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Metallographic preparation techniques for uranium

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Abstract

Existing metallographic preparation techniques for uranium are limited to elucidating specific microstructural characteristics, and some of the techniques are regarded as being environmentally unacceptable. This paper describes a newly developed technique, which is not only more environmentally friendly, but reveals most microstructural features simultaneously. Example microstructures of the various preparation stages are given to highlight the new technique.

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1. Introduction

Historically, metallographic preparation and examination of depleted uranium have been tailored to investigate specific microstructural features [1]. Traditional preparation processes include such techniques as chemical attack polishing and/or oxidation, electropolishing, electroetching, and anodizing. Although these techniques reveal individual microstructural features, a more general technique (which can reveal all aspects of the microstructure simultaneously) has been lacking. Other problems associated with past techniques include etch pitting, loss of inclusions during electro-polishing, and personal and environmental safety concerns. An evaluation of existing techniques has provided the basis for a modification and the subsequent development of a new metallographic preparation technique. This technique defines grain boundaries, sub-grain boundaries, inclusions, impurity segregation and twinning, thus allowing for a broader array of microstructural characteristics to be revealed with a single preparation while minimizing both dangerous chemical mixtures and hazardous waste generation.

2. Specimen preparation

In this section, the specimen preparation steps will be described in some detail. For each stage of the process, the previously recommended procedure will be described, followed by a presentation of the modifications to this procedure in the new technique.

2.1. Sectioning and mounting

2.1.1. Currently recommended [1]

To minimize surface damage, specimens are sectioned with a low speed diamond (or abrasive)

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saw using liberal amounts of non-flammable cutting fluid. The specimens can be mounted in any of the common metallographic mounting materials, such as Bakelite, phenolic, and epoxy. The use of epoxy is discouraged without additional steps (such as nickel plating or coating the specimen with epoxy paint) prior to mounting. The plating or coating step is recommended because the uranium can react with epoxy, which causes a gas evolution problem during curing. However, because uranium surfaces oxidize rapidly when exposed to air, the nickel plating may not adhere. A suggested solution to this problem is to sputter a layer of conductive material onto the oxidized surface prior to the nickel plating process.

2.1.2. New technique

To minimize surface damage and the spread of contamination, specimens are sectioned using low speed diamond or abrasive saw and water as the lubricant. Water is preferred over oil-based lubricants because the residue on the sample quickly evaporates, thereby eliminating solvent cleaning prior to mounting.

While the referenced literature indicates preference for the use of compression mounting, this may not be an acceptable mounting method for material that is fragile or susceptible to phase transformation at or near the molding temperature (180 °C). The mounting method used in this study consists of vacuum impregnation [2] with a slow curing epoxy (10 to 1 ratio of epon 815 resin and diethylenetriamine) followed by pressure curing. This is accomplished using a vacuum chamber with a tilt/pour mechanism to allow simultaneous evacuation of the specimen and epoxy as well as subsequent pouring of the epoxy into the mounting cups. After venting the system, the mounting cups are placed in a pressure vessel to cure at room temperature in a dry nitrogen atmosphere at 5.5–6.9 MPa for a minimum of 5 h, with an overnight cure preferred. This method not only affords excellent penetration of the resin to all surface accessible porosity (making it easier to observe cracking and distinguish between porosity and preparation artifacts) but also eliminates the problem of gas evolution/entrapment in the mounted sample. To further harden the epoxy mounting material, specimens can be final cured for 2 h at approximately 60 °C. This step may be omitted for particularly heat sensitive specimens.

2.2. Grinding and mechanical polishing

2.2.1. Currently recommended [1]

The specimens are sequentially ground through 600 grit (CAMI 14.5) SiC grinding papers using water as a lubricant or 600 (CAMI 14.5) grit aluminum oxide powder in a kerosene vehicle on a cast iron lapping wheel. A uniform 600-grit finish is adequate for subsequent polishing. Coarse polishing is performed using 30-μm diamond abrasive followed by 6-μm diamond abrasive on a nylon lap with a petroleum-base vehicle. These steps are required to remove latent grinding damage. Final mechanical polishing is done on a high-nap cloth with 0.3-μm Al_2O_3 abrasive and a deionized water vehicle on rotating wheels or vibratory polishers (up to 12 h).

2.2.2. New technique

Grinding is accomplished in the manner described above, except that the final grinding step is performed on 800 (CAMI 12.2) grit SiC paper. The final grinding step allows for the elimination of the coarse (30 μm and 6 μm) polishing steps and therefore enhances the ability to retain inclusions in the metallographic sample. Initial mechanical polishing is accomplished on a low nap cloth (Texmet [3] or equivalent) using a 3-μm diamond abrasive, 15 N force, 150 rpm counter rotation, and a propylene glycol lubricant for ~10–15 min. During the final mechanical preparation step, samples are polished on a napped cloth using either of the following methods: (a) 1 μm diamond suspension, 15 N force, 150-rpm counter rotation, and a propylene glycol lubricant for approximately 5–7 min, or (b) 0.3 μm alumina slurry, 15 N force, 150-rpm counter rotation with a water lubricant. Method (a) is preferred because it minimizes oxidation effects during mechanical preparation. Optical examination of the mechanically polished specimen reveals some inclusions, defects (porosity or cracks), and (using polarized light) grain boundaries.

2.3. Electropolishing and electroetching

2.3.1. Currently recommended [1]

To further define microstructural characteristics, specimens are oxidized or chemically prepared using such techniques as electropolishing, electroetching, or anodizing. Some of the more acceptable preparation processes along with their uses and limitations, can be summarized as follows: