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Ion-Beam Etching Techniques in Uranium Metallography

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The Department of Energy (DOE) design labs have recognized the need for metallographic controls, such that digital imaging and quantitative data reports would be in a standardized form for comparison purposes. In addition, there must be standardized sample preparation procedure to ensure that similar features are quantified similarly in the reports. The DOE metallography labs all use different methods for uranium metallography, including mechanical polishing, chemical etching, and image capturing. Some methods include allowing the samples to oxidize in air with no further chemical etching.

Ion etching techniques were successfully developed for optical microscopy (OM), scanning electron microscopy (SEM), and electron backscatter diffraction (EBSD) characterization. It is believed that this process will reduce current hazardous chemical waste streams, as well as establish procedure-based metallography processes to ensure that crucial metallographic techniques will not be lost when key personnel retire.

The best procedures for ion milling for EBSD included using xenon ions at 6kV with slow rotation, using a 70° tilt for 30 minutes to remove the oxide layer, followed by using an 80° tilt for 2 hours to remove the surface damage [1]. However, shorter times are necessary to be competitive with traditional metallography chemical etching techniques, and OM is more forgiving than SEM and EBSD evaluations. The best parameters for OM evaluation of uranium include adequately etching with xenon ions at 6kV by rocking the specimen between 0° and 30° tilt, while rotating about the surface normal, for two to five minutes [1]. The resulting image is shown in Figure 1.

It was also found that depositing a small layer of carbon on the samples reduced the oxidation rate of the etched material. After the ion etching surface preparation, the samples are carbon coated at a rate of 0.2-0.4 Å/s, using a slow rotation, to a total thickness of 25 Å. This minute amount of carbon retards the oxidation rate of the uranium surface and allows evaluation of the sample weeks after preparation, where uranium samples typically are so badly oxidized within minutes that a thorough inspection becomes difficult if not impossible. Figure 2 shows an uncoated, mechanically polished and chemically etched sample that has considerable surface degradation due to oxidation after only 4 hours. In contrast, Figure 3 shows an ion etched and carbon-coated uranium sample that has been exposed to East Tennessee air for 30 days.

Reference:

[1] D.A. Carpenter and R.L. Bridges, "Surface Preparation of Uranium by Ion Milling," *Microscopy Today*, Vol. 16 No. 3 (May 2008).

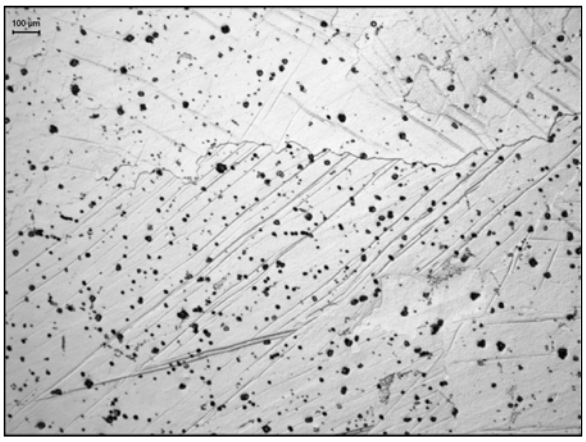

Figure 1: OM image of uranium using ion etching techniques.

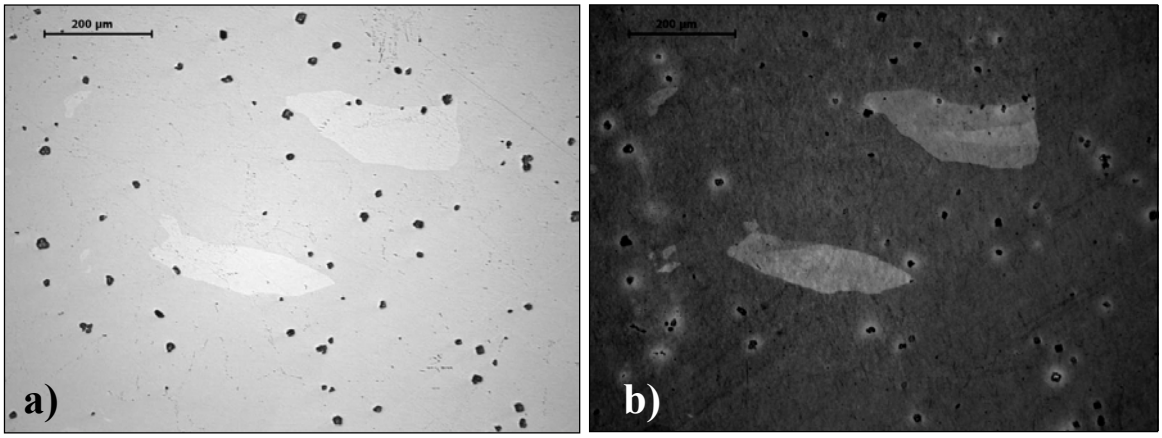

Figure 2: a) Traditionally prepared uranium, and b) surface degradation after only 4 hours.

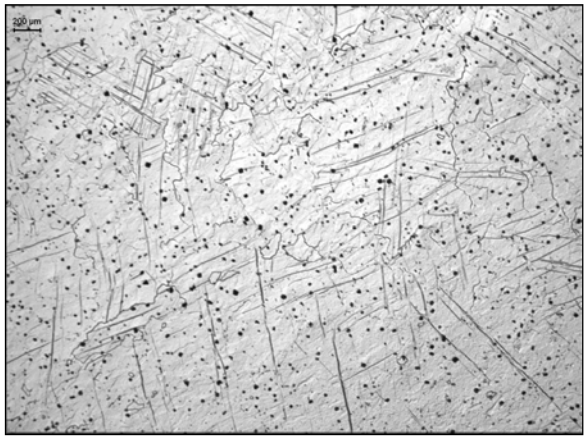

Figure 3: Ion etched and carbon-coated uranium that has been exposed to air for 1 month.

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