Electron-Microscopic Investigation on Precipitation Phenomena in Aluminium-Zinc Alloys

By

Yotaro MURAKAMI*, Osamu KAWANO*
and Hideaki TAMURA*

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The precipitation process, its relation with lattice defects and the effect of cold working on precipitation and grain boundary reaction in the aluminium alloys containing 11.2, 17.4, 28.3 and 40.8% zinc respectively were investigated by means of X-ray diffraction, hardness measurements and transmission electron microscopy. It was found that face-centered cubic transition precipitates were formed in granular shape in 11.2% or 17.4% zinc alloys and were plate-like in 28.3% zinc alloys. The plate-like transition precipitates were formed on {110} matrix planes, perhaps preferentially on the prismatic dislocation loops. The results are shown to be consistent with the hypothesis that in the quenched alloys precipitation occurs on prismatic dislocation loops that result from the condensation of quenched-in vacancies. In Al-28.3 and 40.8% Zn alloys, the grain boundary reaction is predominant at lower aging temperatures.

1. Introduction

The Al-Zn system has an extended solid solubility of zinc in aluminium for a wide range of temperature. Especially on account of high solubility at higher temperature as shown in Fig. 1, a great deal of research works has been done on the precipitation.

Recently the studies on the resistivity changes during pre-precipitation of Al-Zn alloys have been carried out in relation to the excess vacancies retained in quenching[1,2,3]. On the other hand, R. D. Garwood et al.[4] have clarified the structure and orientation relationship of the precipitates in Al-25% Zn alloys.

It was confirmed that the sequence of structural changes during the isothermal decomposition of an aluminium-25% zinc alloy at 200°C involved at least three stages: clustering of solute atoms $\rightarrow$ face-centered cubic transition precipitates $\rightarrow$ equilibrium zinc precipitates.

In this work, experiments have been carried out to examine the aging

* Department of Metallurgy

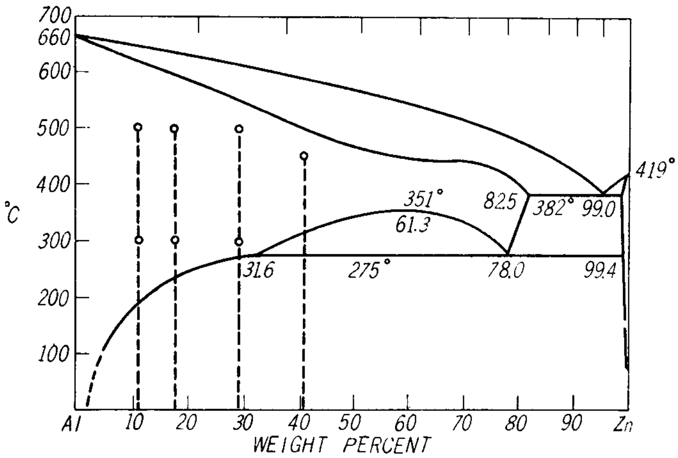

Fig. 1. The phase equilibria of the aluminium-zinc system.

characteristics at various temperature after suitable solution-treatment, the effects of quenching temperature or cold working on precipitation and grain boundary reaction in the aluminium alloys containing 11.2, 17.4, 28.3 and 40.8% zinc respectively by means of the X-ray Laue method, Norelco geiger counter diffraction method, measurements of micro-hardness and transmission electron microscopy. Especially the correlation between precipitation process and lattice defects was examined with transmission electron microscopy.

2. Experimental Procedure

Preparation of Alloys:

The Al-Zn alloys in these experiments were melted by using 99.99% aluminium in a graphite crucible lined with alumina in an atmosphere of argon gas, then were cast into a chill mould after adding zinc of 99.99% purity. Ingots of alloys weighing about 500 g were obtained. The compositions of the alloys and temperatures of the solution treatments are shown in Table I.

Table 1. Compositions of the Aluminium Alloys and Temperatures of the Solution Treatment.

wt. pct. of Zn	Solution Treatment Temperature °C
11.2	500
17.4	500
28.3	500
40.8	450

The ingots were cut into 0.9 mm thickness for X-ray Laue method and measurements of micro-hardness. The specimens for electron microscopic studies were rolled into sheets of 60 μ thick. The specimens were solution-treated for 3 hr at selected temperatures as shown in Table I and then quenched into iced brine. Furthermore, in order to examine the effects of the quenching temperatures, the specimens except Al-40.8%Zn alloys were solution-treated for 3 hr at 500°C, and then were quenched into the salt bath held at 300°C and kept for 30 min at this temperature and finally were quenched into iced brine. The aging characteristics of Al-Zn alloys were studied by tempering at 70°, 110°, 150°, 200° and 250°C.

The large-grain specimens for the Laue method were prepared by the strain-anneal method. The specimens for the study of the effect of cold working by electron-microscopy were cold rolled into sheets of 40 μ thick from the sheets of 60 μ thick above mentioned.

Micro-Vickers Hardness Measurements:

The micro-Vickers hardness was measured on the surface of the sheet specimens by using a "Durimet" micro-Vickers hardness tester under a load of 200 g. The hardness number was taken as the average of four fairly equal values.

X-ray Laue Method:

The Laue method is generally recommended for a rapid survey of the aging process. The large grain specimens of Al-Zn alloys were examined mainly with [100] axis of the matrix parallel to the X-ray beam. Satisfactory photographs were obtained by molybdenum radiation with exposures of 2 hr at 40 mA, 35 KV using a collimeter 5.5 cm in length and 0.5 mm bore with a specimen to film distance of 3.5 cm.

Electron-micrographs:

The structural changes produced by aging have been investigated by examining foils of these alloys by an electron microscope. The foils were prepared by controlled electropolishing and chemical dissolution of the remaining oxide film by the method devised by R. B. Nicholson et al[5]. A small section was cut with scissors from the thinnest parts of the foil and mounted in a microscope specimen holder between two meshes bored at their centers. An examination was carried cut with a Shimazu-SM-D4 type electron microscope operated at 75 KV.

Norelco geiger counter X-ray diffraction method:

The powder of the Al-Zn alloys was aged isothermally in thin quartz tubes 1.5 mm dia. under an atmosphere of argon gas. The structure of precipitates was examined by copper radiation with a Norelco geiger counter diffractometer,

3. Experimental Results

1) Precipitation process

Figs. 2 and 3 show the Debye-Scherrer X-ray diffraction patterns of aged Al-11.2%, 17.4%, 28.3% and 40.8% Zn alloys. In these figures H.Q. means water-quenching from 500°C or 450°C and L.Q. from 300°C. The thick solid lines show aluminium solid solution, the faint solid lines show equilibrium zinc precipitates and the dotted lines show a face-centered cubic transition phase having a lattice constant of 3.990Å. In Fig. 2, the face-centered cubic transition

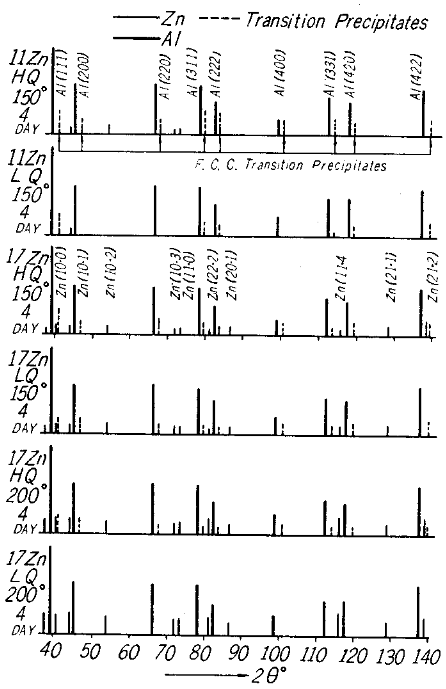

Fig. 2. Debye-Scherrer X-ray diffraction patterns of aged Al-11.2% or 17.4% Zn alloys.

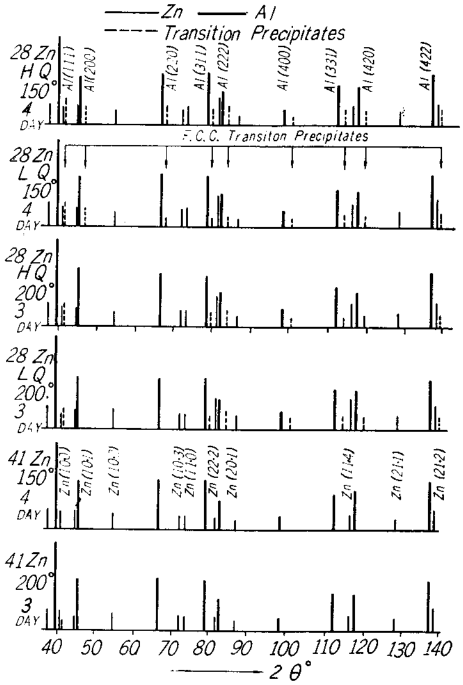

Fig. 3. Debye-Scherrer X-ray diffraction patterns of aged Al-28.3% or 40.8% Zn alloys.

phase is detected more weakly in the specimen quenched from 300°C than in that quenched from 500°C in Al-11.2% or 17.4% Zn alloys. Especially in isothermal aging for 4 days at 200°C the transition phase is not detected in the specimen quenched from 300°C in Al-17.4% Zn alloys. It may be confirmed from these results that the formation of a transition phase is closely connected with lattice defects introduced by quenching. However, in Al-28.3% or 40.8% Zn alloys, the effects of quenching temperatures are not remarkable as shown in Fig. 3. In Al-40.8% Zn alloys, a transition phase is scarcely detected and grain boundary reaction occurs widely in isothermal aging for relatively short time at 150°C.

These results are in accordance with those of the studies by R. D. Garwood et al[4] who showed that a face-centered cubic transition phase had been detected in the Deby-Scherrer X-ray diffraction photographs of an aluminium-25% zinc alloys during isothermal aging at 200°C.

Hardness measurements were made immediately after aging with a micro-Vickers hardness tester. Fig. 4 shows hardness changes of Al-11.2%, 17.4%

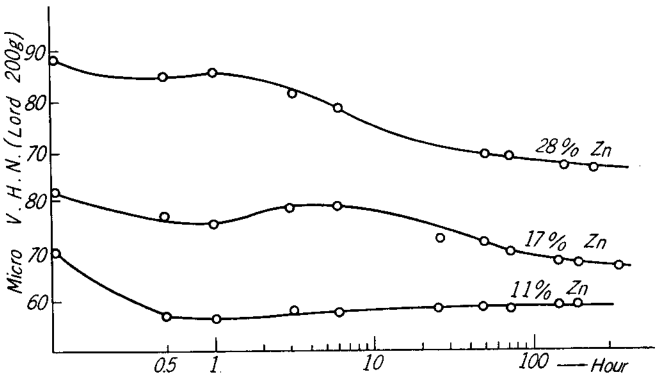

Fig. 4. Hardness changes of Al-Zn alloys aged at 150°C.

and 28.3% Zn alloys aged at 150°C. Hardness drops in initial stage of aging at 150°C may be due to either the reversion of spherical zones which already exist as water quenched state or a full growth of zones. The following increase of hardness curves is clearly due to the formation of transition precipitates. Fig. 5 shows the hardness changes of Al-28.3% Zn alloys as a function of aging temperature. From these hardness changes, the higher the aging temperature, the greater is the initial hardness drop and the sooner is the hardening due to the formation of transition precipitates.

Photos 1 and 2 show the Laue photographs of Al-11.2% Zn alloys aged at 150°C for 4 or 10 days respectively, taken by X-ray beams parallel to the [100] axis of the matrix. In these photographs, the diffuse scattering which appears near the {113} matrix spots shown by the arrows is thought to be due to a transition phase. Such a diffuse scattering is not observed in the specimens immediately after water-quenching. Laue patterns in Al-17.4% Zn alloys aged for 2 days at 200°C as shown in Photo. 3 show many streaks and spots as indicated by the arrows. According to the stereographic projection, it is found that these streaks are due to plate-like precipitates on $\{110\}_{Al}$ and

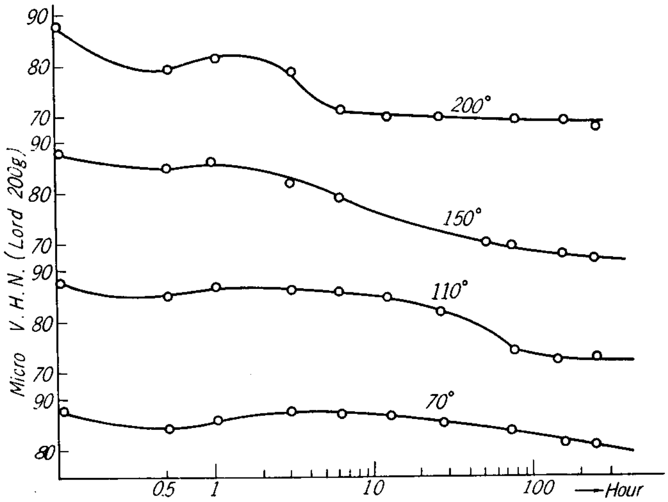

Fig. 5. Hardness changes of Al–28.3% Zn alloys as a function of aging temperature.

$\{111\}_{Al}$ matrix. These spots are thought to be due to transition precipitates and equilibrium zinc precipitates.

Photos 4 and 5 are the Laue patterns of Al–28.3% Zn alloy aged for 3 hr at 150°C or 200°C respectively. In Photo 4, the diffuse scattering and the streaks as shown in Photo 3 appear faintly. It is clear that they are due to face-centered cubic transition phase and that as aging progresses in Al–28.3% Zn alloys, transition precipitates grow up to plate-like shape in these Laue photographs. In Photo 5, the streaks as shown in Photo 3 and the additional streaks are observed as indicated by the arrow. The latter streaks follow zone circles through {111} matrix poles in the stereographic projection. It has been shown previously that such streaks may be caused by the formation of equilibrium zinc precipitates on $\{111\}_{Al}$ matrix planes.

Photo 6 is the Laue photograph of Al–40.8% Zn alloys aged for 4 days at 200°C. In the initial stage of aging at 200°C, the diffuse scattering thought to be due to a transition phase are observed in the single crystal patterns. On further aging, the Laue photograph shows the pattern similar to a polycrystal structure by the grain boundary reaction which occurs at a much faster rate as shown in Photo 6.

Photo 7 shows an electron micrograph of Al–11.2% Zn alloy thin foil after being aged for 17 days at 110°C. In this photograph, preferential precipitates of a face-centered cubic transition phase seem to occur along the dislocation and many circular faint spots which are thought to correspond to the grown zones are also clearly observed. These zones seem not to have any relation with dislocations. On longer aging at higher temperatures, many precipitates of α' transition phases occuring along dislocations are observed.

As for Al–17.4% Zn alloys, in isothermal aging for 7 days at 70°C, any precipitation is not clearly observed in the electron micrographs. Even in the electron micrograph after being aged for 7 days at 110°C, small amount of α' transition precipitates only are observed. However, after 2 days aging at 150°C, an electron micrograph of thin foils of Al–17.4% Zn alloys shows that the precipitates α' transition phase spreads widely over the grains, showing preferential precipitation along grain boundaries at *D* and precipitate-free regions adjacent to boundaries as shown in photo 8. Such precipitate-free regions adjacent to boundaries are called "denuded zones". In these regions the formation of the precipitates is hindered, because grain boundaries are sinks for the vacancies introduced by quenching and therefore these regions have much lower density of such defects. And the matrix and grain boundaries are densely populated with precipitates[6] by the formation of precipitate-free regions adjacent to grain boundaries as shown in photo 8. And many helical or zigzag dislocations and dislocation loops observed in photo 8 eventually become preferred sites for precipitation.

On aging at the temperatures above 200°C, electron micrographs of Al–17.4% Zn show many platelike precipitates with the hexagonal structure of zinc. In Al–11.2% Zn and 17.4% Zn alloys, the grain boundary reaction is not observed by the electron microscopic studies.

As to Al–28.3% Zn alloys, grain boundary reaction is predominant at lower aging temperatures than 150°C. But above about 200°C, the continuous precipitation is observed. Photo 9 shows the discontinuous precipitations in the electron micrograph of a thin foil of Al–28.3% Zn alloy aged for 1 day at 70°C. The large lameller precipitates seems to be easily soluble during electropolishing. Photo 10 shows an electron micrograph of Al–28.3% Zn alloy thin foil after being aged for 30 min at 150°C. In photo 10, dark regions along the grain boundary are thought to be the early stage of grain boundary reactions. In the early stage of grain boundary reaction, grain boundaries may be densely populated with zinc atoms by grain boundary diffusion.

On further aging, grain boundary reaction becomes remarkable. Examples of these are shown in photos 11 and 12. Photos 11 and 12 are the electron-micrographs of thin foils of Al–28.3% Zn alloys aged for 1 hr or 6 hr at 150°C respectively. In photo 12, the small circular spots are thought to be α'

transition precipitates and the lamellar precipitates by grain boundary reaction are thought to be equillibrium precipitates of the zinc phase.

Photo 13 shows the grain boundary reaction-free regions in an electron micrograph of thin foil of Al–28.3% Zn alloy aged for 3 hr at 150°C after water-quenching from 500°C. In the photograph, many dislocation loops that occurred widely within the grains seem to have some relation with the α' transition precipitate. A great number of plate-like precipitates crossed at right angles are observed instead of dislocation loops around P in this photograph. It may be true that the prismatic dislocations caused by the condensation of vacancies on a {111} plane[7] provide suitable sites for nucleation of α' transition precipitate in Al–28.3% Zn alloys.

In isothermal aging for 1 hr or 3 hr at 250°C, electron micrographs in Al–28.3% Zn alloys show the preferred precipitation of the transition phase at the dislocation lines and grain boundaries in photos 14 and 15 respectively. However, in photos 14 and 15, it is clear that the discontinuous precipitation does not occur at such a higher aging temperature.

Photo 16 is an electron micrograph of thin foil of Al–28.3% Zn alloy after being aged for 6 hr at 250°C. In this photograph, equilibrium zinc precipitates along the grain boundaries and in the grain are observed and in addition to them many transition precipitates are found within the grains.

Photos 17 and 18 are the electron micrographs of thin foils of Al–40.8% Zn alloys aged for 3 hr at 150°C or for 1 hr at 250°C respectively. The discontinuous precipitation is predominant at lower aging temperatures, but above about 200°C, the grain boundary reaction is not observed. In photo 18, large equilibrium zinc precipitates are observed.

As for the effects of quenching temperatures, on isothermal aging of Al–28.3% Zn alloys water-quenched from 500°C, the small precipitates spread widely as shown in photo 15. On the other hand, in an electron micrograph of thin foil of Al–28.3% Zn alloys aged for 1 hr at 250°C after quenching from 300°C, precipitates of a large size thought to be due to equilibrium zinc precipitates are observed as shown in photo 19. At any rate, the formation of the transition precipitates tends to be hindered in the specimen quenched from lower temperatures. This phenomena is clearly in accordance with the results of the Debye-Scherrer X-ray diffraction methods. However, the effects of quenching temperatures on the grain boundary reaction are not distinguished remarkably.

2) Effect of plastic deformation on precipitation and grain boundary reaction

Photos 20 and 21 are the electron micrographs of thin foil of 30% rolled

Al-28.3%Zn alloys aged for 1 day at 70°C or for 2 days at 110°C respectively. In these photographs, the grain boundary reaction is clearly observed in the non-recrystallized matrix. The precipitates appear not plate- or lamella-like but very granular in form.

Photo 22 is an electron micrograph of thin foil of Al-17.4%Zn alloy 70% rolled immediately after water-quenching from 500°C and then aged for 3 hr at 250°C. This photograph shows the situation when the whole area of the specimen was recrystallized and the large size precipitates are found along the grain boundaries and within the new grains. The grain boundary reaction does not occur in this specimen.

The structure of the 70% rolled Al-28.3%Zn alloys is shown in photo 23.

Photo 24 to 29 are the electron micrographs of thin foils prepared from Al-28.3%Zn alloys aged for various periods at 70°, 110°, 200° and 250°C in 70% rolled conditions. It seems to be certain that plastic deformation after water quenching increases the number of precipitates and that the grain boundary reaction is not remarkable before the appearance of the recrystallized new grains. After being aged for 1 hr at 70°C precipitates along rolled structures are found clearly as shown in photo 24.

The precipitation on the grain boundaries or within the new grains is illustrated in the 70% rolled specimen aged for 1 hr at 110°C as in photo 25. In this photograph, many dislocation lines are observed. Photos 26 and 27 are the electron micrographs of 70% rolled specimens aged for 11 hr or 3 days at 200°C respectively. In these photographs, recrystallization being brought to completion, new grains are formed and the precipitates exist not only along the grain boundaries but also within the new grains. These electron micrographs show that plastic deformation decreases the size and increases the number of the precipitates. It may be clear that the disappearance of coarse precipitates within the grains can be attributed to a large increase in the number of nucleation sites as a result of plastic deformation. In photos 26 and 27, the discontinuous precipitation which often starts at new grain boundaries in the specimen without deformation is not observed to occur.

Both photos 28 and 29 are the electron micrographs of thin foils of 70% rolled Al-28.3%Zn alloy aged for 3 hr or 3 days at 250°C respectively. In photo 28, the grain boundary reaction is clearly observed at its first stage and a greater number of precipitates are found within the recrystallized new grains.

The large lamellar precipitates caused by the grain boundary reaction is observed at *L* in photo 29. In this way, the grain boundary reaction is thought to become remarkable after the full recrystallization of the matrix.

4. Discussion

On the basis of the present studies, the sequence of structural changes during the aging of Al–Zn alloys involves at least three stages: clustering of solute atoms → α' transition precipitates → equilibrium zinc precipitates. These results are clearly in accordance with the studies by G. D. Garwood et al[4]. The effects of quenching temperatures on precipitation are clearly remarkable in Al–11.2% or 17.4% Zn alloys. In the specimens water-quenched from 300°C, the formation of α' transition precipitates is hindered in comparison with the specimens water-quenched from 500°C. It may be clear that the precipitation of the transition phase is closely connected with lattice defects introduced by quenching.

In Al–28.3Zn alloys, prismatic dislocation caused by the condensation of vacancies on a $\{111\}_{Al}$ plane are thought to provide suitable sites for nucleations of plate-like α' transition precipitates as shown in photo 13.

In Al–28.3% or 40.8% Zn alloys, the grain boundary reaction is predominant at 70°C∼150°C aging temperatures. In this condition, the grain boundary reaction is hindered by 70% rolling after water-quenching. It is uncertain why the grain boundary reaction is hindered by heavy plastic deformation after water-quenching, but it is clear that as the matrix is fully recrystallized the grain boundary reaction begins to occur rapidly at higher aging temperatures than 150°C. And it is general that the lower the aging temperature, the longer the time, and the higher the zinc content, the greater the lamellar precipitation is.

In Al–Zn alloys, the formation of α' transition precipitates is accelerated by plastic deformation after water-quenching. The electron micrographs show that plastic deformation decreases the size and increases the number of the precipitates. It may be thought that dislocation introduced by plastic deformation provide suitable sites for nucleation of precipitates.

5. Conclusion

The precipitation phenomena during aging at various temperatures, the effects of cold working on precipitation and grain boundary reaction in the aluminium alloys containing 11.2, 17.4, 28.3 and 40.8% Zn respectively were studied mainly with the X-ray Laue method, Norelco geiger counter diffraction method, and hardness measurements and electron microscopic observation with thin foils. It may be concluded that:

1) The possible precipitation sequence in Al–Zn alloy in accordance with studies of R. D. Garwood et al[4] can be written as: clustering of solute atoms

→ face-centered cubic α' transition precipitates → equilibrium zinc precipitates.
2) The formation of α' transition precipitates on $\{110\}_{Al}$ planes is hindered by quenching from lower temperatures and accelerated by plastic deformation after water-quenching.
3) In Al–11.2% or 17.4% Zn alloys, α' transition phases are clearly found to precipitate preferentially along dislocation lines.
4) In Al–28.3% Zn alloys, prismatic loops caused by the condensation of vacancies provide favorable sites for nucleation of plate-like α' transition precipitates on $\{110\}_{Al}$ planes.
5) The final stage of the precipitation process in Al–Zn alloys in the formation of plate-like equilibrium zinc precipitates on $\{111\}_{Al}$ planes with the hexagonal structure.
6) In Al–28.3% Zn or 40.8% Zn alloys, the grain boundary reaction is predominant at lower aging temperatures such as 70° to 150°C. The grain boundary reaction is hindered by heavy plastic deformation after water-quenching, but when the matrix is fully recrystallized at higher temperature this reaction begins to occur rapidly.

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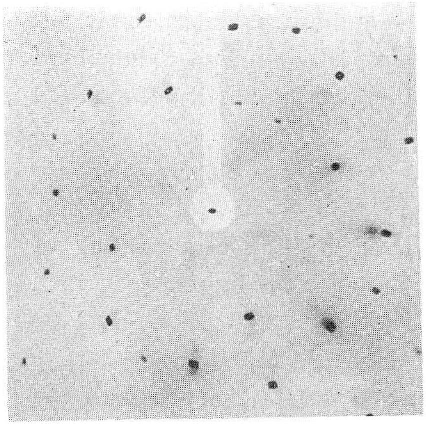

Photo. 1. Al-11.2% Zn alloy, W.Q., aged for 4 days at 150°C. Beam ⊥ (100)

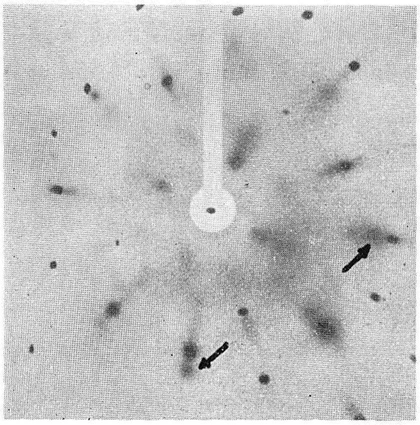

Photo. 2. Al-11.2% Zn alloy, W.Q., aged for 10 days at 150°C. Beam ⊥ (100)

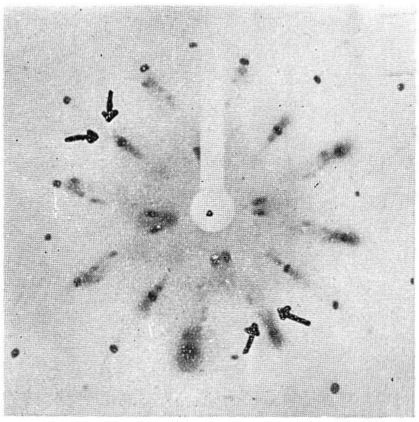

Photo. 3. Al-17.4% Zn alloy, W.Q., aged for 2 days at 200°C. Beam ⊥ (100)

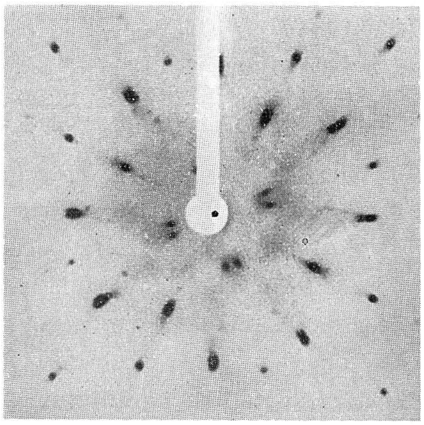

Photo. 4. Al-28.3% Zn alloy, W.Q., aged for 3 hr at 150°C. Beam ⊥ (100)

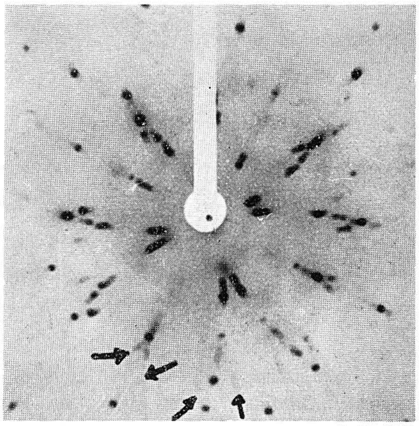

Photo. 5. Al-28.3% Zn alloy, W.Q., aged for 3 hr at 200°C. Beam ⊥ (100)

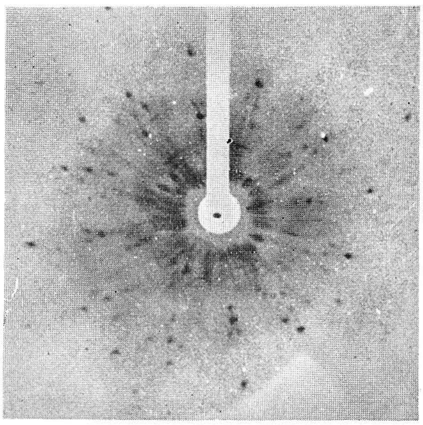

Photo. 6. Al-40.8% Zn alloy, W.Q., aged for 4 days at 200°C. Beam ⊥ (100)

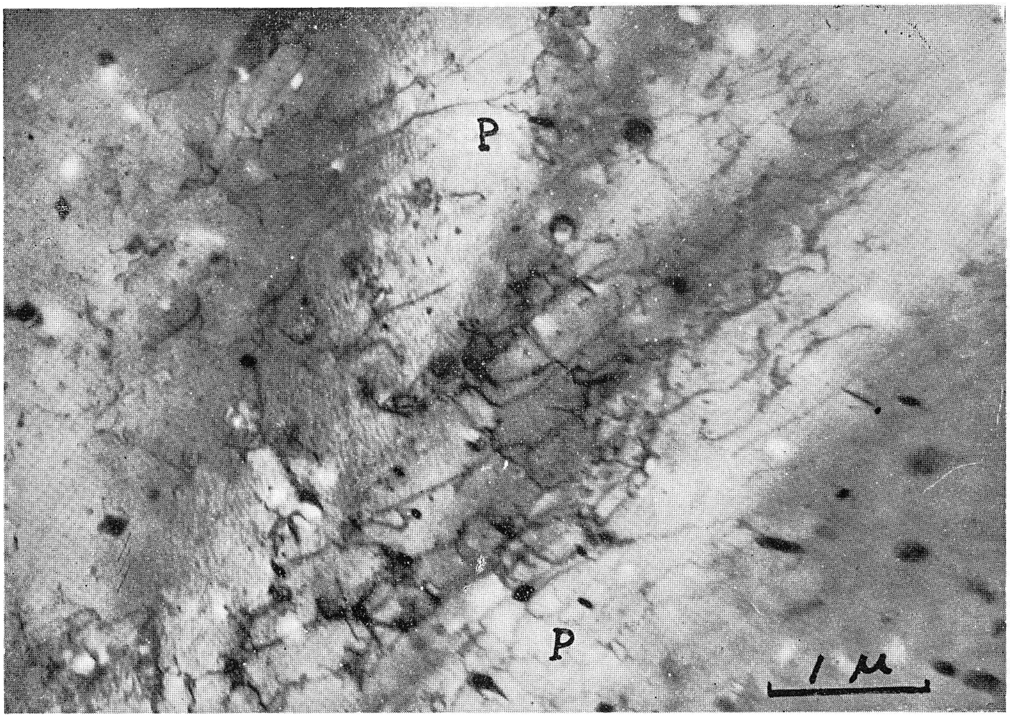

Photo. 7. Al–11.2% Zn alloy, W.Q., aged for 17 days at 110°C. Dislocation available for nucleation sites of transition precipitates at P. ×21,000.

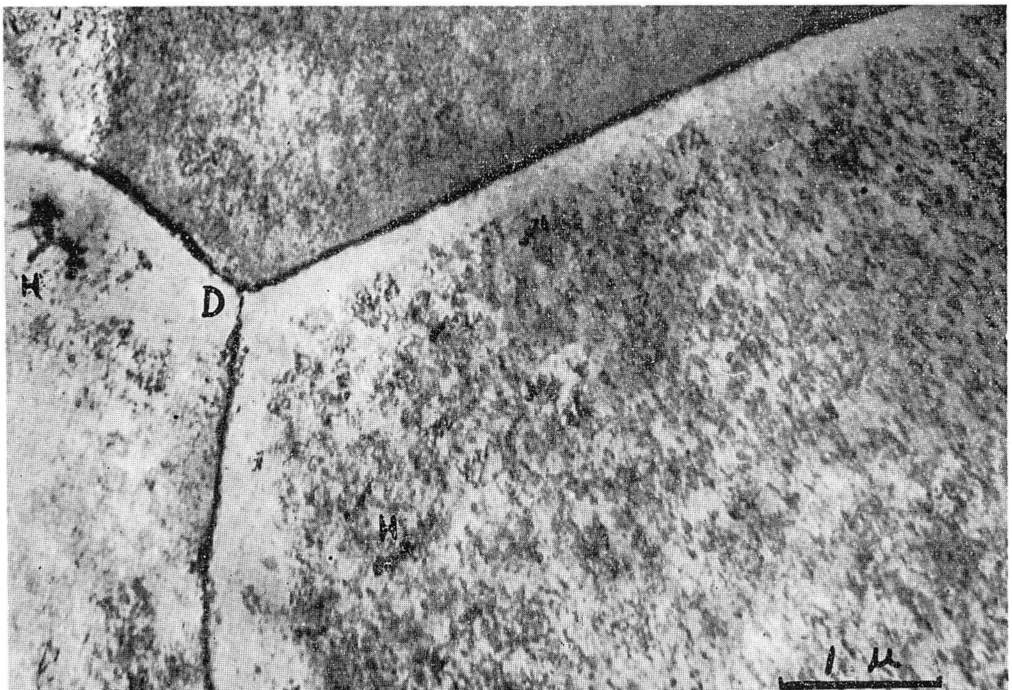

Photo. 8. Al–17.4% Zn alloy, W.Q., aged for 2 days at 150°C. Helical or zigzag dislocation decorated with precipitates at H. Showing precipitate-free regions adjacent to boundaries. ×21,000.

Photo. 9. Al-28.3% Zn alloy, W.Q., aged for 1 day at 70°C. Showing the large lamellar precipitates by grain boundary reaction at D. ×21,000.

Photo. 11. Al-28.3% Zn alloy, W.Q., aged for 1 hr at 150°C. ×21,000.

Photo. 10. Al-28.3% Zn alloy, W.Q., aged for 30 min at 150°C. Dark regions along the boundary is thought to be the first stage of grain boundary reaction. ×21,000.

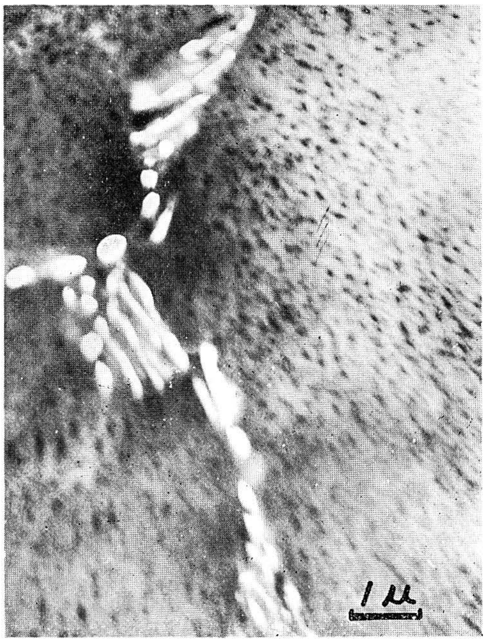

Photo. 12. Al-28.3% Zn alloy, W.Q., aged for 6 hr at 150°C. ×9,000.

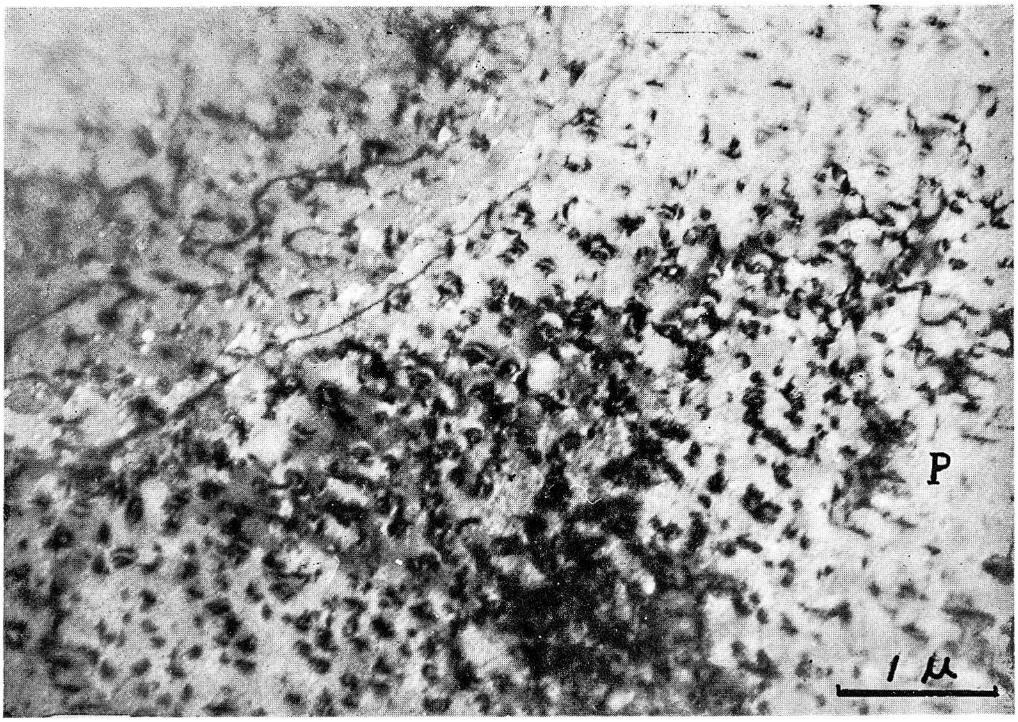

Photo. 13. Al–28.3% Zn alloy, W.Q., aged for 3 hr at 150°C. Prismatic dislocation loops available for nucleation sites of transition precipitates at P. ×21,000

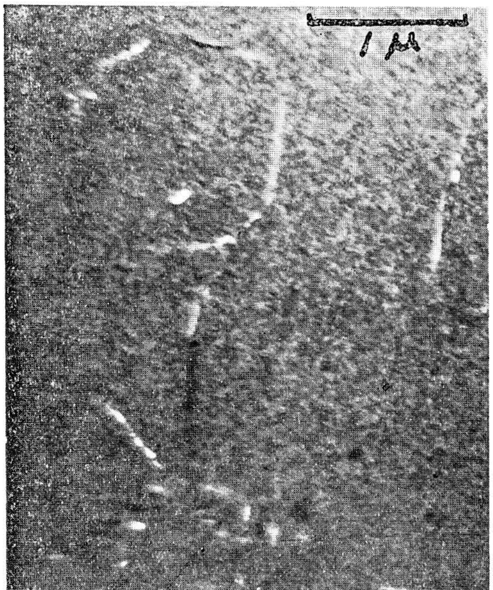

Photo. 14. Al–28.3% Zn alloy, W.Q., aged for 1 hr at 250°C. Notice preferential precipitation of α′ phase along dislocations. ×21,000

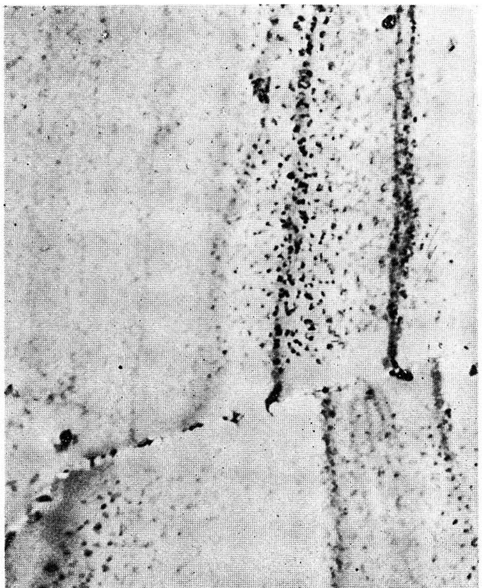

Photo. 15. Al–28.3% Zn alloy, W.Q., aged for 3 hr at 250°C. ×15,000

Photo. 16. Al–28.3% Zn alloy, W.Q., aged for 6 hr at 250°C. Notice the equillibrium zinc precipitates along the grain boundary and in the grain. ×21,000

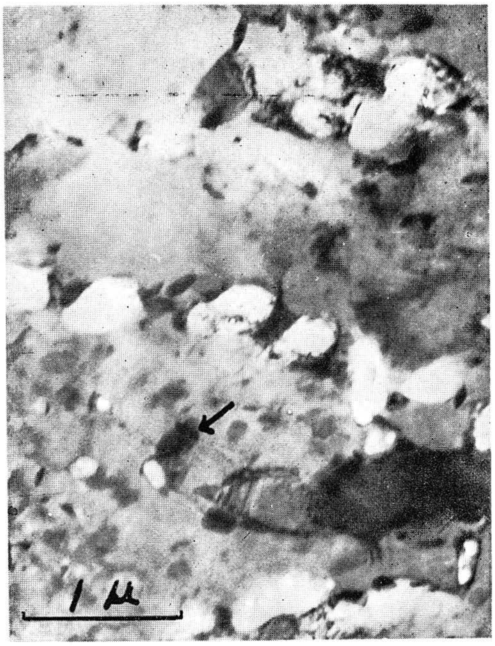

Photo. 18. Al–40.8% Zn alloy, aged for 1 hr at 250°C. ×21,000.

Photo. 17. Al–40.8% Zn alloy, W.Q., aged for 3 hr at 150°C. ×17,500

Photo. 19. Al–28.3% Zn alloy, quenched from 300°C, aged for 1 hr at 250°C. Showing precipitates having a large size thought to be equillibrium zinc phase. ×21,000

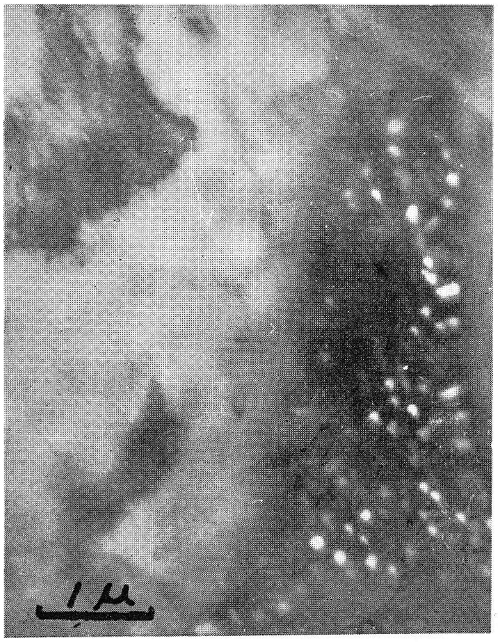

Photo. 20. Al–28.3% Zn alloy, W.Q., 30% rolled, aged for 1 day at 70°C. Showing granular precipitates. ×15,000

Photo. 22. Al–17.4% Zn alloy, W.Q., 70% rolled, aged for 3 hr at 250°C. ×35,000

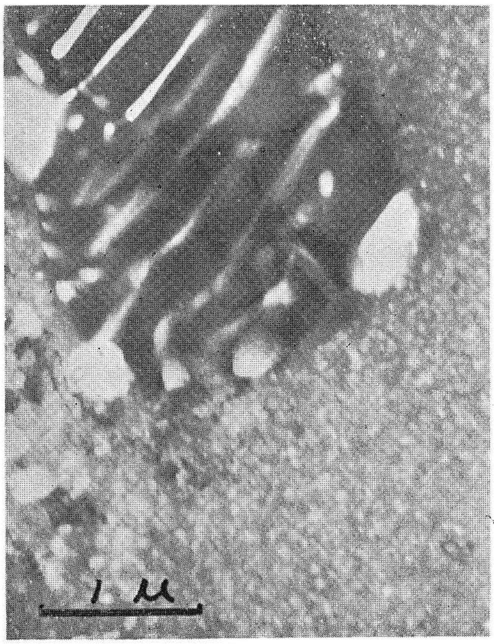

Photo. 21. Al–28.3% Zn alloy, W.Q., 30% rolled, aged for 2 days at 110°C. ×21,000

Photo. 23. Al–28.3% Zn alloy, W.Q., 70% rolled. ×21,000

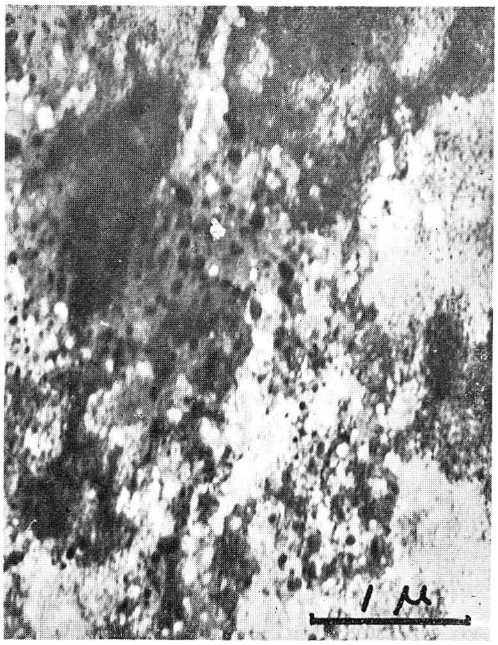

Photo. 24. Al-28.3% Zn alloy, W.Q., 70% rolled, aged for 1 hr at 70°C. ×21,000

Photo. 25. Al-28.3% Zn alloy, W.Q., 70% rolled, aged for 1 hr at 110°C. ×15,000

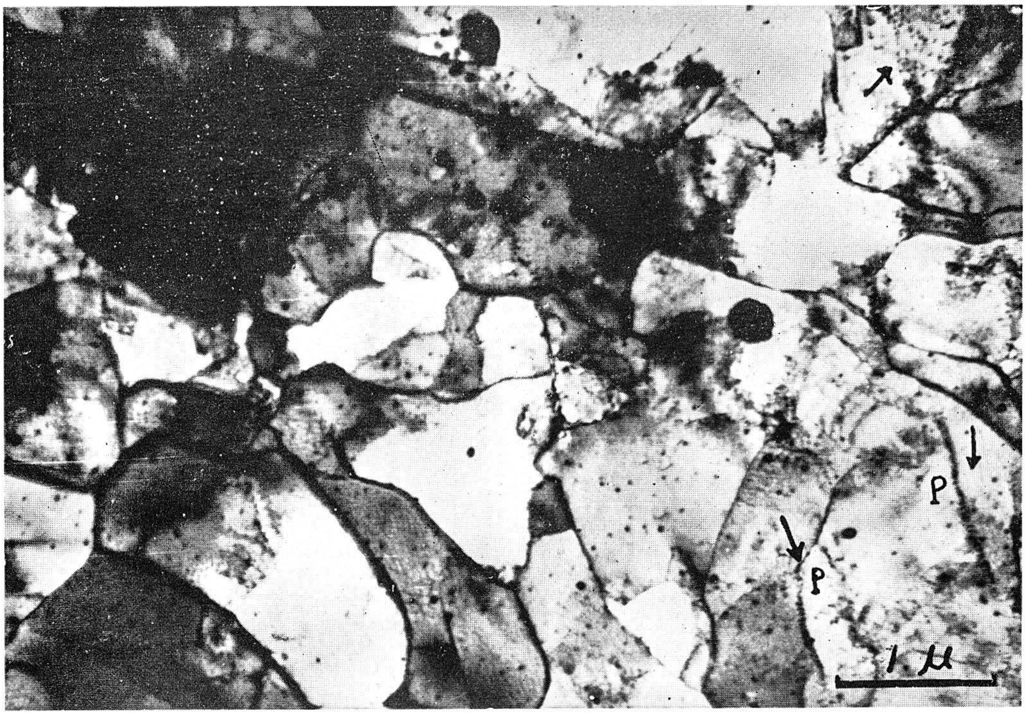

Photo. 26. Al-28.3% Zn alloy, W.Q., 70% rolled, aged for 11 hr at 200°C. Showing preferential precipitation on dislocation lines at P. ×21,000

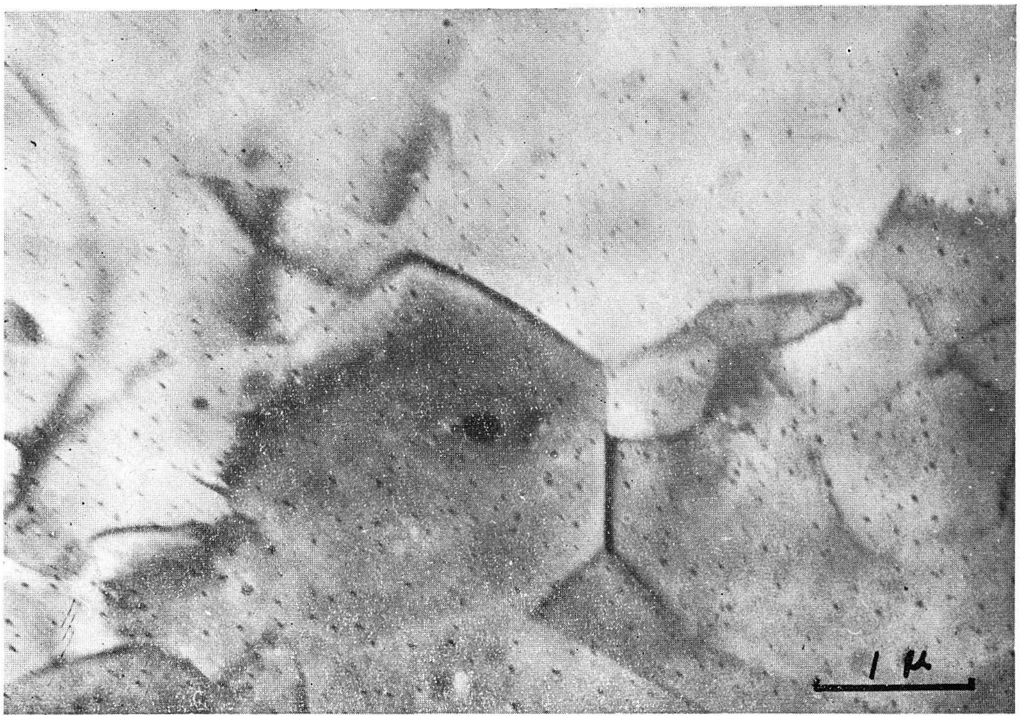

Photo. 27. Al–28.3% Zn alloy, W.Q. 70% rolled, aged for 3 days at 200°C. ×21,000

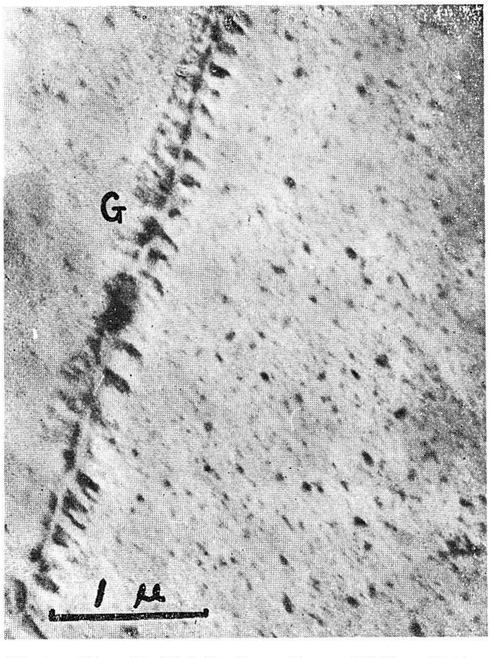

Photo. 28. Al–28.3% Zn alloy, W.Q., 70% rolled, aged for 3 hr at 250°C. Showing the first stage of grain boundary reaction at G. ×21,000

Photo. 29. Al–28.3% Zn alloy, W.Q., 70% rolled, aged for 3 days at 250°C. Showing large lamellar precipitates at L. ×21,000