MEMOIRS OF THE SCHOOL OF ENGINEERING, OKAYAMA UNIVERSITY Vol. 6, No. 1, SEPTEMBER 1971

Deformation and Prefered Orientation of Precipitates in Cold Worked Al-Zn Alloys

Mutsuo OHTA,* Fumio HASHIMOTO** and Hironobu MAEDA*

* *Department of Industrial Science*
** *Department of Electronics*

(Received April 30, 1971)

Fiber textures of drawn wires of Al-Zn alloys containing precipitates were studied mainly by X-ray methods. In supersaturated solid solution the wire texture was a double fiber texture with [100] and [111]. It was considered that α phase precipitates were rotated with matrix, while they were deformed. And then the matrix containing stable precipitates (Zn) had [100] and [111] textures. The intense spots, corresponding to [111] fiber texture of matrix, in the Debye rings of $(002)_p$ and $(101)_p$ planes of precipitates were clearly observed. But, on the other hand, spots, corresponding to [100] fiber texture of matrix, shown the prefered orientation of precipitates in $(002)_p$ Debye ring were very weak and brodening.

§ 1. Introduction

The various precipitates are formed on ageing Al-Zn alloys. Hirosawa[1] had investigated fiber textures of extruded aluminium alloy rod by X-ray methods. Prefered orientation of precipitates in drawn Al alloy wire, however, was scarcely reported.

Al-Zn alloy is known as one of age-hardenable aluminium alloy. The precipitation process in Al-Zn alloy is summaried as follow;

Supersaturated Solid Solution $\alpha \rightarrow$ Spherical G. P. zones $+ \alpha \rightarrow$ elliptic G. P. zones $+ \alpha \rightarrow$ Rhombohedral transition phase $+ \alpha \rightarrow$ cubic transition phase $+ \alpha \rightarrow$ Zinc-rich stable precipitates $+ \alpha$.

In this work, fiber textures of supersaturated solid solution, and alloys containing α phase or Zn-rich stable precipitates were studied.

§ 2. Experimental Procedure

Specimens were made from 99.996% pure aluminium and 99.999% pure zinc. Each alloy was melted in the high alumina crucible in the air. The nominal atomic compositions of alloys used in this study were Al-6.8at%Zn and Al-10at%Zn. Ingots of 15mm in diameter were forged at about 400°C to 5mm thick sheets, and then cold rolled to the thickness of 2.0mm with intermediate annealing at about 450°C. Strips for drawing were cut down from these sheets. The specimens for X-ray small angle scattering method were rolled to the thickness of about 0.05mm.

Specimens were cooled in electric furnace or quenched into iced water after homogenized at 400°C for 1∼5 hours. Specimen containing supersaturated solid solution was immediately drawn after quenching, and specimen containing α' cubic transition phase was annealed at 125°C for 25 hours after quenching, and then was drawn. To obtain hexagonal zinc precipitates, the specimen was cooled in electric furance. Deformation rate was 75∼96% in reduction of area by wire drawing.

After these treatments, specimens were examined by metallurgical microscope and X-ray method with monocromated Cuk_α radiation.

Since effective thickness for diffraction in case of X-ray used is about 0.1mm from surface, specimen was electropolished less than one-third in diameter to obtain the X-ray photography of central part of specimen.

§ 3. Result and Discussion

Photo. 1 shows the fiber texture of Al-6.8at%Zn alloy that is quenched and subsequently drawn with reductions in cross-sectional area of 91 per cent. The wire textures are [100] texture in addition to same [111] texture as pure Al. Besides, diffraction spots of this alloy is too broding along the Debye incomparison with pure Al drawn same rate.

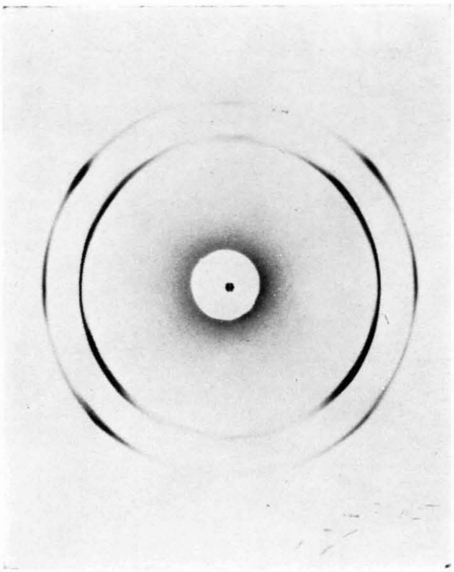


Photo. 1 Diffraction pattern of wire of Al-6.8at% Zn alloy after quenching from 400°C into ice water.

Photo. 2 is powder photograph of Al-6.8at%Zn alloy that is annealed at 125°C for 25 hours after quenching from 400°C. The lines correspond to phase is detected outside each line of matrix because of lattice constant of phase smaller than that of matrix.

Small angle scattening of X-ray from Al-6.8 at%Zn alloy aged at 125°C for 25 hours after quenching from 400°C into ice water is shown in **Photo. 3.** Photo. 3 (a) is undrawn specimen. That is not shown the usual halo due to spherical G. P. zone, but uneven halo. This may be considerable that the shape of cluster in matrix is not spherical, but rod shape. Considering the heat treatment carried out, the scattering body may be crystallite of α' phase. The X-ray small angle scattering photography of Al-6.8at%Zn alloy applied the same heat treatment and cold rolled with reduction in thickness of 90% is shown in photo. 3 (b). Arrow shows direction of rolling. The region of scattering angle is considerably narrower than one of not rolled sample. This corresponds to elogation of crystallite along rolling direction, that is to say, it may be deduced that intermediate α' phases are given the deformation when working a sample.

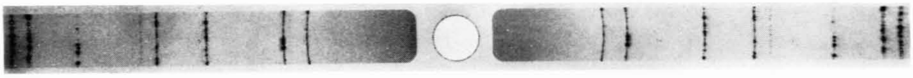


Photo. 2 Powder diffraction pattern of Al-6.8atZn alloy annealed at 125°C for 25 hours after quenching from 400°C into ice water.

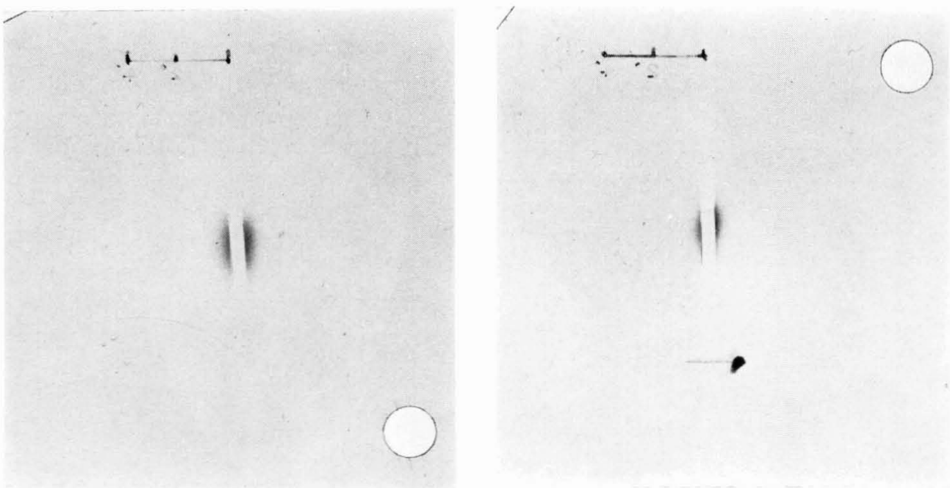


Photo. 3 Small angle scattering of X-ray from Al-6.8at%Zn alloy aged for 25 hour at 125°C after quenching from 400°C into ice water. (Arrow shows the rolling direction)

Photo. 4 shows Debye photograph of drawn wire of alloy containing α' phase. On the outside of maximum scattering intensities on Debye rings of $(111)_M$ and $(200)_M$ planes, the spot from α' phase are detected, but not observed at somewhere else. From this fact, it is considered that deformation of α' phases are undergone in the same way of that of matrix, while α' phases are deformed themselves when deforming the specimen.

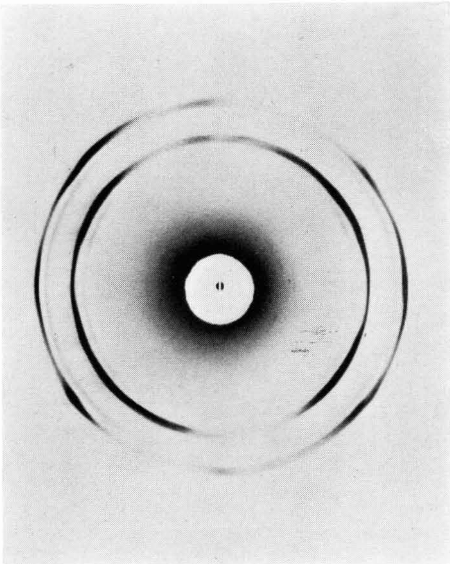


Photo. 4 Diffraction pattern of drown wire of Al-6.8at%Zn alloy aged for 25hr. at 125°C after quenching from 400°C into ice water.

Photo. 5 shows the photograph of metallurgical microscope with drown wire of alloy containing precipitates (Zn). This is shown that the precipitate particles are assembled in large quantities at exterior side than interior side of wire. And the state of distribution of precipitate particles is cell structure.

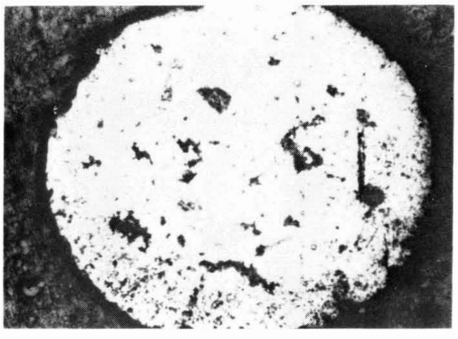


Photo. 5 Photograph of metallugical microscope of drawn wire of Al-10at%Zn Containing precipitates (Zn). ×500

Figure 1 illustrates the pattern that is obtained with monochromatic X-ray from a cold-drawn wire of Al-Zn alloy containing precipitates, assuming that the deformation of precipitate particles is undergone in the same way to that of matrix worked. The black spots represent [111] fiber texture of matrix and a white dot represents [100] fiber texture of matrix. The thick lines and thin lines represent Debye rings of matrix and precipitates, respectively. There are some crystallographic relations between solid solution and precipitates, that is, plates on $\{111\}_M$ $(111)_M /\!/ (001)_p$, $\langle 110\rangle_M /\!/ \langle 110\rangle_p$[2]. Consequently, Fig. 1 describes where locate ideal poles for $(111)_M$, $(200)_M$, $(101)_p$ and $(002)_p$ planes in a wire of Al-Zn alloy having two [111], [100] fiber textures assuming the precipitates deformed with attaching themselves matrix.

The spost of fiber texture of precipitates lie at the same angle to matrix in the Debye rings, since $(002)_p$ planes are parallel to $(111)_M$. Namely, there are some pairs of intense spots on the inside of two Debye rings, $(002)_p$ and $(111)_M$ planes. On the other hand, some spots on the outer Debye rings represent the pole figure of drawing textures for $(200)_M$ and $(101)_p$ planes. In this case, there appeare the spots in not pair but separately because of $(101)_p$ being not parallel to $(002)_M$.

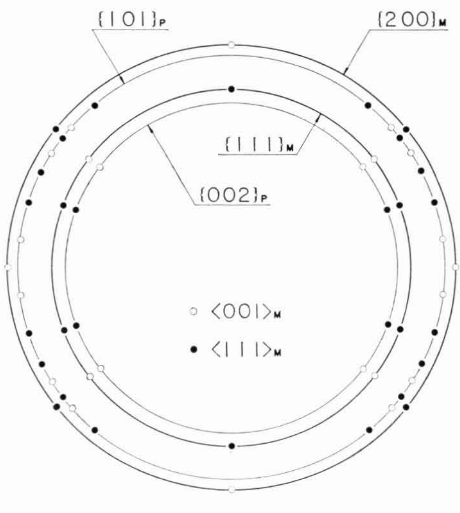
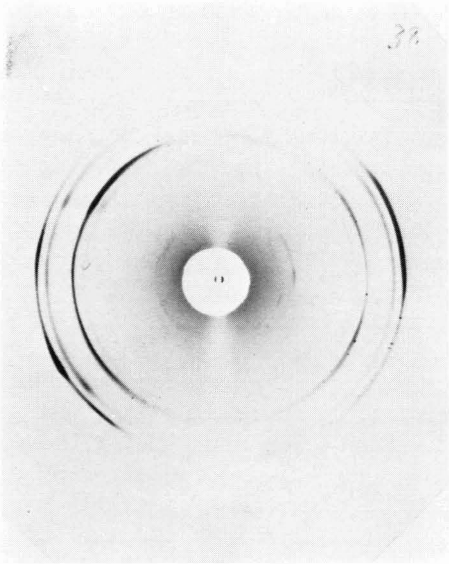


Fig. 1 Pattern of drowing Al-Zn wire Radiation of a single wavelength incident perpendicularto the wire axis, which is vertical.


(a)

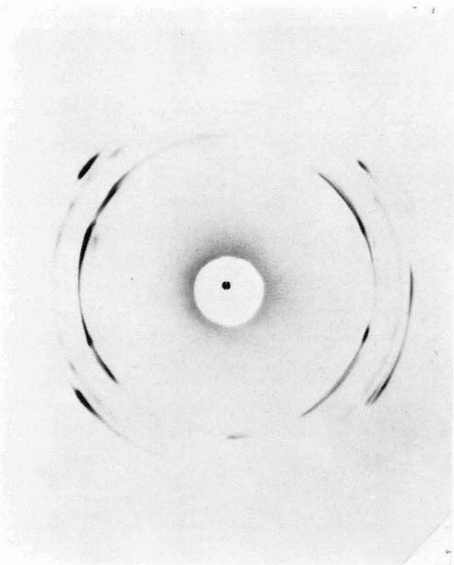

(b)

Photo. 6 Photographs of cross section of drown Al-10at%Zn alloy cooled from 400°C to room tempe rature at rate of 12°C/hr.

The Debye photograph of drawn wire of Al-10at%Zn alloy which is cooled from 400°C to room temperature at a rate of 12°C/hr. after holding at 400°C for 1 hour and drawn with reduction in cross-sectional area of 75 per cent, is shown in **Photo. 6.** Photo. 6 (a) representes the diffraction pattern from the near surface of specimen because of wire being about 0.7 mm in diameter, and (b) is X-ray photograph from central part of same specimen electropolished less than 0.3mm in diameter. Photo. 6 (a) shows that the wire texture for matrix is a double fiber texture lacking in clearness, in particulary, the spots corresponding to [100] fiber axis broaden fairly and the spots correaponding to fiber texture of precipitates are also faint. In the central part of same specimen (photo. 6 (b)), intense spots equivalent for fiber axis are clearly observed. [100] fiiber axis on the Debye ring of $(002)_p$ plane, however, is weak and brodening. And there recognize only four spots on the ring of $(101)_p$ planes. For parent phase, the spots corresponding to the both fiber texture, [100] and [111] are appreciated obviously.

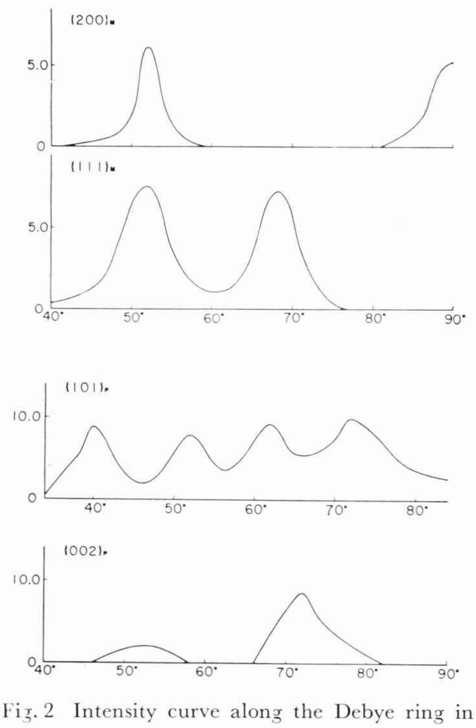


Fig. 2 Intensity curve along the Debye ring in photo. 6 (b).

Fig. 2 shows the intensity along the Debye ring in photo. 6 (b). Namely, blackness along Debye rings are measured by photometer and reduced to intensity from by standard intensity scale. The spot located at 52° 34′ is corresponding to [100] drawing texture. This spot is seemed very weak in intensity and brodening, on the other hand, intensity of spot of [111] fiber axis is considerably strong.

In the $(101)_p$ plane, four peaks of intensity is all corresponding to [111] fiber axis, and peaks corresponding to [100] fiber axis is not recognized. But, if it appear, then there should be located at 48° 40′, 57° 14′ and 82° 13′. At those place, the intensity is not zero as shown in $(002)_p$ diffraction ring, so that the peak corresponding to [100] fiber texture may be masked.

§ 4. Summary

The experimental results and discussion obtained by X-ray method and observation of metallugical microscope may be summarized as follows:

(1) In supersaturated solid solution fiber axes were [100] texture in addition to same [111] texture as in pure Al.

(2) It was considered that α' phase were rotated with matrix while deforming themselves.

(3) Precipitates were moved in attaching to matrix, and then they showed the apperent fiber texture. Namely, prefered orientation was the same one of matrix.

(4) The part of sureface: the fiber textures of matrix were [111] and [100] axes. According to darkness of diffraction spots, [111] axis component seemed stronger than [100] axis component. The intense spots on the Debye rings of $(002)_p$ and $(101)_p$ planes of precipitates were clearly observed with equivalent for [111] fiber texture of matrix, but, on the other hand, the spots equivalent for [100] fiiber axis of matrix were less than [111] fiber axis.

(5) The central part: the fiber texture of matrix was same with one of surface part and each spot corresponding to fiber texture was more clear. On the diffraction rings of $(002)_p$ and $(101)_p$ planes of precipitates, the spots corresponding to [100] axis were observed, in addition to the spots due to crystallographic relation between matrix and precipitates. And diffraction pattern of precipitates resembled to one of matrix, namely, there were fairly clear prefered orientation in precipitates.

(6) With respect to microstructure of cros-section of drawn wire after furnace cooling, the precipitate particles were assemabled in large quntities at exterior side than interior part of wire.

Reference

1) H. Hirosawa: J. Japan Inst. Metals, **2** (1962), 95.

2) A. H. Geisler, C. S. Barrett and R. F. Mehl: Trans. AIME Vol. 152, p201 1943.