TRANSMISSION ELECTRON MICROSCOPY OF URANIUM*

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A technique that has received wide attention in recent years is the direct examination of metals in the electron microscope. This is accomplished by thinning the metal sufficiently, usually by electropolishing, so that it is transparent to the electron beam. The important feature of this technique is that it permits the direct observation of the configurations and movements of dislocations and other crystallographic defects. Numerous applications of this technique have been made in the studies of deformation mechanisms, precipitation from solid solutions, phase transformations, and irradiation damage.[1)] The application of this procedure to the problems of deformation and irradiation behavior of uranium appeared to have great potential; therefore, the development of a technique for the thinning of uranium was undertaken.

The main difficulty was the removal of the anodic film that formed during electropolishing. Many different combinations of solutions were tried for electropolishing and removing the anodic film. The usual result was a concurrent removal of the film and dissolution of the uranium sample such that any regions of the samples that were thinned sufficiently for transmission were destroyed.

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During the course of these efforts, a report from Harwell[2] described a technique for thinning irradiated uranium. This procedure consisted of electropolishing first in H_3PO_4 (85%), and then in a solution of H_2SO_4, H_2O, and glycerine. The latter solution removed the anodic layer that formed in the H_3PO_4. On the basis of the Harwell experience, other electropolishing solutions were tried. The H_2SO_4 solution was used to remove the anodic film.

Excellent results were obtained with a solution of 800 cc ethyl alcohol, 500 cc ethylene glycol, and 500 cc H_3PO_4 (85%) with the uranium cathode at 30 ma/cm^2 and at room temperature. This solution is used frequently for the electropolishing of standard metallographic samples. The anodic film that formed was removed by electropolishing in a solution composed of 75 cc H_2SO_4 (98%), 7 cc H_2O, and 18 cc glycerine with a platinum cathode at 6 volts and a temperature between 0 and 5°C. The composition and electrical conditions for the latter solution were the same as those employed at Harwell but the temperature was lowered below room temperature. Comparision of samples polished in the H_2SO_4 solution for various times and temperatures indicated that during the electropolishing the film formed by the H_3PO_4 solution was dissolved concurrently with the formation of film from the H_2SO_4 solution. Reducing the temperature of the H_2SO_4 solution retarded the formation of that film but did not appreciably lower the rate of dissolution of the H_3PO_4 film.

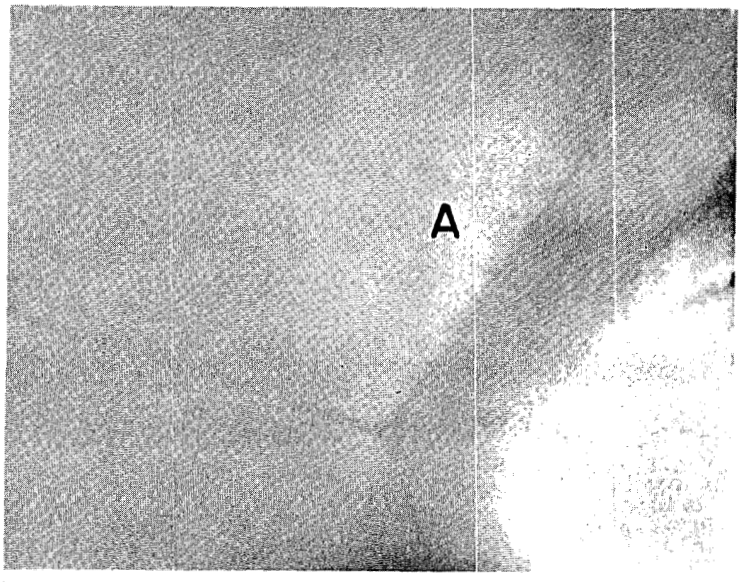
Examples of the structures that were seen in samples prepared in these solutions by the "window" technique[1] are shown in figs. 1 and 2. The samples were taken from uranium foil, 3 mils thick, which was slightly cold worked in the as-recieved condition. Some samples were annealed for one hour at 600°C to recrystallize the structure; others were beta transformed at 720°C for one hour. Both heat treatments were done in evacuated "Vycor" capsules.

Several observations were made concerning the nature of the dislocations as a function of the two heat treatments. Many more dislocations were seen in the beta-transformed foil than in the recrystallized foil. In the beta-treated foil, many of the dislocations were aligned in "chains" that formed the subgrain boundaries. Somewhat similar configurations have been seen in polygonized aluminum. None of the dislocations moved in the heat of the electron beam, as in the case of aluminum and stainless steel. No stacking faults were observed in the uranium, as have been seen in stainless steel. Future work with this technique should reveal the significance of these observations as related to the mechanical, chemical, and irradiation properties of uranium.

References

1) G. Thomas, "Transmission Electron Microscopy of Metals" (John Wiley & Sons, New York, 1962)

2) B. Hudson, K. H. Westmacott, and M. J. Makin, "Dislocation Loops and Irradiation Growth in Alpha Uranium", Harwell (UK) Report, AERE R3752 (1961)



(X 30,000)

Fig. 1 Dislocations in Recrystallized Uranium. The photograph shows the intersection of three grains. The short, dark lines near A are dislocations.

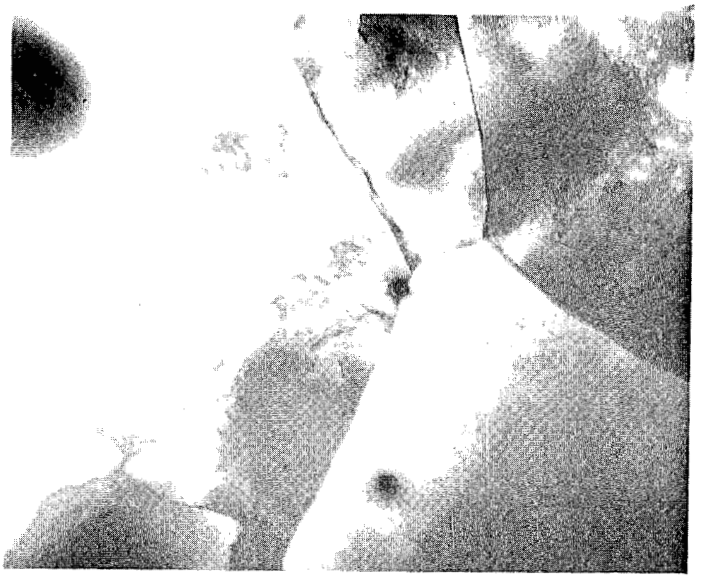


Fig. 2a. (X 11,000)

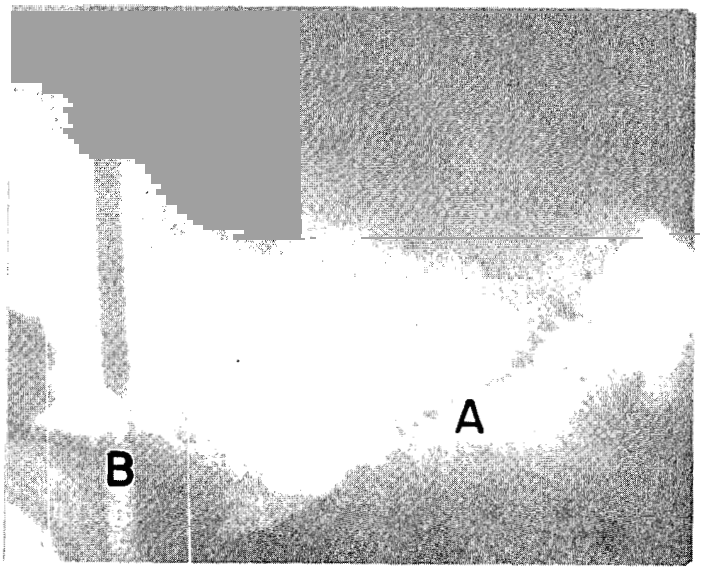


Fig. 2b. (X 30,000)

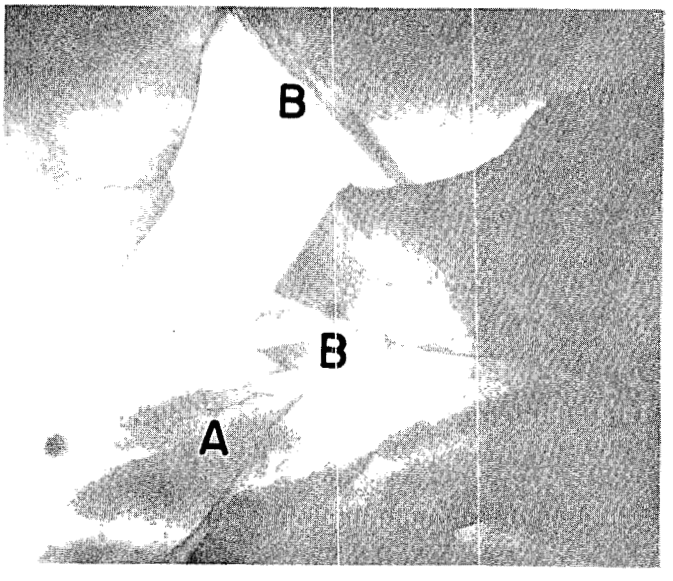


Fig. 2c. (X 7,000)

Fig. 2. Dislocations and Twins in Beta-Transformed Uranium. Figure 2a shows the general grain structure. In Figures 2b and 2c, "chains" of dislocations forming subgrain boundaries are at A and twins are at B. Numerous individual dislocations (short, dark lines) are evident in all photographs.