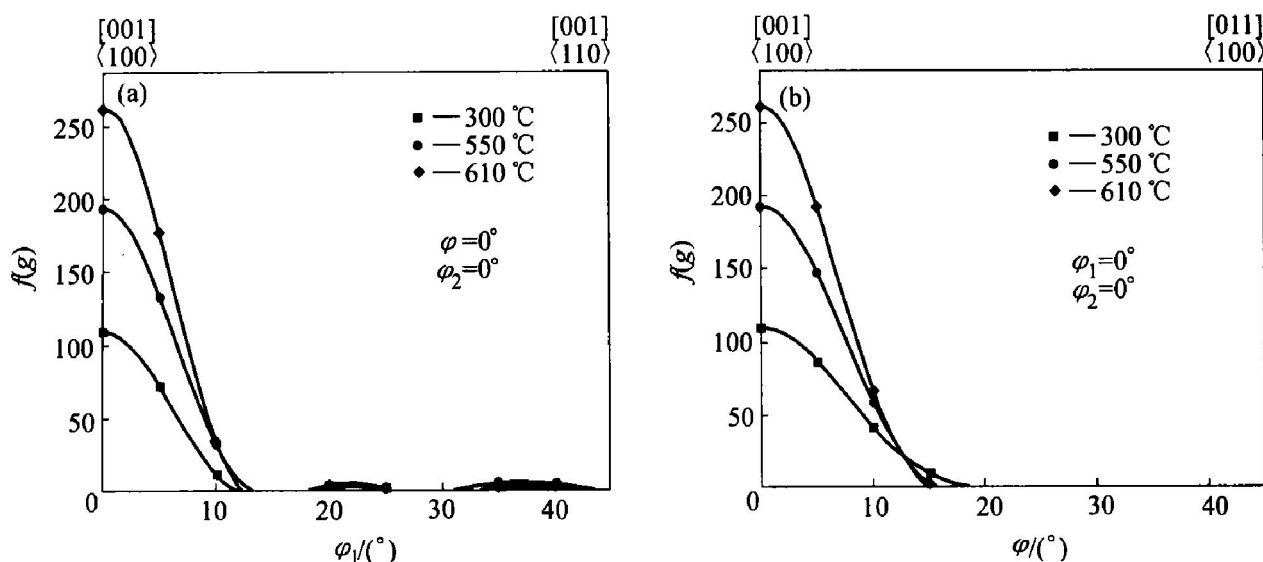


Fig. 2 ND- R_{cube} , RD- R_{cube} fibers of samples annealed at different temperatures
(a) ND- R_{cube} ; (b) RD- R_{cube}

in multistage annealing. The cube component increased with annealing stages. The Cu-type components have remained even through the second-stage ($400\text{ }^{\circ}\text{C}$, 2 h) annealing. And the scattering width of the cube texture after the second-stage annealing is slightly wider in φ_1 direction, while narrower in φ direction than that through the final one. So it can be said that through the second-stage of annealing the recrystallization has not been completed. Due to the low temperature annealing ($180\text{ }^{\circ}\text{C}$) the energy stored during rolling has been released partially so that the recrystallization proceeds very slowly and many random components have not energy enough to nucleate or grow, so the random components are much weaker through the second- and third-stage annealing.

Compared Fig. 1(b) and Fig. 5 with Fig. 3(c), the third stage annealing mode is the most effective step on strengthening cube recrystallization texture, i. e. the cube component in the sample treated with

the third stage annealing is the strongest in the three annealing modes. And the other random component is very weak in the sample.

The textures of the sample as-rolled and annealed at $180\text{ }^{\circ}\text{C}$ and $230\text{ }^{\circ}\text{C}$ are shown in Fig. 6 to elucidate the role of low-temperature annealing. In the as-rolled case the cube component cannot be observed, but it is much stronger for the sample annealed at $180\text{ }^{\circ}\text{C}$ than at $230\text{ }^{\circ}\text{C}$.

The orientation mappings of the samples annealed at $180\text{ }^{\circ}\text{C}$ and $230\text{ }^{\circ}\text{C}$ are shown in Fig. 7, where it can be seen that more cube nuclei formed at $180\text{ }^{\circ}\text{C}$ than at $230\text{ }^{\circ}\text{C}$.

4 DISCUSSION

Multistage annealing is a very useful process for strengthening cube texture, and each stage may play a different role.

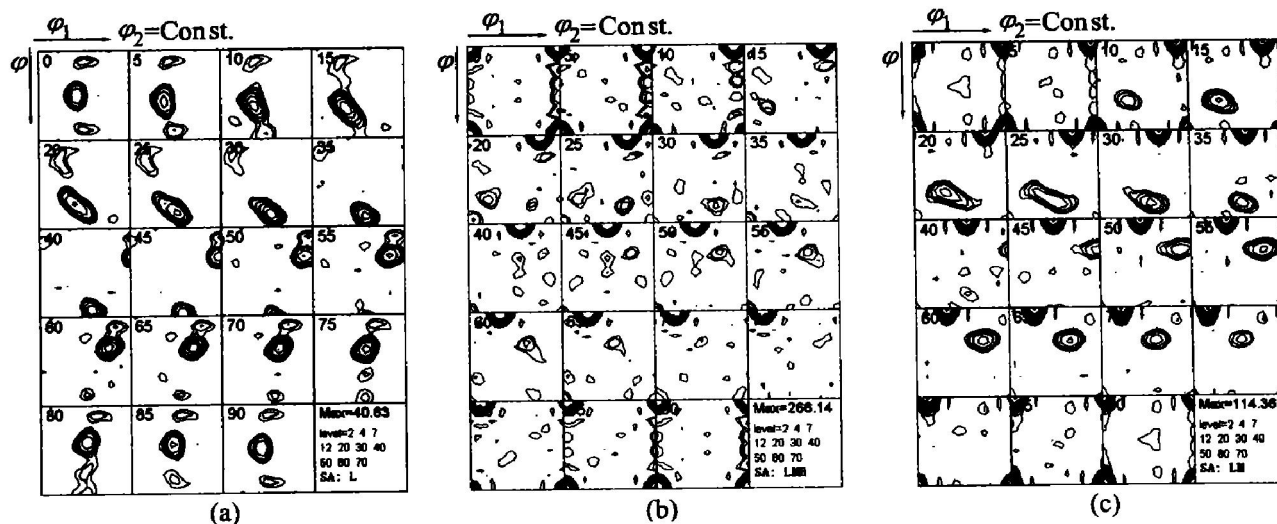


Fig. 3 ODFs of samples annealed in different stages

(a) $-180\text{ }^{\circ}\text{C}$, 2 h; (b) $-180\text{ }^{\circ}\text{C}$, 2 h + $400\text{ }^{\circ}\text{C}$, 2 h; (c) $-180\text{ }^{\circ}\text{C}$, 2 h + $400\text{ }^{\circ}\text{C}$, 2 h + $550\text{ }^{\circ}\text{C}$, 2 h

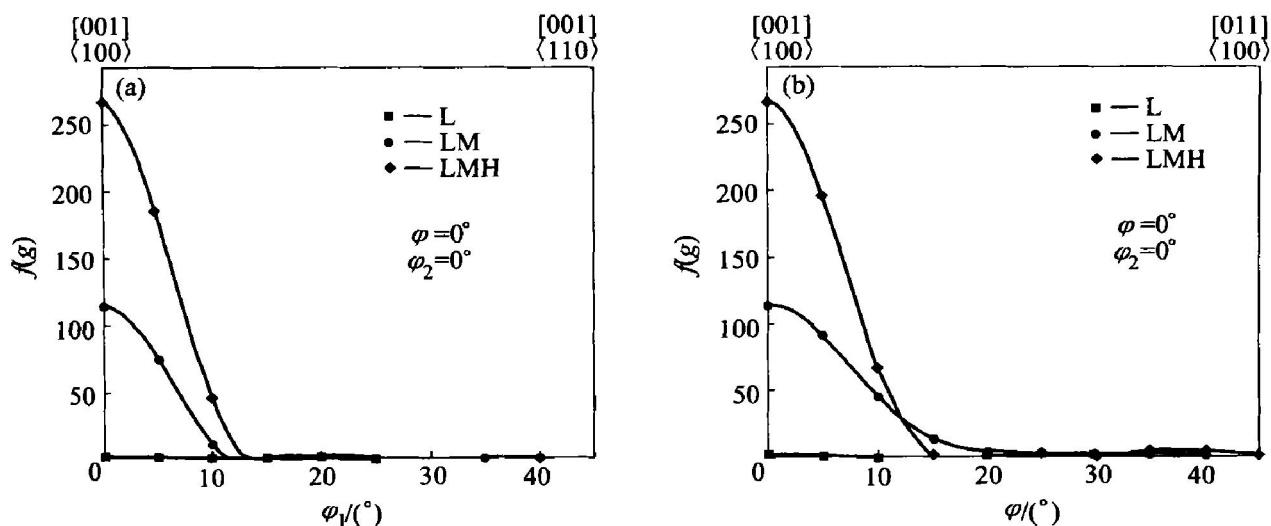


Fig. 4 ND-Rcube(a), RD-Rcube(b) fibers of samples in different annealed stages

(L $-180\text{ }^{\circ}\text{C}$, 2 h, LM $-180\text{ }^{\circ}\text{C}$, 2 h + $400\text{ }^{\circ}\text{C}$, 2 h; LMH $-180\text{ }^{\circ}\text{C}$, 2 h + $400\text{ }^{\circ}\text{C}$, 2 h + $550\text{ }^{\circ}\text{C}$, 2 h)

4.1 Low temperature stage

It is well known that temperature has an exponential effect on the cube component, and it is accepted that the stronger cube component can be obtained at high temperature. However although it is very weak at low temperature (e. g. $180\text{ }^{\circ}\text{C}$), the cube orientation grain can nucleate preferably. Fig. 6 shows that the strong cube component in the sample annealed at $180\text{ }^{\circ}\text{C}$ for 2 h is strong even though it can't be observed in the ODF. Fig. 7 shows a lot of fine cube grains with large angle boundaries. These high mobile grain boundaries will grow into the deformed matrix quickly during subsequent annealing. It is found that only cube orientation grain can be nucleated preferably at $180\text{ }^{\circ}\text{C}$ because of its lower nucleation activation energy.

The cube orientation is usually found in the shear bands^[5]. The heterogeneity of plastic deformation results in local stability of the cube orientation in transition bands^[7]. The cube orienta-

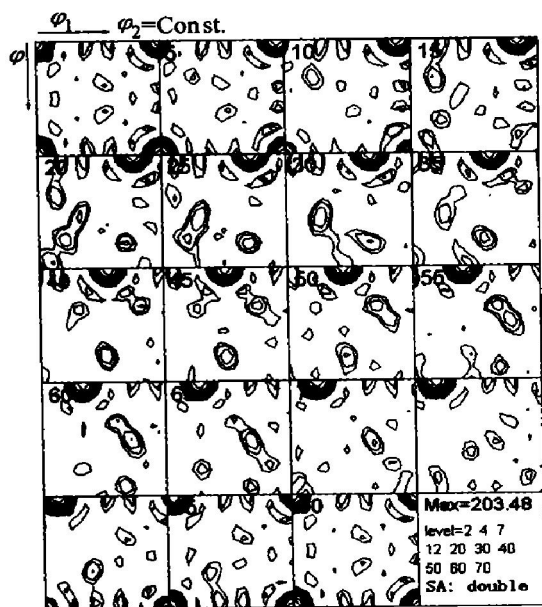


Fig. 5 ODF of samples through the second-stage annealing ($180\text{ }^{\circ}\text{C}$, 2 h + $550\text{ }^{\circ}\text{C}$, 2 h)

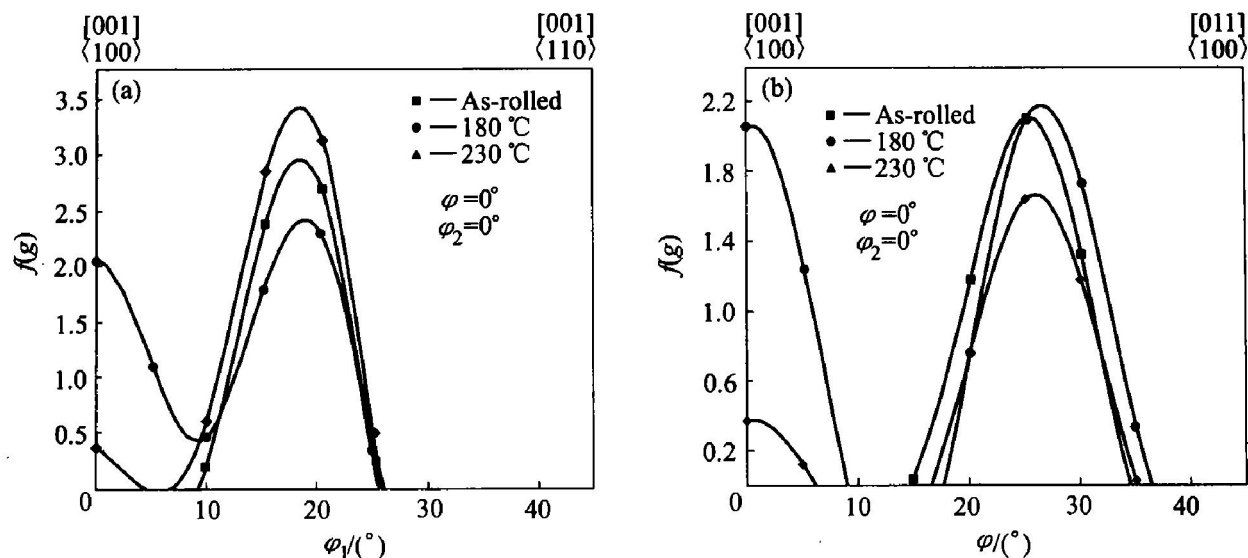


Fig. 6 ND- R_{cube} , RD- R_{cube} fibers of samples as-rolled and annealed at different low temperatures
(a) —ND- R_{cube} ; (b) —RD- R_{cube}

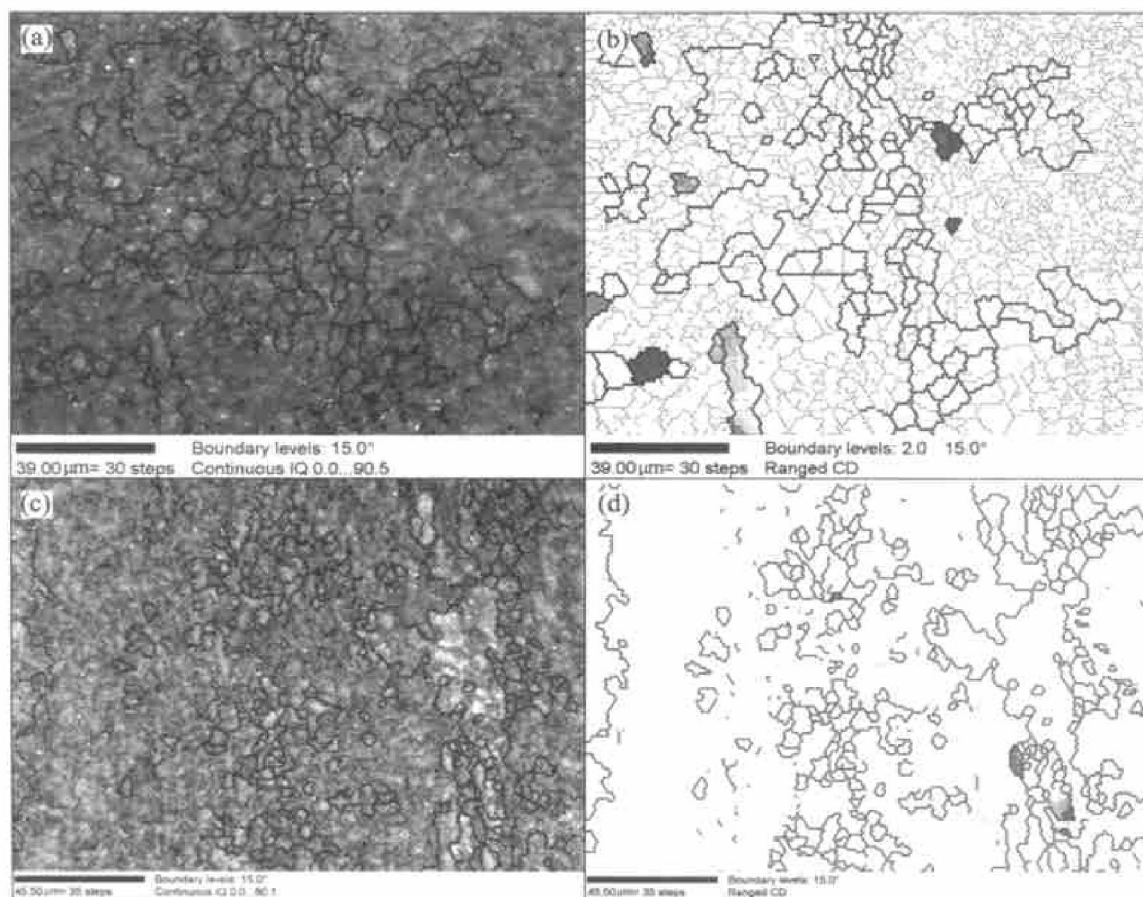


Fig. 7 Orientation mapping in foils annealed at 180 °C and 230 °C for 2 h
(a) and (c) —IQ (Image Quality) map where darker color refers to more severe distortion in lattice;
(b) and (d) —Orientation mapping (dark: cube orientation); (a) and (b) —180 °C, 2 h; (c) and (d) —230 °C, 2 h

tion is symmetric with respect to the principal strain axes in rolling, and metastable, might be expected to remain in the deformed structure as a transition band connecting the split parts of the cube. And a finite element simulation of plain strain compression of a polycrystal sample with several S-oriented grains and many elements per grain showed that transition bands

can form which contain near-cube oriented crystal-lites^[8].

Due to the low Taylor factor, the cube orientation at least tends to minimize the total shear strain and therefore the stored dislocation density. In addition, it was suggested^[9] that recovery should be relatively quick because of the lack of elastic interactions

between the active slip systems in the cube orientation. These types of cube subgrains can grow more easily and form cube nucleus during low-temperature annealing.

4.2 Middle temperature stage

It is believed that if the aluminum foils had no impurities, the special boundaries would have a higher migration enthalpy than random boundaries due to their lower energy^[10]. At lower impurity levels, the impurities would be segregated along special grain boundaries, washing out the anisotropy of migration. At high purity levels, because of the interaction between boundary and segregates, the anisotropy of migration does reoccupy. The boundaries of the cube grains can migrate faster than random boundaries because of $40^\circ \langle 111 \rangle$ rotation relationship with the major rolling components. Therefore, the cube orientation has nucleated at low temperature. During the middle-temperature annealing, the cube nuclei will grow into the rolled matrix and suppress the nucleation and growth of other orientation grain during the middle-temperature annealing.

In addition, a great deal of stored energy is released gradually. The migration rate of the boundaries is slowed down, and some orientation grains even cannot nucleate or grow up. As shown in Fig. 3(b), the rolling components are not completely consumed. The recrystallization proceeds slowly and has not been completed after middle-temperature annealing. Therefore, the middle-temperature annealing is favorable to development of the cube component as well.

4.3 High temperature stage

As shown in Fig. 3(c), the recrystallization has been completed, and several random components appeared, which are much weaker compared with those in Fig. 1(b), including R component. Because high temperature favors growth of cube component because of its large grain sizes and $40^\circ \langle 111 \rangle$ relationship to the deformed matrix as well, this stage can strengthen cube component further and accelerate the recrystallization.

5 SUMMARY

The multistage annealing benefits the development of cube component. At 180°C , the cube component will nucleate more easily because of its relatively quick recovery and lower nucleation activation energy. At 400°C the cube nuclei grow favorably because the impurity content on the special boundary is lower than that on the random boundary. At 550°C

the cube grains grow faster because of their larger size, and the $40^\circ \langle 111 \rangle$ relationship to the deformed matrix as well.

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