METALS, CERAMICS,
AND MATERIALS

CONF-680614

Technical Papers of the 22nd METALLOGRAPHIC GROUP MEETING

held June 19-21, 1968

Gulf General Atomic, Inc. San Diego, California

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DTIE Issuance Date: December 1971

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METALLOGRAPHIC PREPARATION OF PLUTONIUM

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ABSTRACT

Procedures are given for electropolishing and electroetching pure plutonium and delta phase stabilized plutonium alloys. The electrolytes used in this investigation are based on ortho-phosphoric acid. Photomicrographs are included which are representative of results obtainable using the techniques described.

INTRODUCTION

Until recently at Rocky Flats the procedure for preparing plutonium specimens for metallographic examination had been to use the electrolytic swab techniques described by Greeson, et. al.[1] The swab method, unfortunately, was found to be unsatisfactory because of the difficulty in consistently producing acceptable results. The purpose of this study, therefore, was to develop a more reliable method for revealing plutonium microstructures. This report describes an electrolytic apparatus and polish-etch conditions which were used to routinely produce metallographic results of the highest quality. The materials studied were alpha phase plutonium and delta-stabilized Pu-1 w/o Ga alloys.

EQUIPMENT

The experimental apparatus used for this work consisted of two commercial units, 1) the electrolytic cell of the Struers* Disa-pol

*Struers Scientific Instruments, Copenhagen, Denmark

electropolisher and 2) a Buehler** direct current power supply. The most important component of the apparatus is the polishing cell, the pertinent features of which are shown in the schematic drawing in Figure 1. The sample to be polished (a in Figure 1) which is the anode is clamped to the top of a mask (b) over an orifice which has a specific area. A stainless steel cathode (c) located beneath the specimen is fixed, therefore, a constant anode-cathode distance is always maintained. The electrolyte (d) which is contained in a glass bowl is projected upward onto the surface of the sample by the action of the pump (e). Hence, during polishing, the electrolyte is constantly being recirculated. The recirculation system is one of the most desirable features of the Disa-pol because not only is the sample always exposed to fresh electrolyte but also reaction products are flushed away and the sample surface cooled somewhat by the flow of the electrolyte.

EXPERIMENTAL PROCEDURE

Prior to electrolytic preparation, specimens were mounted in a cold-setting resin known as Koldmount.*** Since this material is a non-electrical conductor, a copper clip was attached to the sample to provide electrical continuity. The mounted samples were mechanically polished by 1) sanding on 180, 320, 400 and 600 grit SiC papers, 2) rough lapping with 6μm diamond paste on metcloth and 3) final polishing with 1μm diamond abrasive on nylon cloth. Carbon tetrachloride was the lubricant used in all the mechanical preparation steps.

**Buehler Ltd., 2120 Greenwood St., Evanston, Illinois

***Vernon-Benshoff Co., 413 N. Pearl St., Albany, New York

Before electropolishing the desired voltage was selected on the power supply and the proper electrolyte placed in the polishing unit. The electrolyte used for alpha plutonium contained 8 parts phosphoric acid, 5 parts glycerol and 5 parts alcohol. For the delta phase alloys a solution made up of 2 parts phosphoric acid, 1 part glycerol and 1 part 2-ethoxyethanol was used. These are essentially the same mixtures described by Greeson, et. al.[1]

Electropolishing commenced when the pump was activated. After a predetermined time interval polishing was completed and the specimens were etched by simply decreasing the voltage to the proper setting. No handling of the specimens was required at any time during electropolishing or electroetching. Etched samples were rinsed with alcohol and dried by an air blast.

RESULTS AND DISCUSSION

The parameters for polishing and etching the materials studied are given in Table I. All procedures were carried out at room temperature.

Polishing voltages were determined from curves relating cell resistance to cell voltage. According to Tegart[2] the voltage which corresponds to the maximum cell resistance is the optimum polishing voltage. The time periods listed in Table I correspond to the amount of polishing necessary to completely dissolve the worked surface layer produced during mechanical polishing.

Voltages used for etching were determined by trial and error. Since etching characteristics vary slightly from sample to sample, the times listed are average values.

Figures 2 through 5 are examples of microstructures commonly encountered at Rocky Flats. Figure 2 is a micrograph showing the grain structure of alpha plutonium. Grain structures of homogenized high purity and recrystallized nominal purity Pu-1 w/o Ga alloys are exhibited in Figures 3 and 4, respectively. Figure 5 illustrates a Pu-1 w/o Ga alloy in the cored condition. Although inclusions are not retained in etched specimens, the artifacts usually seen in micrographs of plutonium are notably absent.

CONCLUSIONS

It has been shown that by using the techniques described high quality results can be obtained on both alpha and delta plutonium. Due to the automatic nature of the equipment and efficiency of the metallographic operations has been greatly increased.

ACKNOWLEDGMENT

The author would like to thank D. V. Ferrera for his aid in preparing the specimens used for this research. The work reported here was performed under AEC contract AT(29-1)-1106.

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2. W. J. McG Tegart, Electrolytic and Chemical Polishing of Metals, Pergamon Press Inc., New York, New York (1959) 22.

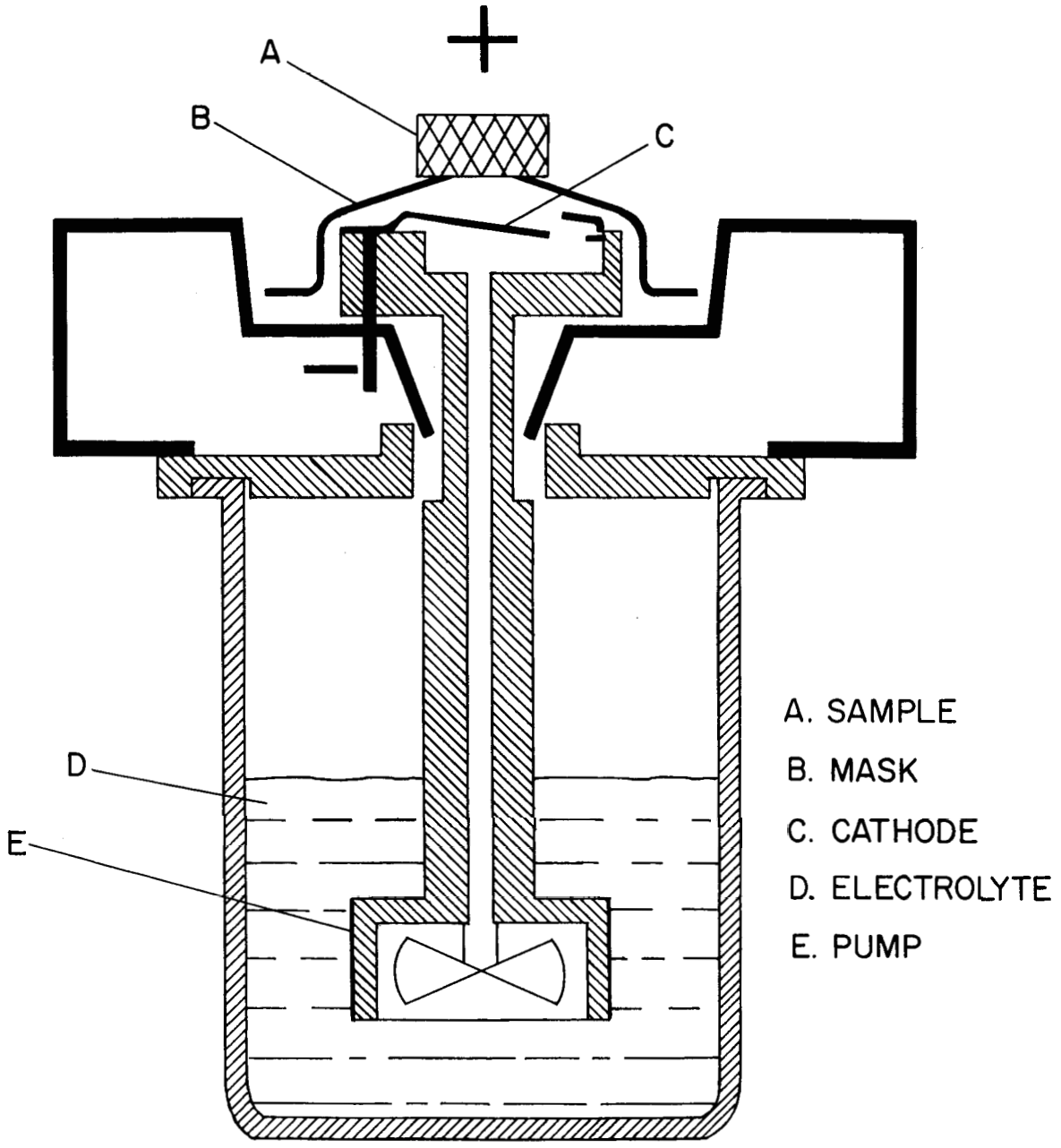

Figure 1 - Schematic drawing of the Electrolytic Cell.

TABLE I

CONDITIONS FOR POLISHING AND ETCHING
ALPHA AND DELTA PLUTONIUM

Pu Phase	Electrolyte	Micro-Structure	Polish Volts	Polish Time min.	Etch Volts	Etch Time min.	Illumination
Alpha	8-5-5*	grain structure	9	4	2	4	Polarized
	8-5-5	inclusions	9	2			Bright field
Delta	2-1-1**	inclusions	9	2			Bright field
Delta	2-1-1	worked structure	9	4	4	4	Bright field
Delta	2-1-1	cored structure	9	4	4	4	Bright field
Delta	2-1-1	recrystallized grain structure	9	4	2	4	Bright field
Delta	2-1-1	homogenized grain structure	20	2	9	4	Polarized
					2	4	

*8 parts phosphoric acid, 4 parts glycerol, 4 parts alcohol
**2 parts phosphoric, 1 part glycerol, 1 part 2-ethoxyethanol

Figure 2. Grain Structure of Alpha Plutonium
Polarized Illumination X250

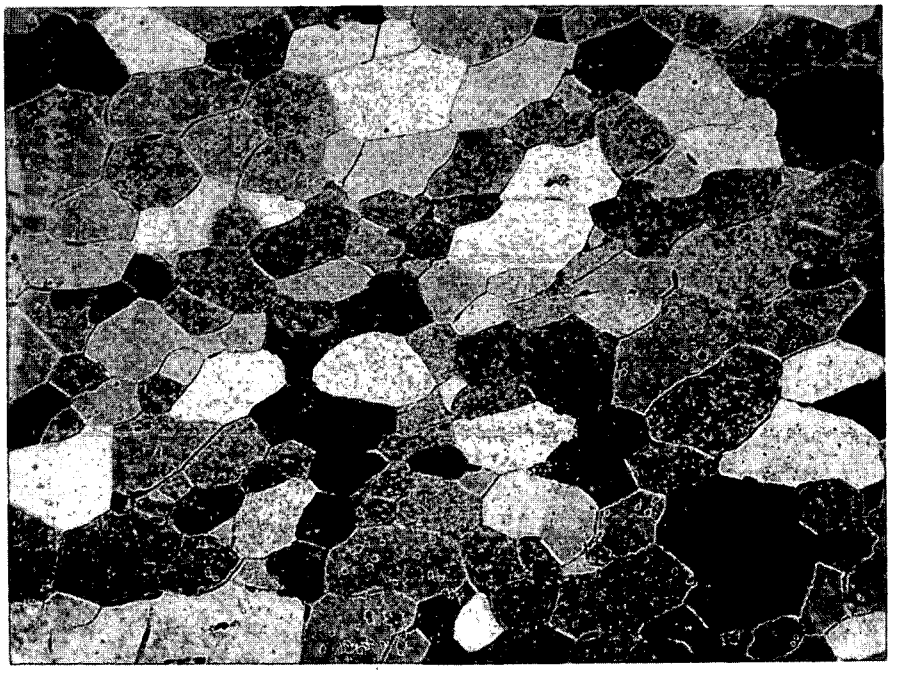

Figure 3. Grain Structure of Homogenized High
Purity Delta-Stabilized Pu-1 w/o Ga Alloy.
Polarized Illumination X250

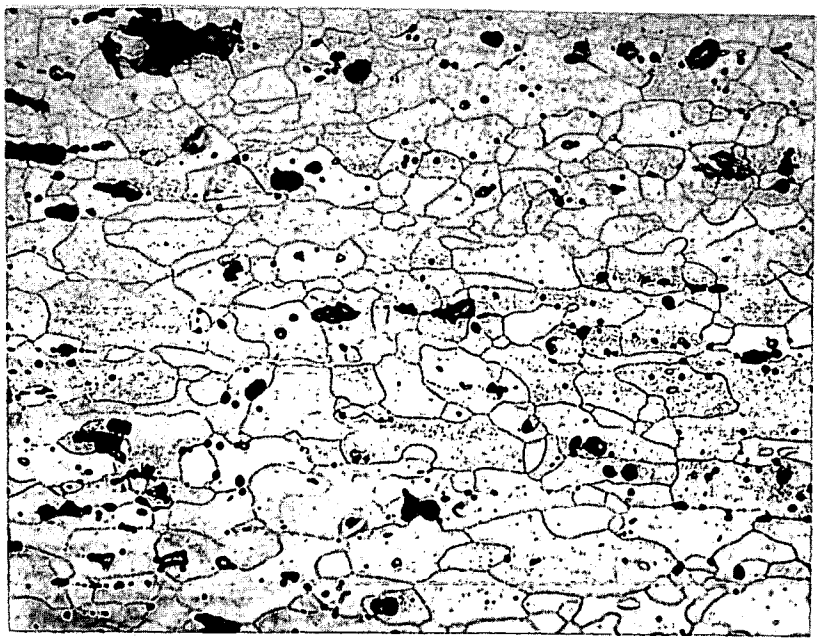

Figure 4. Grain Structure of Normal Purity Delta-Stabilized Pu-1 w/o Ga Alloy in the Recrystallized Condition.
Bright Field X250

Figure 5. As Cast Delta-Stabilized Pu-1 w/o Ga Alloy Showing Coring.
Bright Field X500