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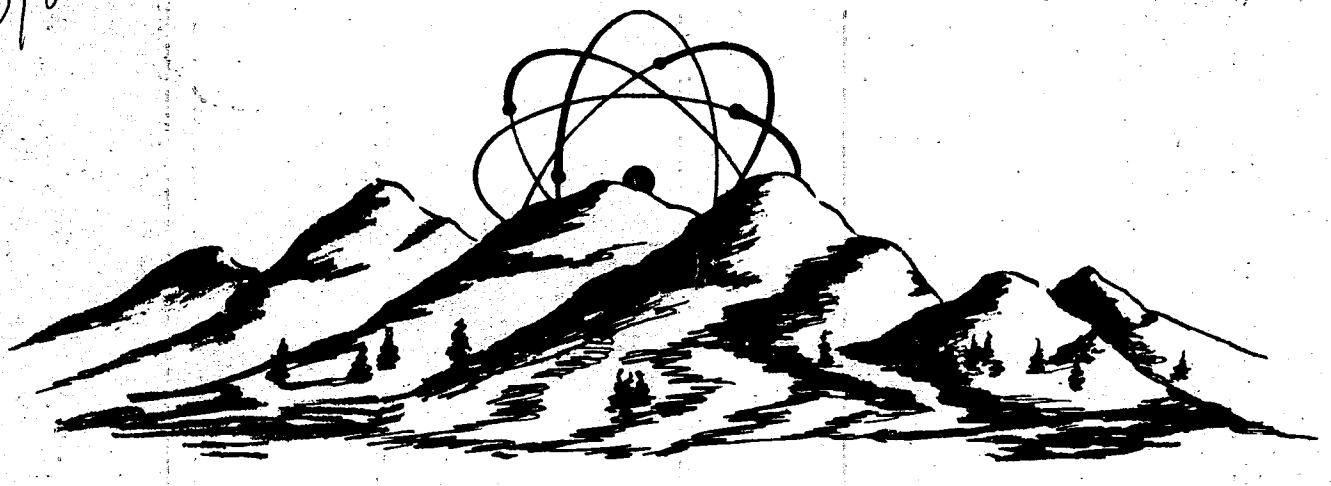

SIMPLIFIED METALLOGRAPHIC TECHNIQUES FOR URANIUM ALLOYS AND OTHER METALS

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SIMPLIFIED METALLOGRAPHIC TECHNIQUES FOR URANIUM ALLOYS AND OTHER METALS

Ross J. Jackson, W. L. Johns, and Arthur E. Calabra

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ACKNOWLEDGMENT

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SIMPLIFIED METALLOGRAPHIC TECHNIQUES FOR URANIUM ALLOYS AND OTHER METALS

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Abstract: A simplified metallographic preparation method has been developed and proves suitable for many uranium alloys, as well as for beryllium and stainless steel. Samples used in the study are taken from a cut-off wheel or 600-grit paper and placed in an electrolytic cell. Through the procedures developed, mounting and polishing steps have been eliminated and economies in operations achieved.

INTRODUCTION

Investigations were begun to develop simplified metallographic procedures suitable for uranium alloys, as well as for beryllium and stainless steel. Two procedures gave satisfactory results. In the first procedure, a standard (normal) electropolishing cell was used. In the second, use was made of an electrolyte-abrasive slurry on a rotating cathode.

Elimination of mounting and polishing steps was possible through the described procedures, and in addition economies in operations were realized.

DISCUSSION

Experimental: Two experimental procedures were developed. Procedure A involves a normal electropolishing cell and is used for uranium alloys, stainless steels, and beryllium. Procedure B employs an electrolyte-abrasive slurry on a rotating cathode and is used for beryllium.

PROCEDURE A – A standard electropolishing cell is used. The electrolyte is a proprietary phosphoric acid-base solution marketed as "Power-Kleen" which is used commercially to "brighten" metals. The supplier of this electrolyte is Molectrics, Inc., 459 N. Eucalyptus Avenue, Inglewood 3, California. The solution behaves similar to an orthophosphoric acid, sulfuric acid, and a water mixture with added metal cations. Data obtained from a spectrographic and chemical analysis of a similar solution are given in Table I. The polishing and etching conditions found to be optimum are listed in Table II.

PROCEDURE B – This method combines electrolytic and mechanical polishing techniques simultaneously and leads to rapid metal removal rates. A commercially available Struers DP-IV Electrolytic Lap-Polishing Unit is used. The electrolyte and other variables involved in this procedure are listed in Table III.

TABLE I. Spectrographic and Chemical Analysis of Electrolyte Used in Procedure A.

Spectrographic Analysis: Results are in percents.

Al – 0.005	Cu – 0.02
P – Major	Fe – 0.2
Be – <0.00025	Mn – 0.007
Ca – 0.001	Ni – 0.015
Cr – 0.015	Si – 0.003
	Mg – 0.001

Chemical Analysis:

pH	– <0.05
H_3PO_4	– 10.30 Molar
Specific Gravity, 60/60°F	– 1.68
Cl^-	– Negative
NO_3	– Negative
$SO_4^=$	– Positive (Probably about 3.6 M in H_2SO_4)

RESULTS

Procedure A: Sequence photomicrographs illustrating the simplified metallographic procedure for a uranium alloy and a stainless steel are shown in Figures 1 and 2, respectively. The first three photographs in each sequence show the attainment of an etched surface starting from 600-grit scratches. The last two photographs illustrate the reversibility of the method.

Figure 3 is a current versus time plot for various voltages. The graph shows that about 90 seconds are required for stabilization of the current. Figure 4 is a current density versus voltage plot using a series circuit. The data for this curve were obtained after a stabilization period of 90 seconds. The curve shows a polishing plateau at 0.4 amperes per centimeter^2 (amps/cm^2). The best etching characteristics were obtained below 0.1 amps/cm^2.

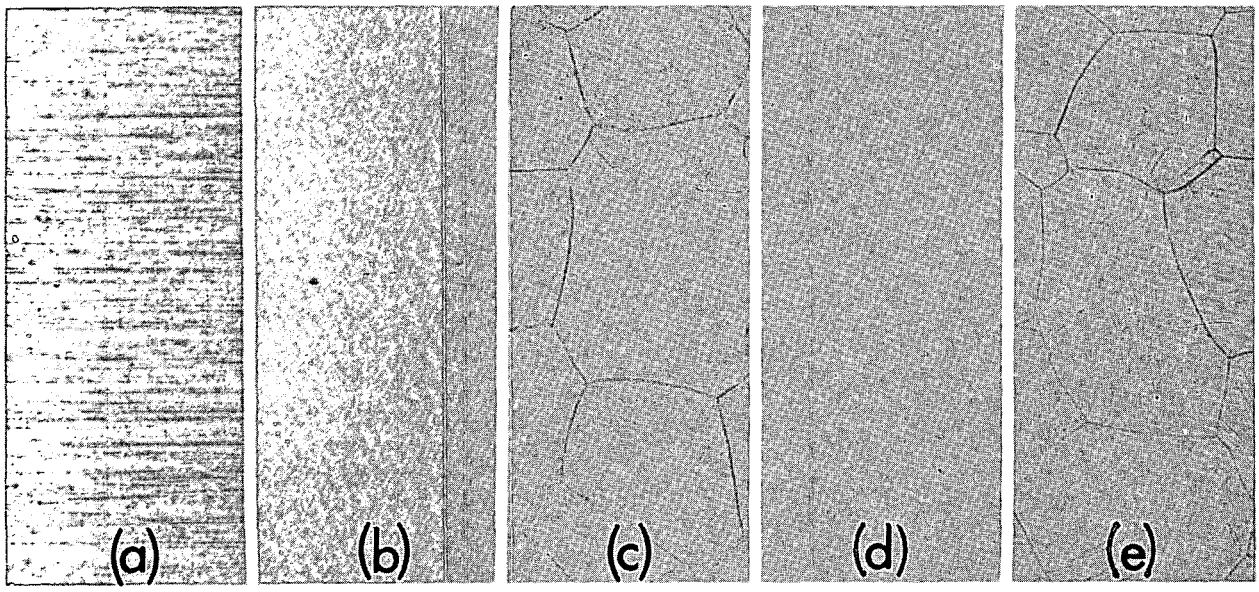


FIGURE 1. Sequence photomicrographs illustrating the simplified metallographic procedure for a gamma-quench uranium 15 at/% niobium alloy. (a) 600-grit scratches, (b) Electropolished 0.3 amps/cm^2 for 6 minutes, (c) Electroetched 0.1 amps/cm^2 for 4 minutes, (d) Repolished at 0.3 amps/cm^2 for 3 minutes, (e) Re-etched at 0.1 amps/cm^2 for 4 minutes. Bright-field illumination. Magnification 200X.

TABLE II. Optimum Polishing and Etching Conditions Using Procedure A. Solution Temperature Held Constant at 23°C.

Material	Polishing Current Density (amps/cm^2)	Polishing Time* (min)	Etching Current Density (amps/cm^2)	Etching Time (min)
Uranium Alloys: Gamma-quench U-Nb, U-Mo, U-RE, and U-Nb-Zr	0.4	6	0.10	4
Uranium	0.4	6	View polished surface with polarized light.	
Stainless Steel, 304	0.4	4	0.06	15
Beryllium	0.1 (≈15 volts)	5**	Etching accompanys polishing. Surface also responds to polarized light.	

* Polishing time starting from 600-grit scratches.

** Apply potential prior to immersing sample. The solution chemically attacks beryllium.

TABLE III. Parameters for Electrolytic-Lap Preparation of Beryllium (Procedure B).

Variable	Value
Electrolyte-Abrasive Slurry	2 volumes Nitric Acid 1 volume Hydrochloric Acid 147 volumes Ethylene Glycol 20 volumes 0.05-micron gamma-alumina
Cathode	Stainless steel covered with nylon cloth
Voltage	25 volts for 1 cm^2 sample
Current	0.6 amps/cm^2
Time from 600-Grit Scratches	2 min

Figures 5 through 12 are photomicrographs of materials prepared with the proposed procedure. Figure 12 is an electron micrograph of a replicated surface prepared using the prescribed technique. Figure 13 is a transmission-electron micrograph of a uranium alloy thinned in the electrolyte.

The technique is also suitable for obtaining zero root-mean-square (rms) finishes on machined surfaces. Metal removal rates as high as 0.003 cm/min have been obtained at high current densities. At these high current densities however, it is difficult to cool the electrolyte. For beryllium, the solution acts as a chemical polish. Scratches are readily removed in this manner, but the resulting microsurface is heavily pitted. For this reason, it is necessary to apply the potential prior to immersing the beryllium sample in the electrolyte.

For gamma-quenched uranium alloys that have been subsequently aged at 400-600°C, the method is not applicable. In these instances, the sample is given a mechanical polish followed by an electrolytic etch in 10-percent oxalic acid in water at 10°C for 4 minutes at 2 volts.

Procedure B: Photomicrographs illustrating the use of Procedure B on commercially pure beryllium are shown in Figure 14. Figure 14(a) is the initial surface showing 600-grit scratches. Figure 14(b) is after 30 seconds of electrolytic-lap polishing and shows partial removal of scratches. Figure 14(c) is after 120 seconds and shows the same area in the scratch-free condition.

For both procedures using beryllium, the method is lacking in that inclusions are consumed. This is a deficiency that is common to most electrolytic procedures involving beryllium.

Both methods however yield induced-deformation-free surfaces in which many microstructural features are resolved. When inclusion retention and an induced-deformation-free surface are required, it is best to use the following method. Grinding on 240, 320, 400 and 600-grit papers followed by polishing on 6 microns, 3 microns, and 1 micron diamond paste.

Intermediate to each of the above steps is a 15-second dip in a 10 percent H_2SO_4 in water solution to remove deformed metal. The sample is then placed for 24 hours on a vibratory polisher using 0.05-micron gamma-alumina suspended in water to which has been added 20 ml of 10 percent CrO_3 in water. This is followed by a 5-second swab with a 2 percent HF solution in ethyl alcohol. The sample is then viewed under polarized illumination.

CONCLUSIONS

Both of the proposed simplified metallographic procedures, A and B, have proven to be useful and versatile in that various metals and alloys have been successfully prepared.

The processes yield reproducible results. There are also economies in elimination of the mounting and mechanical polishing steps.

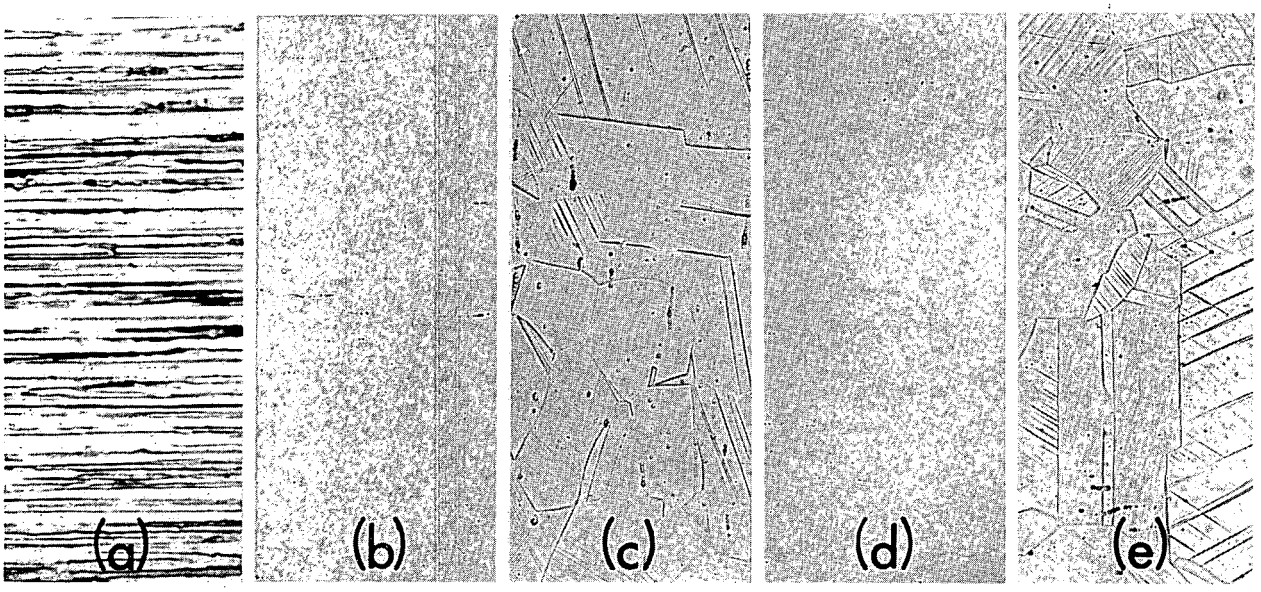


FIGURE 2. Sequence photomicrographs illustrating the simplified metallographic procedure for Type-304 stainless steel. (a) 600-grit scratches, (b) Electropolished at 0.4 amps/cm^2 for 4 minutes, (c) Electroetched at 0.06 amps/cm^2 for 15 minutes, (d) Repolished at 0.4 amps/cm^2 for 2 minutes, (e) Re-etched at 0.06 amps/cm^2 for 10 minutes. Bright-field illumination. Magnification 200X.

FIGURE 3. Current versus time plots for various voltages for the orthophosphoric base solution. Type-304 stainless steel sample. Sample size 1.0 cm^2. Stainless steel cathode. Anode to cathode distance 1.0 inch.

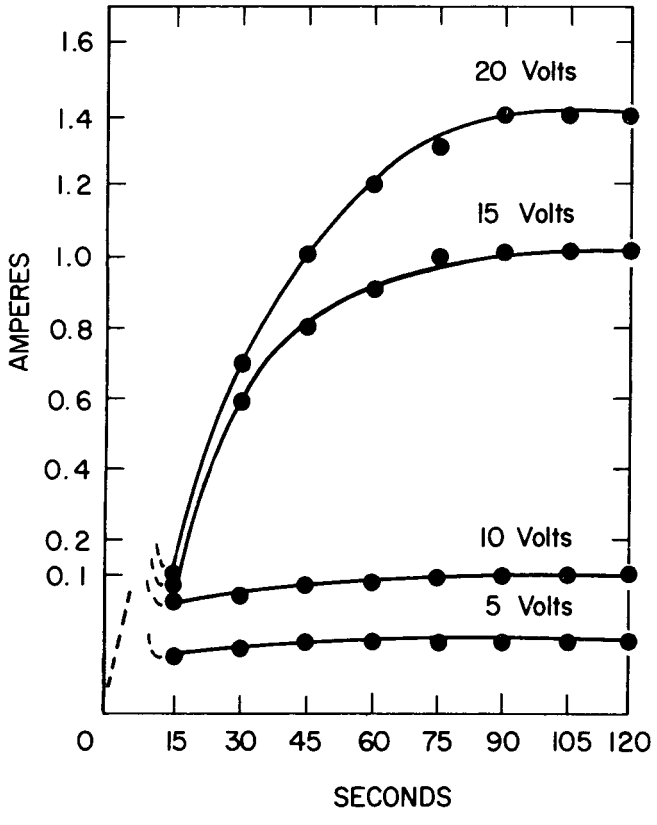
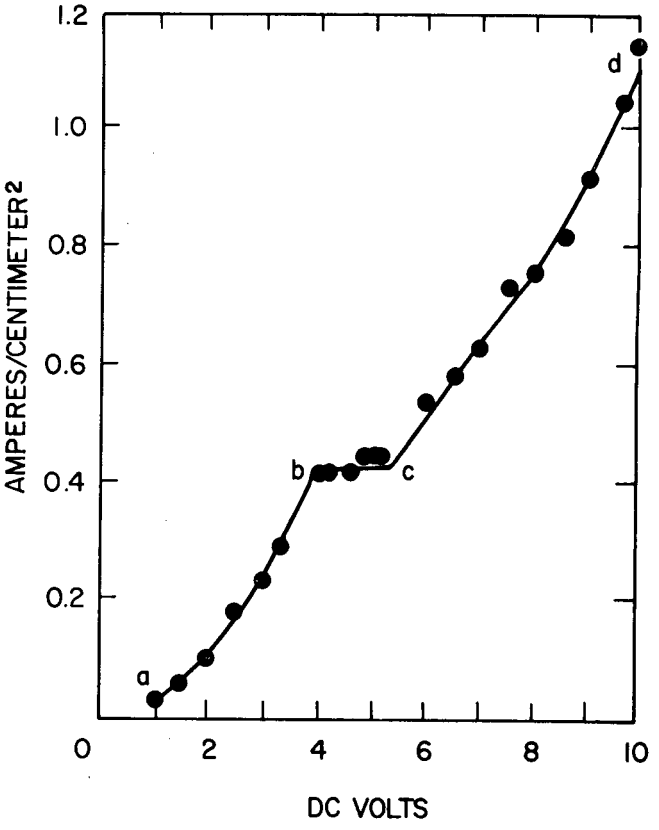


FIGURE 4. Current density versus voltage plot for the orthophosphoric base solution in a series circuit cell. Type-304 stainless steel sample. Sample size 1.0 cm^2. Stainless steel cathode. Anode to cathode distance 1.0 inch. Solution temperature 21°C. Reading taken after a 90-second hold. Region B-C represents the polishing plateau.



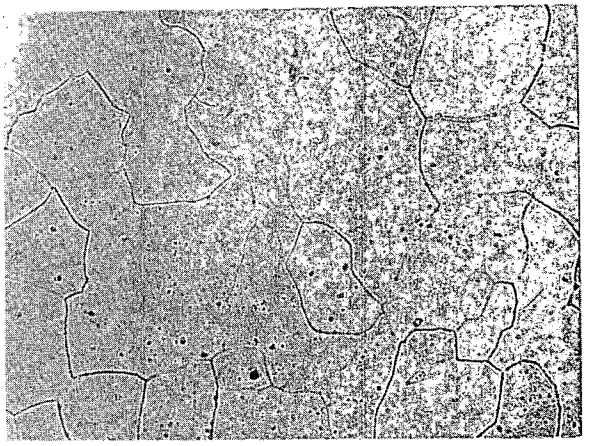


FIGURE 5. Beryllium, as-cast. Prepared under Procedure A. Bright-field illumination. Magnification 150X.

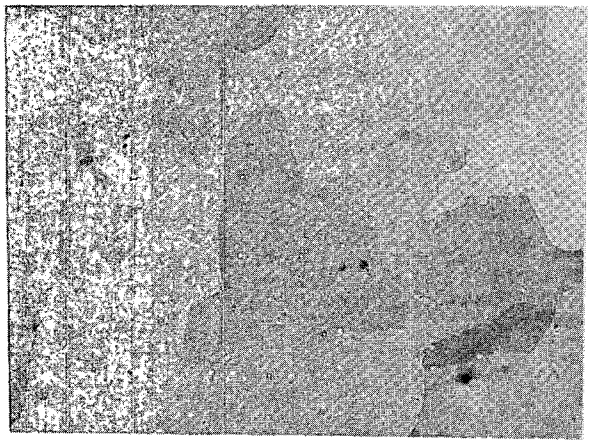


FIGURE 6. Beryllium, as-cast. Prepared under Procedure A. Polarized illumination. Magnification 200X.

FIGURE 7. Uranium 15 at/% niobium alloy. Sample held 2 days at 1100°C and water quenched. Banded α'' structure. Magnification 50X.

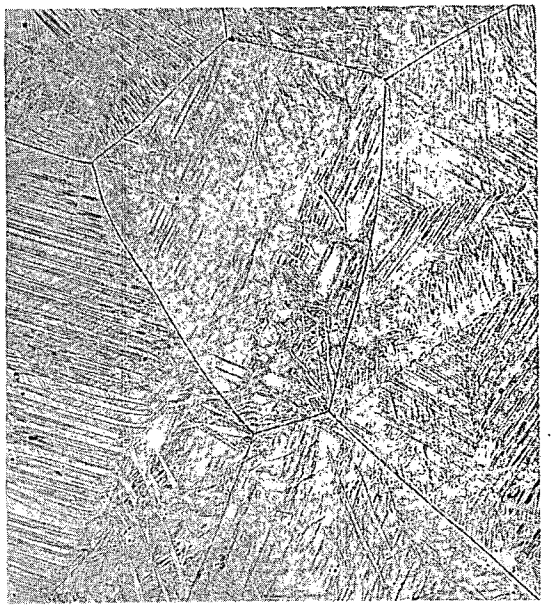
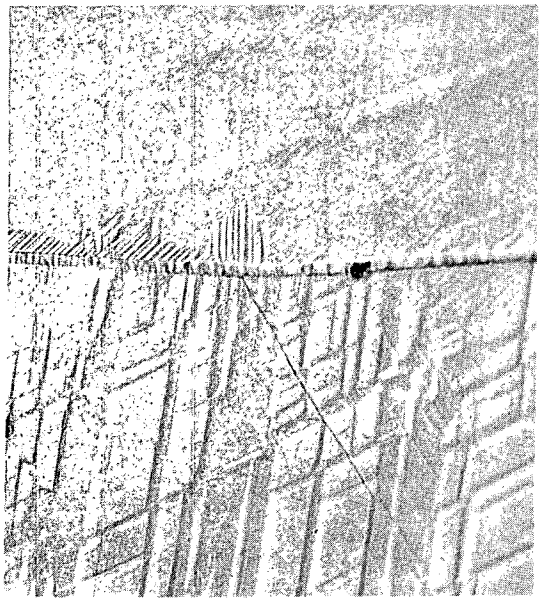


FIGURE 8. Uranium 15 at/% niobium alloy. Sample held 5 days at 1100°C and water quenched. Banded α'' structure. Magnification 500X.



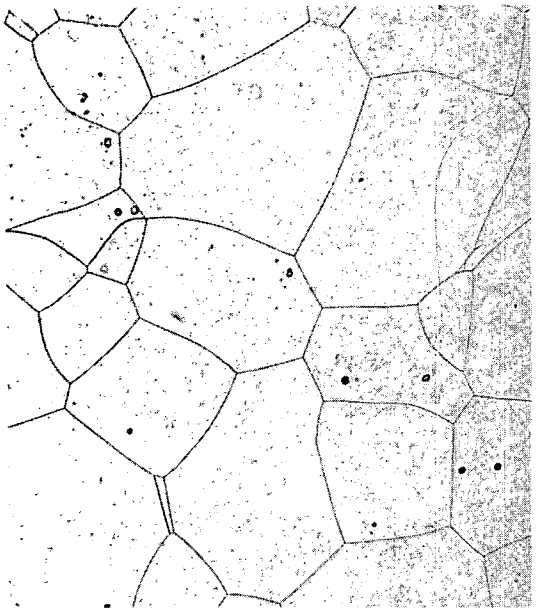


FIGURE 9. Uranium 19 at/% niobium alloy. Sample held 20 hours at 900°C and cooled at 1000°C/second. Banded, single-phase structure. Magnification 100X.

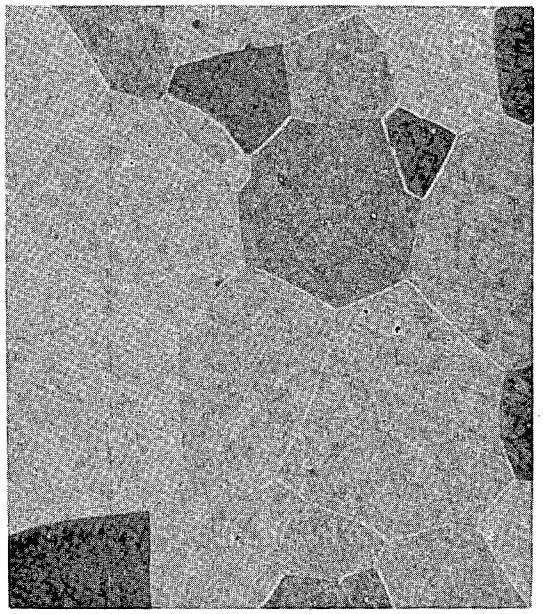


FIGURE 10. Same sample and surface conditions as in preceding photograph under polarized illumination. Magnification 100X.

FIGURE 11. Uranium 15 at/% niobium alloy. Sample held 24 hours at 850°C and cooled at 10°C/second. Subgrain boundaries and etch pits in banded, single-phase structure. Magnification 200X.

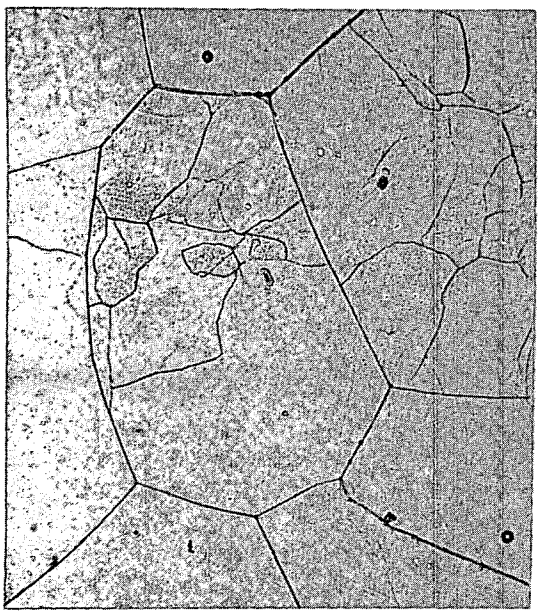
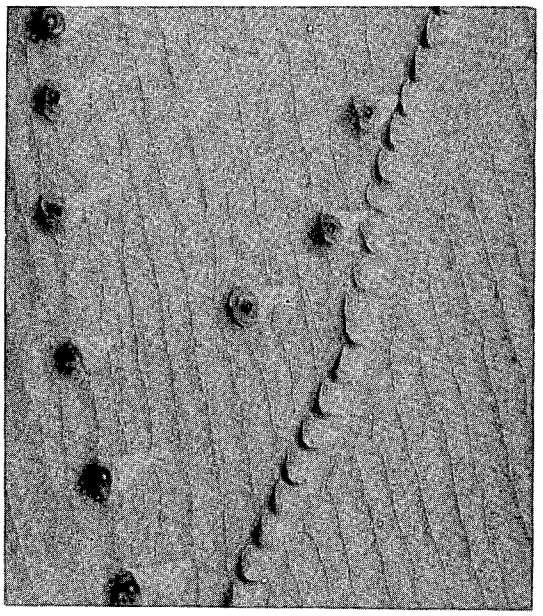


FIGURE 12. Replica electron micrograph of preceding sample. Etch pits and subgrain boundary in banded structure. Chromium-shadowed carbon replica. Magnification 4000X.



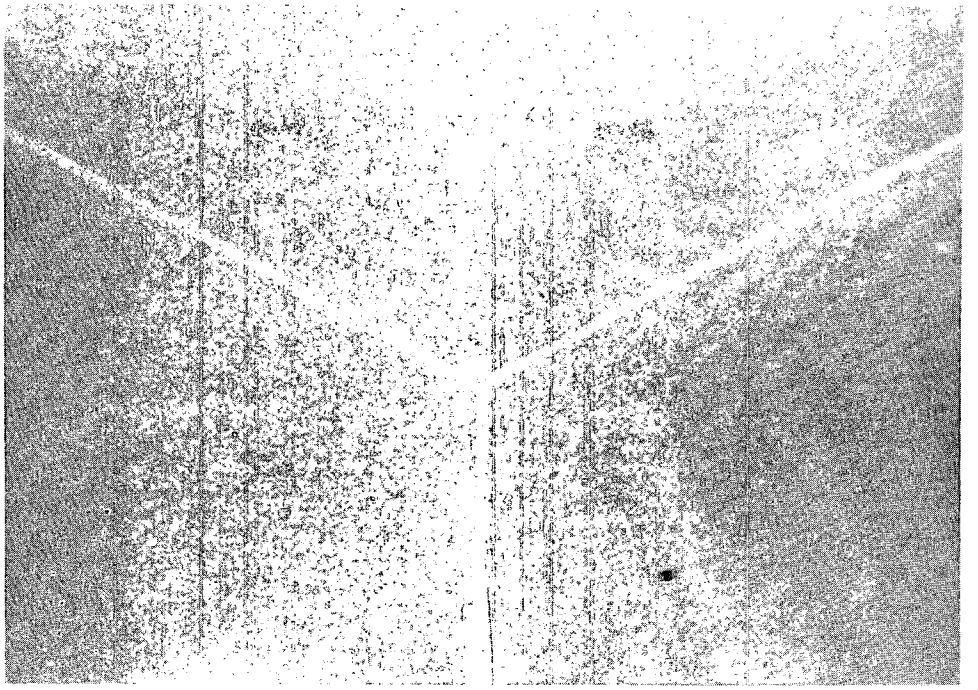


FIGURE 13. Transmission electron micrograph of a uranium 15 at/% niobium alloy thinned in the electrolyte used in Procedure A. Sample held 24 hours at 850°C and cooled at 1000°C/second. Banded, single-phase α'' structure. Magnification 28,000X.

FIGURE 14. Sequence photomicrographs showing the preparation of beryllium using Procedure B. (a) Starting surface showing 600-grit scratches, (b) Shows partial removal of scratches at 30 seconds of electrolytic-lap polishing, (c) Shows scratch-free surface after 120 seconds of electrolytic-lap polishing. Polarized illumination. Magnification 100X.