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METALLOGRAPHIC TECHNIQUES OF U-BASE ALLOYS

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AEC RESEARCH AND DEVELOPMENT REPORT

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ABSTRACT

Methods are described for metallographic preparation of binary uranium alloys having small percentages of molybdenum, niobium or silicon. Details are given of mounting, attack-polish technique, and electrolytic etching. Photomicrographs are shown of sintered specimens of U-Mo and U-Nb and of a cast specimen of U-Si.

I. INTRODUCTION

The use of uranium-base alloys for reactor fuels has created a need for investigating metallographic techniques other than the conventional methods of polishing and etching. This has been made necessary in view of the difficulty in clearly defining the structure of these two-component systems. Several alloys of uranium were studied including those with varying percentages up to five weight percent of molybdenum, niobium, and silicon.

II. MATERIAL

The U-1.4 w/o Mo, U-1.75 w/o Mo, and the U-3.0 w/o Nb alloys were made by powder metallurgy methods, cold pressed at 50 tsi and sintered for 10 hours at 1100°C *in vacuo*. Subsequently, one of the U-Mo alloys and one of the U-Nb alloys were hot rolled and gamma treated.

The U-4.0 w/o silicon alloy was made by casting. It was homogenized for one week at 850°C to allow the silicon to disperse throughout the casting, then epsilonized at 875°C for 8 hours and furnace cooled.

III. METALLOGRAPHIC PREPARATION

A. Mounting and Polishing

All of the specimens were mounted to facilitate polishing. The porous alloys as determined by density measurements, were mounted in methyl methacrylate monomer in order to fill in the pores and to prevent liquid seepage during examination; the dense alloys were mounted in bakelite.

All of the specimens were polished on No's.0 through 4/0 emery paper and in some cases, particularly with the porous alloys, kerosene was used as a lubricant to facilitate polishing. To remove most of the disturbed metal, it was necessary to polish the specimens on microcloth-covered laps with 8 micron and 1/4 micron Diamet-Hyprez as the polishing abrasive. The specimens were throughly washed in alcohol to remove kerosene and dried before final polishing. An ordinary final-polish with Linde "A" alumina abrasive was not satisfactory since a layer of disturbed metal still remained on the sample. In order to remove this layer it was necessary to adapt attack-polishing techniques.(1)

In the attack-polish technique the sample is polished by a dilute acid or base which will etch and polish at the same time. With this method the disturbed layer is removed as fast as it is formed.

III. METALLOGRAPHIC PREPARATION (cont'd)

In the case of the uranium-base alloys, it was found that most of the acids which would attack or etch uranium caused excessive staining of the alloy matrix. The acids included various concentrations of hydrofluoric, nitric, and oxalic acids, alone or in mixed aqueous solutions. It was found upon experimentation that the best solution for a clean polish, free of disturbed metal, for the uranium-niobium and uranium-silicon alloys was a composition of six grams of potassium ferricyanide, two grams of sodium hydroxide, and two grams of Linde "A" alumina in 400 cc of water.(2) For the uranium-molybdenum alloys, a solution of 10 cc of nitric acid and 10 cc of glacial acetic acid was added to 400 cc of water plus two grams of Linde "A" abrasive.

The final polish was accomplished by polishing on a micro-cloth lap with one of the above solutions, depending on the alloy being studied. The lap was covered with a bakelite disc, in order to protect the bronze wheel from the corrosive action of the various solutions used.

B Etching.

A number of etchants were tested in order to delineate the structure of the various alloys. In all of the uranium-base alloys, the best results were obtained by electrolytic etching.

For the uranium-molybdenum alloys, the most successful electrolyte used was a solution of concentrated nitric and acetic acids (1:1). The open circuit potential was 10 volts. A current density of 0.024 amp/sq. in. was maintained for 1 to 3 seconds. Photomicrographs of two alloys etched in this manner are shown in Figs. 1 and 2.

For the uranium-niobium alloys, an electrolyte of an aqueous solution of 50% phosphoric acid was found to give satisfactory results. The etching time was 1 to 5 seconds with a current density of 0.024 amp/sq. in. The open circuit voltage was 10 volts. The photomicrographs in Figs. 3 and 4 show the same alloy with different treatments.

In the case of the U-Mo and U-Nb alloys, the mounted sample was placed in a beaker containing a stainless steel cathode. The circuit was closed when the sample was touched for the desired length of etching time by a stainless steel needle connected to the anode.

III. METALLOGRAPHIC PREPARATION (cont'd)

In the case of the uranium-silicon alloy, an electrolyte containing 1 part perchloric acid [3] in 20 parts ethyl alcohol was found to give the best results. The current density varied between 0.03 and 0.04 amp/sq. in. for 2 to 10 seconds etching time. The open circuit voltage was very high, in this case, 35 volts. This electrolyte was utilized after the sample was broken out of the bakelite mount and care was taken to remove all the bakelite adhering to the sample. With no organic material present, the solution is relatively safe. One precaution to be observed in the use of this solution, is the prevention of a spark between the anode and the stainless steel cathode, which will cause the ethyl alcohol to ignite. The burning alcohol can be smothered without any danger of explosion. Fig. 5 is a photomigraph of a U- 4.0 w/o Si alloy which was espilonized.

REFERENCES

1. "Attack-Polish Method of Metallographic Preparation" by D.B. Metz and H. Woods, Sylvania Electric Products Inc, Met Labs Report No. SEP-42, October 30, 1950.

2. "Attack Method for Preparing Tungsten" by H. Woods, Metals Progress Feb. 1947, p. 261.

3. P.A. Jacquet Metal Finishing, Nov. 1949, P. 62.

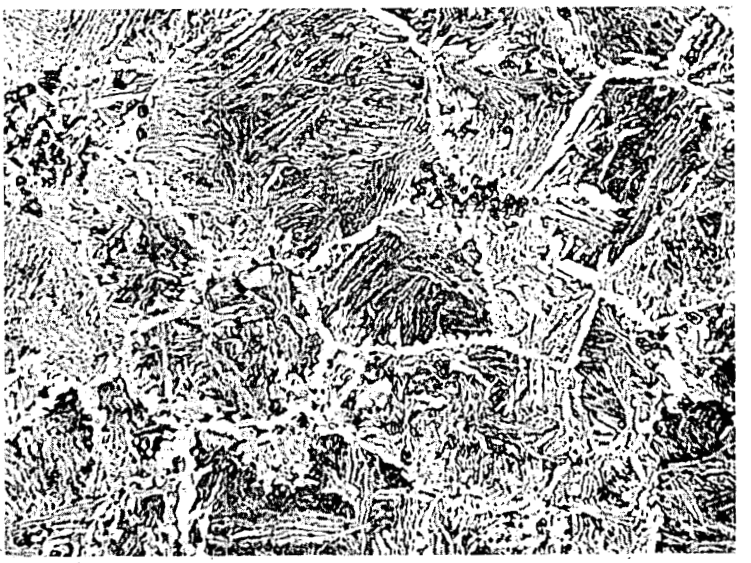

Fig. 1 U-1.75 w/o Mo Alloy. Sample prepared by cold pressing at 50 tsi and sintered for 10 hrs. at 1100°C in vacuo. Etched electrolytically with HNO_3 + HA_c

Plate No. 10867 X500

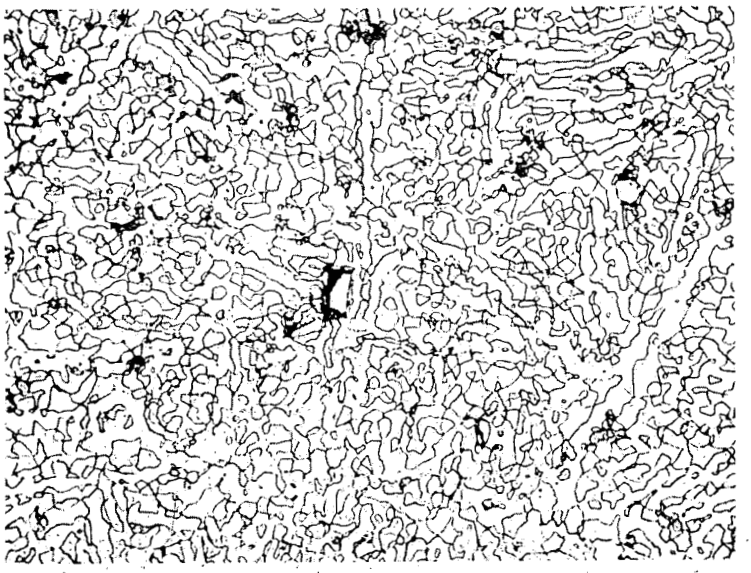

Fig. 2 U-1.4 w/o Mo Alloy. Prepared by cold pressing at 50 tsi and sintering in vacuo for 10 hrs. at 1100°C. Hot rolled at 600°C to 84% R.T. Gamma treated for 15 minutes at 700°C, air cooled. Electrolytically Etched.

Plate No. 12911 X500

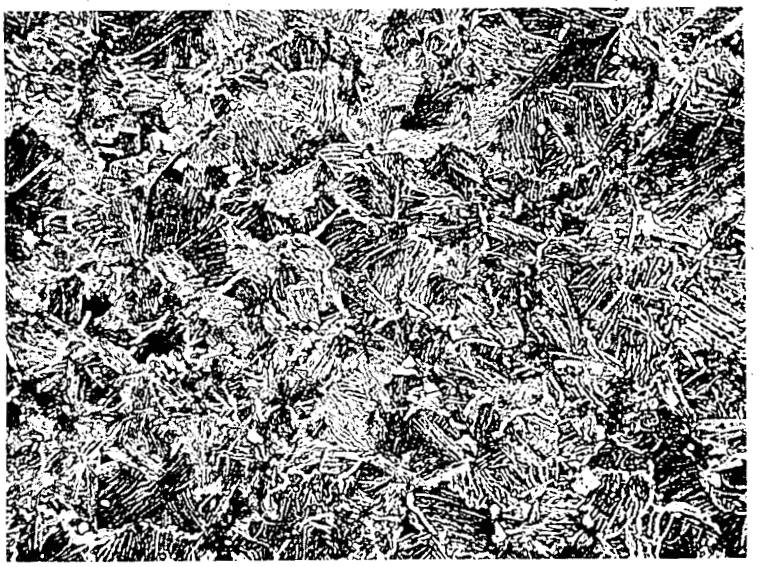

Fig. 3 U-3.0 w/o Nb alloy prepared by cold pressing at 50 tsi. Sintered for 10 hrs. in vacuo at 1100°C. Electrolytically Etched.

Plate No. 11152 X500

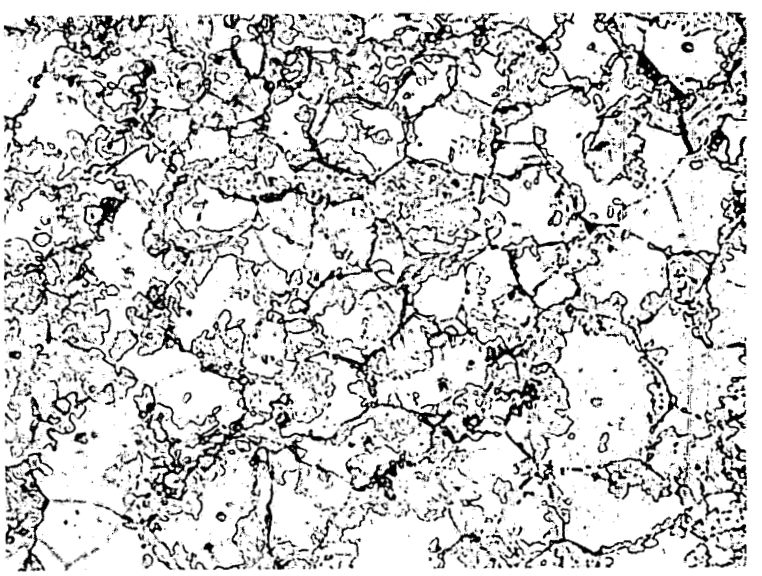

Fig. 4 U-3.0 w/o Nb alloy prepared by cold pressing at 50 tsi and sintering at 1100°C for 10 hrs. in vacuo. Hot rolled at 600°C to 80% R.T. Gamma treated at 850°C for 15 minutes and air cooled. Electrolytically Etched.

Plate No. 12903 X500

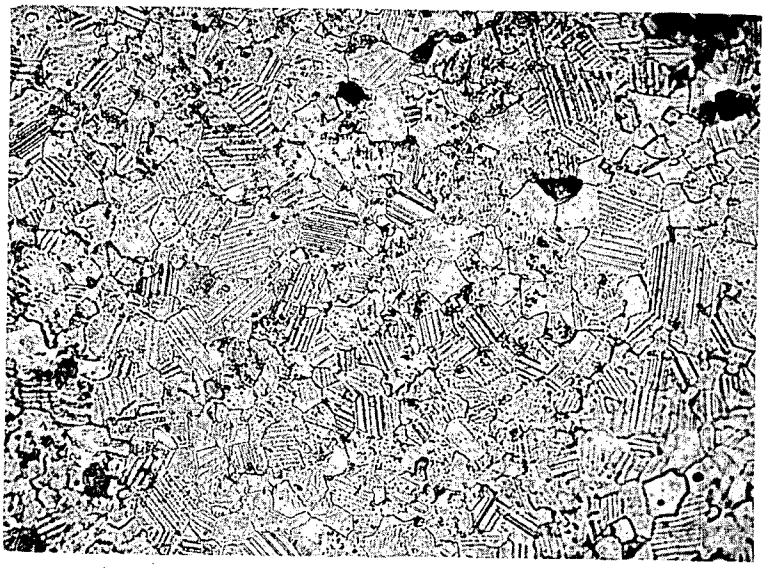

Fig. 5 U-4.0 w/o Si alloy prepared by casting. Homogenized for 1 week at 850°C. Epsilonized at 875°C for 8 hrs. Furnace cooled. Electrolytically Etched.

Plate No. 13643 X500