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Study on precipitation phenomena during aging of the Al–Ag alloy in thin evaporated films.

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In the present work various stages of precipitation during aging of supersaturated solid solution of the Al–Ag alloy were studied on thin evaporated films by means of both electron microscopy and diffraction. The combined application of the two methods on the same specimen is important and interesting, but such studies have not been carried out with success in the case of evaporated films. The quenching of supersaturated solid solution in thin films has been believed to be highly difficult: for instance, Winkelmann (1955) considered it as almost impossible in the Al–Ag alloy. We have succeeded, however, in quenching well oriented evaporated films of about 800 Å thick of the Al–20 wt.% Ag, formed on rocksalt heated at 440° C., by the following procedure: After a film was homogenized *in vacuo* at about 500° C. for 2 hr., carbon dioxide gas was suddenly spurted into the chamber and the film was blown away into a narrow glass tube which was cooled beforehand with liquid nitrogen.

In a micrograph obtained from a film which had been left undone for about 1 month after the quenching and then was irradiated with electron beam of ordinary intensity for 30 min., the formation of spherical aggregates of about 100 Å in diameter could be observed by careful inspection. They were randomly distributed in general, but some of them appeared to lie nearly along the ⟨110⟩ direction. When the film was heated at 80° C. for about 1 hr., the spherical aggregates appeared over the whole region of the film, as shown in Fig. 1(*a*). Fig. 1(*b*) is an electron-diffraction pattern corresponding to Fig. 1(*a*) and exhibits no streak emerging from the diffraction spots. The film subjected to further annealing at 165° C. for 20 min. gave the electron micrograph shown in Fig. 1(*c*), in which fine lines are observed along two ⟨110⟩ directions perpendicular to each other. These lines are considered to be due to fine lamellae of a new phase which corresponds to the earliest stage of the γ' precipitate having a structure coherent with the {111} plane of the matrix. This new phase is believed to form by capture of silver atoms at extended dislocations. Fig. 1(*d*) is a diffraction pattern corresponding to Fig. 1(*c*). Here, streaks emerging from the diffraction spots begin to appear. When the film was further annealed at 200° C. for 2 hr., the precipitates with regular habit were formed, as seen in Fig. 1(*e*). The greater part of the contrast seems to arise from the Bragg reflection. In a corresponding diffraction pattern, Fig. 1(*f*), strong diffraction spots due to the hexagonal phase are observed flanking the spots due to the matrix. The axial ratio of the hexagonal phase was estimated to be 1·633 during the whole period after the hexagonal spots began to appear. After a further annealing at 250° C. for about 10 hr. the precipitates became somewhat well-defined in habit, as seen in Fig. 1(*g*). From this micrograph we can conclude that the habit of the precipitates is parallelepipeds with thickness of about 500–600 Å along a ⟨111⟩ direction. The values of thickness were estimated from the fringes of equi-thickness observed within images of precipitates. From the fringes we could deduce also the value of the Fourier coefficient, $V_{10\bar{1}3}$. The value obtained, 5·89 volt, agrees with the theoretical value $|V_{10\bar{1}3}| = 5{\cdot}82$ volt. The axial ratio of the hexagonal phase in this stage, 1·612, corresponded exactly to the γ' phase. When the film was heated at about 400° C. for 4 hr. and then slowly cooled down, the axial ratio decreased to 1·590, corresponding to the γ precipitate.

Castaing (1955) observed by electron microscopy and diffraction the precipitation phenomena of the Al–Cu alloy in thin sections formed by electro-polishing and ion bombardment from the bulk alloy. His method has an advantage in studying the connection between the precipitation and mechanical property. The present method, on the other hand, has advantages, as stated above, in the easy preparation of specimens and the successive observation of phenomena during aging in the same film.

A detailed report on the present work will be published elsewhere.

References

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Transmission of Kodak No Screen and DuPont Type 508 Film.

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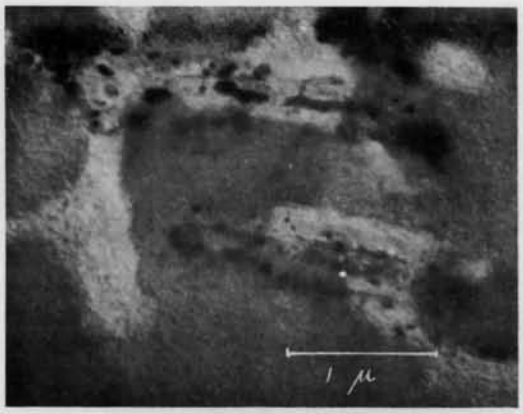
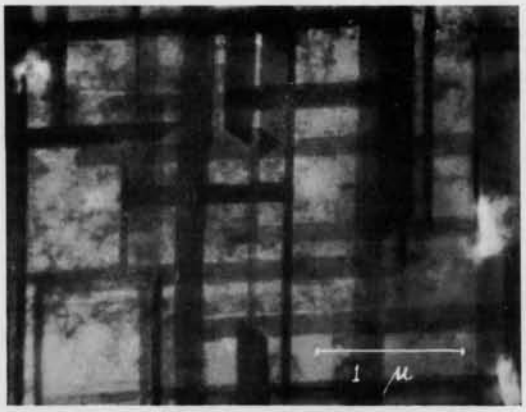
The recently published 'Comparison of Various Commercially Available X-ray Films' (*Acta Cryst.*, 1956) lists various characteristics, including the absorption of Cu radiation for 41 films. However, absorption data for Mo and other common radiations are not included. In this note the percentage transmission of two widely used U.S.A. X-ray films, Eastman Kodak No Screen and DuPont Type 508, are given for 9 characteristic X-ray wavelengths, Ag $K\alpha$ to Cr $K\alpha$.

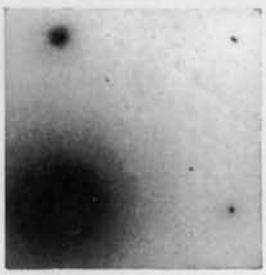
The various wavelengths were obtained from pure metals used as specimens in the Norelco fluorescence analysis unit (Parrish, 1956). A quartz crystal cut parallel to $10\bar{1}1$ was used as a monochromator. The detector was a NaI.Tl scintillation counter with discrimination only against photomultiplier tube noise (Parrish & Kohler, 1956). The transmission was calculated from the intensities recorded before and after insertion of the film in front of the counter tube. The measured values plotted



(a)

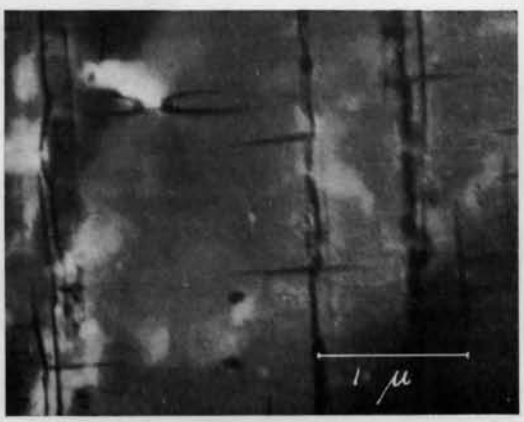


(e)

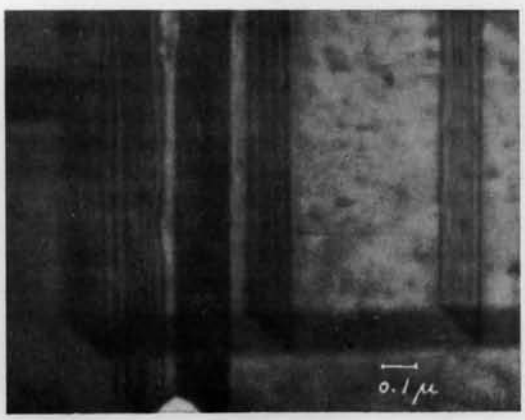


(b)

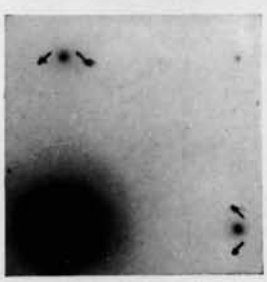
(f)



(c)



(g)



(d)

Fig. 1. Arrows in (*d*) indicate the directions of streaks emerging from the diffraction spots. Arrows in (*f*) indicate the diffraction spots due to the hexagonal phase.

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