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Lattice Rotation and Recovery in Cube-oriented Grains in Partially Recrystallized Pure Aluminium after Rolling of Heavy Reduction

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Abstract

The mechanism of formation of sharp cube texture was investigated by transmission X-ray diffraction at the same point of specimens after various thermal and mechanical treatments. In cube oriented grains formed by partial annealing after heavy cold rolling, lattice rotation occurs mainly around transverse direction during light rolling. Upon slow heating, polygonization was observed in deformed cube grains. Polygonization was attributed to recovery in TD rotated cube regions. After formation of cube texture with orientational spread of around TD, grain growth takes place on final annealing at high temperature among TD- rotated cube grains, resulting in formation of sharp cube texture.

1. Introduction

To intensify cube texture, partial annealing and light rolling before final annealing are effective in high purity aluminium foil used for electrolytic capacitors [1]. However, the mechanism of sharp cube texture formation is still not yet clear [2]. To clarify the mechanism, lattice rotation and subsequent changes accompanying annealing treatment were investigated by the micro- Laue method. Preliminary study showed that the grain size of cube grain after partial annealing was so small that detection of asterism in the micro-Laue diffraction pattern was impossible. Therefore, cube grains were grown to larger size by the two or three step processes proposed by one of the authors [3,4].

2. Experimental Procedures

Starting materials were foils with 110 μm in thickness partially annealed at 250°C for 6 h and rolled 17% reduction after a prior cold rolling reduction of 98%. Hot rolled plate of 6mm thick was cold rolled to 132 μm before partial annealing. Contents of iron, silicon and copper are 16, 20, 40 ppm in mass%, respectively. The procedure of the micro- Laue camera method is as follows. First, the position of collimator with 0.3mm diameter was recorded in the optical microscope attached to flat cassette camera, and secondly, the specimens were placed, in the point, so that the progressing change at the same point in the specimen could be studied. The distance from specimen to film (Polaroid type 57) was 15mm. Unfiltered copper radiation accelerated at 30 kV was used. The Camera length and the centre of film were determined from the Debye rings due to the characteristic X-ray. X-ray exposure was carried out either at cube grains formed by annealing up to around

290℃ at heating rate of 20℃/h in order to catch the early stage of recrystallization and etched by anodic oxidation or at the markers made on an arbitrary point on the surface of foils with a marker pen. Annealing was carried out in air.

3. Results and Discussion

Figure 1 shows the softening curve at final annealing for partially annealed and lightly rolled foil after 98% reduction. According to microscopic observation, at around 290℃ growth of cube grains could be recognized and at complete recrystallization is nearly attained at 400℃. Further annealing caused slight decrease in hardness.

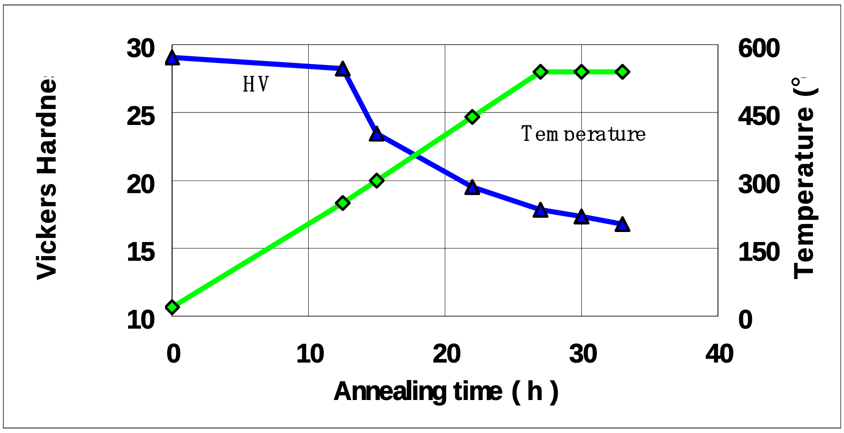


Figure 1: Softening curve at final annealing at heating rate of 20℃/h. Temperature changes are also plotted.

Figure 2 shows changes in the transmission X-ray photographs with progressing final annealing. As will be described later, under the present condition of X-ray diffraction, {311} Laue spots appear preferentially in the early stage of recrystallization more intense than spots with other indices, although the reason is not known. Judging from the fact that the Bragg angles are nearly equal for {111} K_β Debye and the {311} spots in the case of exact cube orientation, it is evident that annealing up to 310℃ caused preferential formation of TD-rotated cube grains.

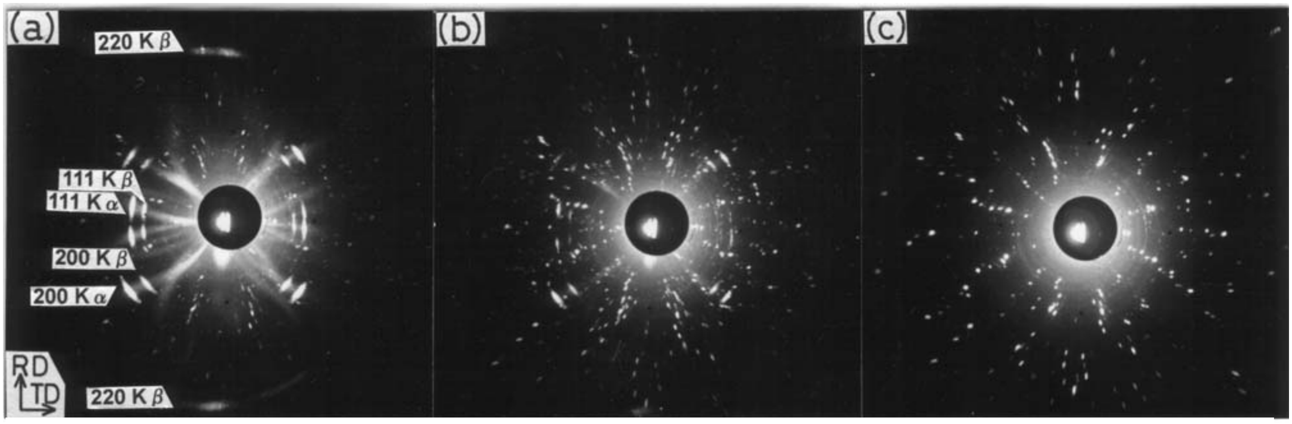


Figure 2: Transmission X-ray photographs obtained at the same point (a) after interruption of final annealing at 310℃, (b) at 320℃ and (c) after final annealing at 540℃.

At 320℃, the intensity of Debye arcs decreased, implying that un-recrystallised regions were consumed by TD-rotated cube grains. After final annealing up to 540℃, the number of Laue spots decreased and intensity of spots became intense, suggesting that grain growth occurred among TD-rotated cube grains.

Stereographic projection showing orientation of cube grains in Figure 2 is shown in Figure 3. Decrease of number of grains exposed under X-ray is from approximately 10 at 320℃ to 6 at 540℃ for {311} spots. It is known that sharp cube texture is controlled by additional growth selection effects before complete recrystallization by competition with the deformed matrix with S-orientation [5,6]. It is also known that even after the completion of recrystallization, the cube texture becomes sharp during grain growth on further annealing [7,8]. The present results show that sharpening during grain growth occurs with consumption of TD-rotated cube grains by more exact cube grains formed at an early stage of recrystallization even after complete recrystallization.

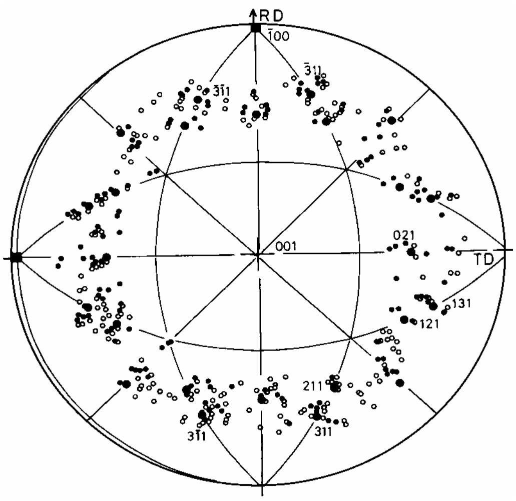


Figure 3: Stereographic projection showing the orientation of cube grains in Figure 2. Large solid circles indicate the cube orientation determined from the set of most intense spots in Figure 2(c). Poles of lattice planes caused Laue spots are also indicated by solid and open small circles for 540 and 320℃, respectively. Small circles are grouping with equivalent indices, but all of them could not be discriminated.

○ 320 ℃

● 540 ℃

Figure 4 shows the microstructure after additional light rolling. At the point indicated by symbol +, transmission X-ray diffraction patterns were taken with progressing annealing. Obtained results are shown in Figure 5. Asterisms are faintly observable in Figure 5(a) as indicated by arrows, suggesting that lattice rotation around TD took place during light rolling. Since detection of asterism is restricted by various factors [10], it is possible that actual lattice rotation is more large than that observed in Figure 5(a). Upon annealing up to 300℃, Debye arcs due to diffraction at un-recrystallized region disappeared, indicating complete recrystallization was attained. Grain growth among TD-rotated cube grains was more pronounced after slight deformation than in the one step process (Figure 2).

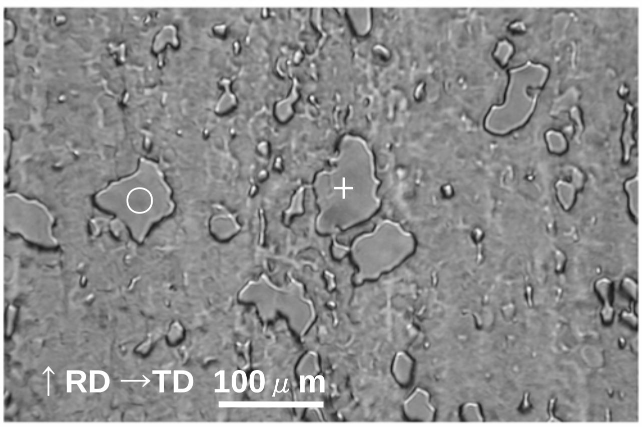


Figure 4: Optical micrograph obtained after second additional rolling. Symbol **+** indicates the point at which X-ray beam struck the specimen during exposure. Specimen was treated by Barker method after electrical polishing. Similar results to Figure5 were obtained at the point indicated by open circle, as reported elsewhere [9].

Figure 6 shows stereographic projection showing orientation of cube grains observed in Figure 5(d). Judging from the number of {311} spots, at least six cube grains survived in the exposed area during final annealing, resulting in sharp cube texture formation.

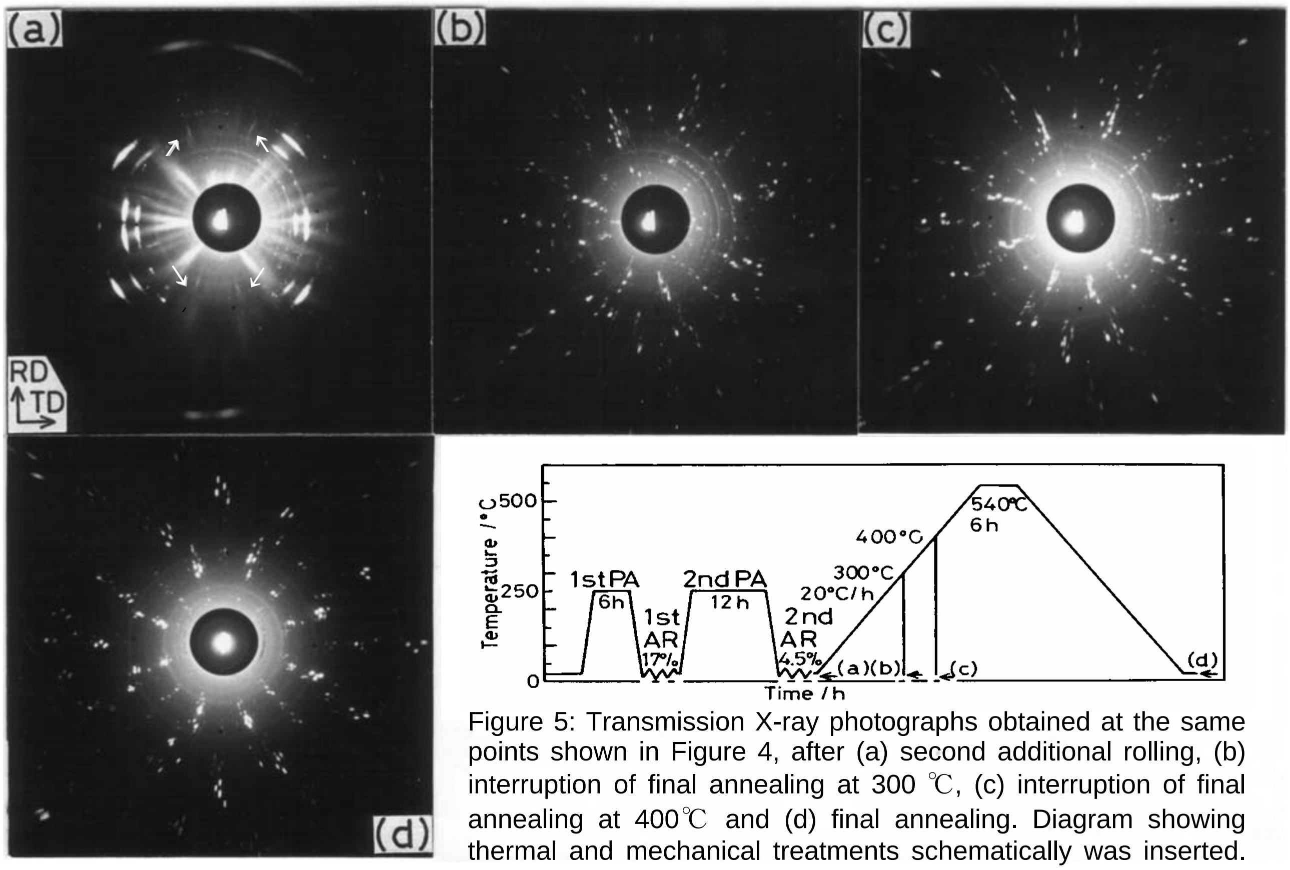


Figure 5: Transmission X-ray photographs obtained at the same points shown in Figure 4, after (a) second additional rolling, (b) interruption of final annealing at 300 ℃, (c) interruption of final annealing at 400℃ and (d) final annealing. Diagram showing thermal and mechanical treatments schematically was inserted. White arrows in (a) indicate asterisms of 311 spots.

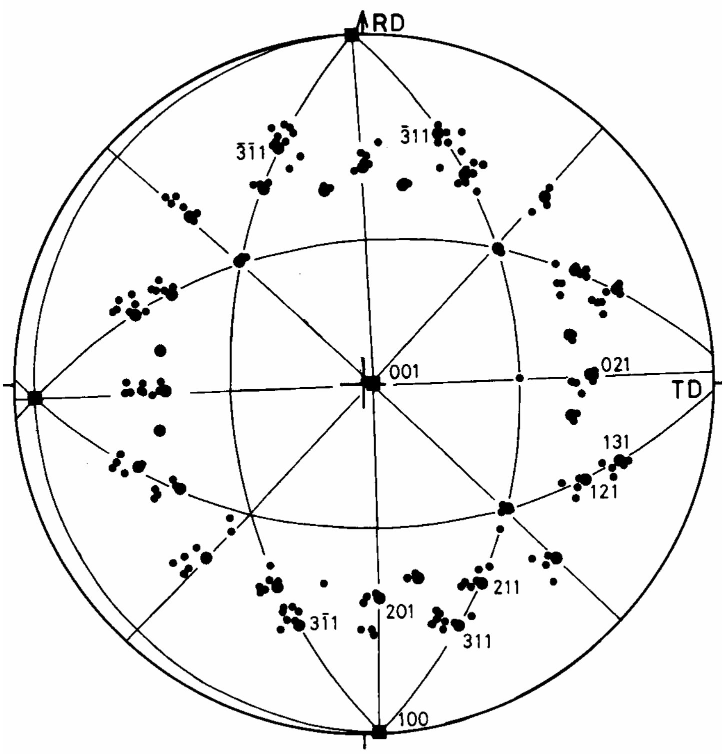
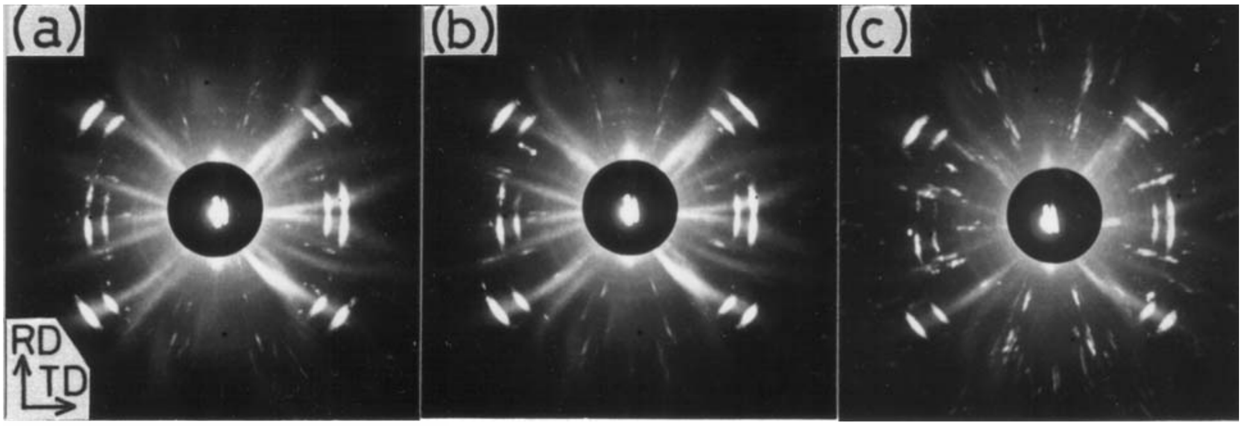


Figure 6: Stereographic projection showing the orientation of cube grains in Figure 5(d). Large solid circles indicate the cube orientation determined from the set of most intense spots in Figure 5 (d). Poles of lattice planes caused Laue spots are also indicated by solid small circles. Small circles are grouping with equivalent indices, but all of them could not be discriminated.

Figure 7 shows transmission X-ray diffraction patterns with progressing treatment from third partial annealing to final annealing at the same point in the specimen. After third partial annealing, two TD rotated cube grains of which orientations are shown in Figure 8 were observed. By this three step process, the symmetry in Laue pattern of cube orientation with regard to RD became pronounced.

As mentioned above, the Bragg angles are almost equal for {111} Debye ring due to Kβ radiation and the {311} Laue spot in the case of exact cube orientation. Therefore, it is possible to determine whether detected grains are TD-rotated cube grain or not on the basis of the relative position of {111} Kβ ring and {311} spots.

Upon third light rolling, asterisms were observable as in Figure 7(b). After annealing up to 300℃, asterisms became spotty and more clear and intense than after third light rolling, and continuous background decreased whereas the exposure times were equal. This implies that in cube grains recovery (polygonization) took place. Further annealing resulted in sharp cube texture formation.



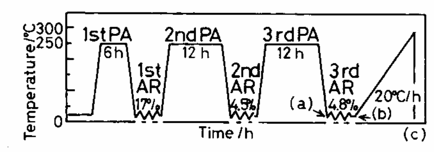


Figure 7: Transmission X-ray photographs obtained at the same point after (a) third partial annealing, (b) third additional rolling and (c) interruption of final annealing at 300℃. Schematic diagram showing thermal and mechanical treatments was inserted.

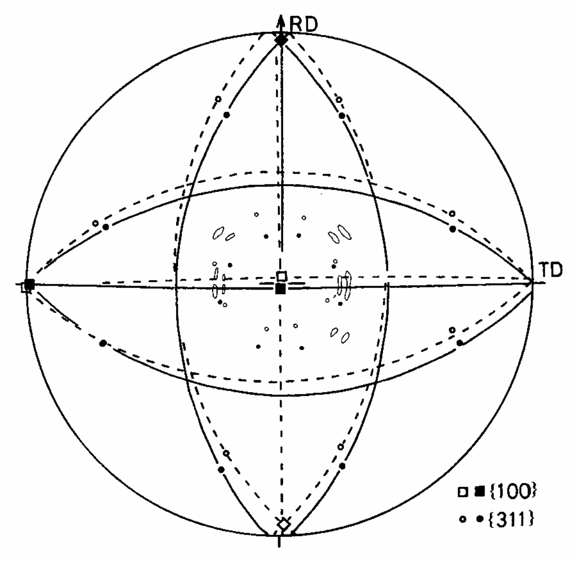


Figure 8: Stereographic projection showing orientations of cube grains in Figure 7(a). Two TD-rotated cube orientations were determined from the position of {311} poles. {110} great circles could be determined because {311} poles belong to <110> zone axis.

It is well known that at the early stage of plane strain compression or rolling of single crystal in the cube orientation lattice rotation occurs predominantly around TD in opposite direction, i.e. the one direction towards the exact cube orientation and the other away from cube orientation [11-14]. It was shown theoretically [15] that relaxing normal-rolling shear component can produce rotation toward the exact cube orientation. Therefore, it is suspected that the roles of an additional light rolling played in the special process for sharp cube texture [1] are not only to increase the driving force in the un-recrystallized region for growth, which decreased during partial annealing, but also to rotate cube orientation close to the exact cube orientation.

In the present study, it was shown that in cube grains imbedded in the un-recrystallized region lattice rotation takes place mainly around TD and TD-rotated cube grains are formed by subsequent final annealing. Cube grains formed at the early stage with close to the exact cube orientation begin grain growth at the expense of TD rotated cube grains at the late stage of final annealing at high temperature, resulting in sharp cube texture formation.

4. Summary

In cube grains embedded in partially recrystallised structures, lattice rotation occurs predominantly around TD in opposite direction in accordance with that in single crystal with cube orientation subjected to rolling or plane strain compression. Since one of the direction is directed close to the exact cube orientation, it is suspected that one of the roles of additional rolling is to form the lattice region, which serve as the origin of cube texture. Upon annealing, polygonization takes place in the cube grains and the recovered cube grains begin growth at the expense of surrounding un-recrystallized region, resulting in the formation of cube texture with orientational spread around TD after complete recrystallization. On further annealing at slow heating rate to high temperature, the cube texture sharpens through grain growth among TD-rotated cube grains.

Acknowledgments

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