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METALLOGRAPHIC PREPARATION OF HOLMIUM

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UNION CARBIDE CORPORATION
NUCLEAR DIVISION
OAK RIDGE Y-12 PLANT

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ABSTRACT

Cathodic etching of holmium metal under an argon atmosphere produced a structure that was essentially identical in major details to that produced by careful hand polishing or chemical polishing. In addition, the surface produced was relatively reaction free.

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SUMMARY

Initially, polishing of the rare-earth metal, holmium, was by conventional silicon carbide grinding and hand polishing with diamond abrasives and alcohol. Subsequently, both chemical polishing and cathodic vacuum etching were used to produce a distortion-free structure. All of the procedures for chemical polishing and cathodic etching must be used with great care to prevent the formation of artifacts.

INTRODUCTION

Preparing samples for microscopic examination becomes a part of the evaluation of both metals and nonmetals in determining material properties and characteristics. A true structure is essential to a meaningful examination and is the goal of metallographic preparation. The rare-earth metal, holmium, was prepared for examination by procedures of mechanical polishing and etching,⁽¹⁾ and mechanical polishing followed by a chemical polish and etch.⁽²⁾ A procedure was developed for mechanical polishing and cathodic vacuum etching. Results of the three methods compare well. In cases where a quick examination is essential, hand polishing can be trusted to reveal major features; but, cathodic vacuum etching produces the cleanest surface for more detailed study. Another alternative, reported by Peterson and Hopkins⁽³⁾ but not investigated, is the electropolishing of holmium with a perchloric acid electrolyte.

SAMPLE PREPARATION

MOUNTING

The first samples were not mounted for polishing. When the phases and inclusions were observed and the necessity was established for cathodic vacuum etching, Bakelite^(a) pressure mounts were made with a copper contact from the specimen to the back surface of the mount, as described by Ladroga.⁽⁴⁾ This technique provides an electrical contact as well as a cooling surface for the specimen.

MECHANICAL POLISHING

Careful hand polishing of holmium, as is true of all metallographic polishing, attempts to provide a distortion-free surface for accurate determination of structural features. A relatively scratch-free surface was obtained by grinding on silicon carbide papers lubricated with kerosene-paraffin and polishing on diamond laps using alcohol as the lubricant. The steps in this procedure are listed in Table 1. More than one phase was evident and there were fine-line grain boundaries, as shown in Figure 1. A short final polish on a high-speed wheel oxidized the surface sufficiently for the structure to be seen with polarized light (see Figure 2).

Table 1
MECHANICAL POLISHING PROCEDURE FOR HOLMIUM SPECIMENS

Step	Abrasive and Backing Material	Abrasive Size	Lubrication
1	Silicon Carbide Paper	240 Grit	Kerosene and Paraffin
2	Silicon Carbide Paper	320 Grit	Kerosene and Paraffin
3	Silicon Carbide Paper	400 Grit	Kerosene and Paraffin
4	Silicon Carbide Paper	600 Grit	Kerosene and Paraffin
5	Diamond, Metcloth	6 Microns	Ethyl Alcohol
6	Diamond, Metcloth	3 Microns	Ethyl Alcohol
7	Diamond, Metcloth	1 Micron	Ethyl Alcohol
8	Linde B, Gamal ⁽¹⁾	0.05 Micron	Water

(1) Used only when the chemical polish or cathodic vacuum etch is not the final step. Linde B is manufactured by the Union Carbide Corporation-Linde Division.

(a) Manufactured by the Union Carbide Corporation-Plastics Division.

C794(b)

Figure 1. GRAIN STRUCTURE AS WELL AS OTHER PHASES AND INCLUSIONS ON A HAND-POLISHED HOLMIUM SAMPLE. (As Polished; Bright Field Illumination; 100X)

CHEMICAL POLISHING

Questions concerning the accuracy of the structure were raised since nothing had been done to remove the worked surface layer. Hand polishing, as outlined in Table 1, was the first step. Then a mixed acid solution reported by W. A. Roman⁽²⁾ was used in chemical polishing and etching. The procedure used is given in Table 2. Good agreement with the initial hand-polished surfaces was obtained, as seen in Figures 3 and 4. Surfaces of specimens chemically polished have a multicolored appearance to the eye, indicating an induced oxide layer, though the finely dispersed eutectic-like precipitate is most clearly defined.

C794(c)

Figure 2. IDENTICAL AREA SHOWN IN FIGURE 1 SHOWING TWINNING ALONG SCRATCHES. (As Polished; Polarized Light; 100X)

CATHODIC VACUUM ETCHING

A need for producing an undisturbed and oxide-free surface led to the use of the Numec Cathodic Vacuum Etcher (Model 4).^(b) Cathodic vacuum etching removes a surface layer by positive-ion bombardment in an inert-gas atmosphere to reveal the microstructure of the specimen. This method of final preparation was used to investigate the accuracy of the structure revealed by hand polishing and oxidizing and chemical polishing. The cathodic vacuum etching data for the specimens are pres-

(b) Manufactured by Nuclear Materials and Equipment Company.

Table 2
CHEMICAL POLISHING PROCEDURE FOR HOLMIUM SPECIMENS

Step	Abrasive and Backing Material	Abrasive Size	Lubrication
<u>Mechanical</u>			
1	Silicon Carbide Paper	240 Grit	Kerosene and Paraffin
2	Silicon Carbide Paper	320 Grit	Kerosene and Paraffin
3	Silicon Carbide Paper	400 Grit	Kerosene and Paraffin
4	Silicon Carbide Paper	600 Grit	Kerosene and Paraffin
5	Diamond, Metcloth	6 Microns	Ethyl Alcohol
6	Diamond, Metcloth	3 Microns	Ethyl Alcohol
7	Diamond, Metcloth	1 Micron	Ethyl Alcohol

Chemical Polish

1. Prepare Solution: 20 mls lactic acid, 5 mls phosphoric acid, 10 mls acetic acid, 15 mls nitric acid, and 1 ml sulfuric acid.
2. Swab the Surface for 10 - 15 Seconds With a Cotton Swab Dipped in the Prepared Solution.
3. Rinse in Ethyl Alcohol.

C818-2(a)

Figure 3. AS-POLISHED SURFACE SHOWING SEVERAL BLOCKY GRAY PHASES AND EUTECTIC DOTS; GRAIN BOUNDARIES ARE INDISTINCT. (As Polished; Bright Field Illumination; 500X)

C818-2(E)

Figure 4. MIXED-ACID CHEMICAL POLISHING OF THE SAME SPECIMEN AS SHOWN IN FIGURE 3. (Note Fewer Scratches and Sharply Defined Eutectic Areas with some Needles in the Grains. Grain Boundaries are Distinct only where Eutectic Surrounds the Grain. Bright Field Illumination; 500X)

ented in Table 3. Figures 5 and 6 illustrate the similarity of the results obtained. The initial cathodic etch produced a structure, shown in Figure 7, that was characterized by a decoration of the structure within the grains. This condition probably resulted from excessive heat that caused a reaction product to form. A reduced etch time and prolonged cooling time avoided this condition.

The etched surfaces were clean and essentially reaction free for extended times after etching, even with only minor efforts to control the storage condition. A not-too-dry desiccator has been sufficient to preserve specimens for seven months.

Table 3
CATHODIC VACUUM ETCHING DATA FOR HOLMIUM SPECIMENS

Parameter	Specimen Number ⁽¹⁾		
	C794(f)	C818-2(d)	C818-2(c)
Bombarding Gas	Argon	Argon	Argon
Gas Pressure (microns)	100	100	100
Voltage (kv)	1.5	2 - 3	2 - 3
Current Density (ma/cm ²)	2.5 - 3	3	10
Time: Cool Cycle (minutes)	1	1	30
Etch Cycle (seconds)	15	15	30
Total (minutes)	15	8	4

(1) Shown in Figures 5, 6, and 7, respectively.

C794(f)

Figure 5. CATHODICALLY VACUUM-ETCHED SPECIMEN. (Compare this Photomicrograph with Figure 2 which is the As-Polished Structure as Shown in Polarized Light. Cathodic Vacuum Etching Gives a Less Distorted, Cleaner Metal Surface. Polarized Light; 100X)

C818-2(d)

Figure 6. CATHODIC VACUUM ETCHING OF THE SPECIMEN SHOWN IN FIGURE 4 WITH A CHEMICAL POLISH. (The Grain Boundaries are More Distinct and Twins Across the Grains are Shown. Eutectic Structure is not as Distinct as Before. Bright Field Illumination; 500X)

C818-2(c)

Figure 7. SAMPLE THAT WAS INCORRECTLY ETCHED BY THE CATHODIC VACUUM TECHNIQUE. (Decoration of some Undetermined Aspects of the Grain from Letting a Sample Overheat in the Process of Etching. Grains are Defined, but Interpretation of Structure is Obscure. Bright Field Illumination; 500X)

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