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PLUTONIUM METALLOGRAPHY AT LOS ALAMOS

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Plutonium Metallography at Los Alamos

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From early days of the Manhattan program to today, scientists and engineers have continued to investigate the metallurgical properties of plutonium (Pu). Although issues like aging was not a concern to the early pioneers, today the reliability of our aging stockpile is of major focus. And as the country moves toward a new generation of weapons similar problems that the early pioneers faced such as compatibility, homogeneity and malleability have come to the forefront. And metallography will continue to be a principle tool for the resolution of old and new issues.

Standard metallographic techniques are used for the preparation of plutonium samples. The samples are first cut with a slow speed diamond saw. After mounting in Epon 815 epoxy resin, the samples are ground through 600 grit silicon carbide paper. PF 5070 (a Freon substitute) is used as a coolant, lubricant, and solvent for most operations. Rough mechanical polished is done with 9-μ diamond using a nap less cloth, for example nylon or cotton. Final polish is done with 1-μ diamond on a nappy cloth such as sylvet. Ethyl alcohol is then use ultrasonically to clean the samples before electro polishing. The sample is then electro-polished and etched in an electrolyte containing 10% nitric acid, and 90% dimethyleneformalmide. Ethyl alcohol is used as a final cleaning agent.

Although standard metallographic preparation techniques are used, there are several reasons why metallography of Pu is difficult and challenging. Firstly, because of the health hazards associated with its radioactive properties, sample preparation is conducted in glove boxes. Figure 1 shows the metallography line, in an R&D facility. Since they are designed to be negative in pressure to the laboratory, cross-contamination of abrasives is a major problem. In addition, because of safety concerns and waste issues, there is a limit to the amount of solvent that can be used.

Secondly, Pu will readily hydride or oxidize when in contact with metallographic polishing lubricants, solvents, or chemicals. And water being one of the most reactive solutions, is not used in the preparation. Figure 2 shows an example of a plutonium sample in which an oxide film has formed on the surface due to overexposure to solutions. It has been noted that nucleation of the hydride/oxide begins around inclusions and samples with a higher concentration of impurities seem to be more susceptible to this reaction. Figure 3 shows examples of small oxide rings, forming around inclusions.

Lastly, during the cutting, grinding, or polishing process there is enough stress induced in the sample that the surface can transform from the soft face-centered-cubic delta phase (30HV) to the strain-induced monoclinic alpha' phase (300HV). Figure 4 and 5 shows cross-sectional views of samples in which one was cut using a diamond saw and the other was process through 600 grit. The white layers on the edges is the strain induced alpha' phase. The "V" shape indentation in Figure 5 was caused by a coarser abrasive which resulted in transformations to a depth of approximately 20 μm. Another example of the transformation sensitivity of plutonium can be seen in Figure 6, in which the delta phase has partly transformed to alpha' during micro hardness indentation.

Figure 1. This is a photograph of glove boxes in the R&D plutonium metallography laboratory.

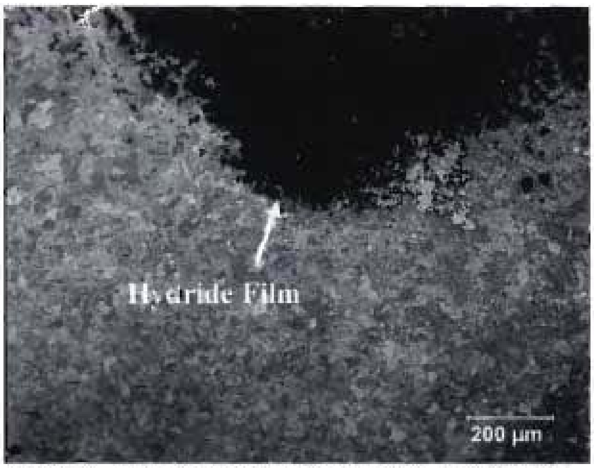

Figure 2. This micrograph shows the formation of a plutonium hydride film (dark) spreading over the surface of the sample.

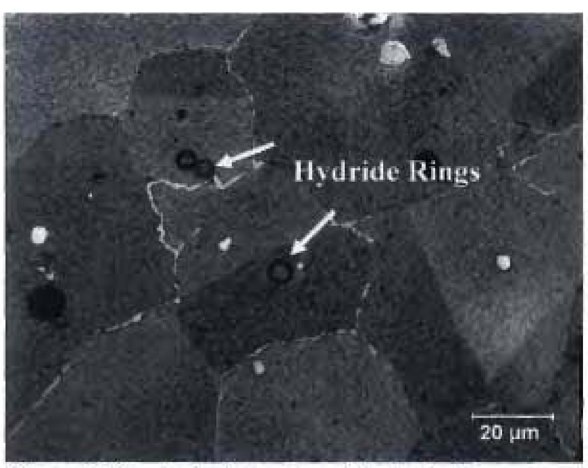

Figure 3. The dark rings around the inclusions are an indication that they are nucleation sites for the formation of plutonium hydride.

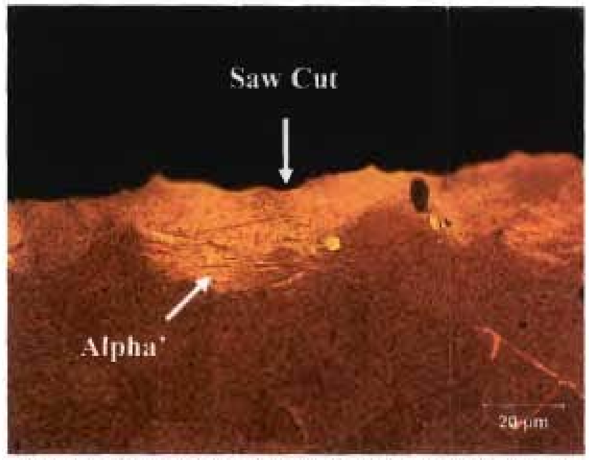

Figure 4. The light layer in this micrograph shows the depth (30 μm) of the stress induced alpha' (light phase) as the result of cutting with a diamond saw.

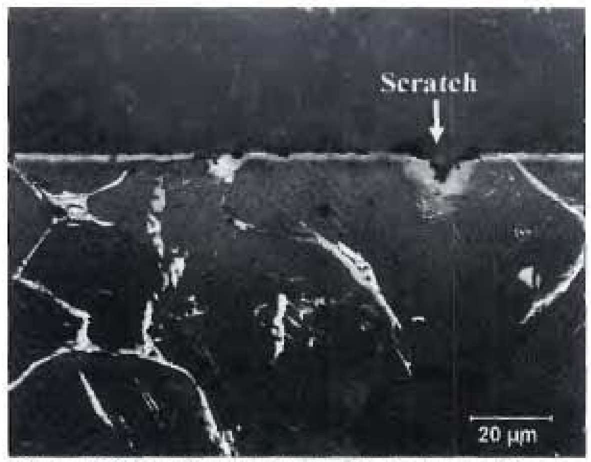

Figure 5. The delta (dark) transformed to alpha' (light) as a result of a 10-μm scratch on the surface of the sample. The scratch was due to cross contamination from a larger abrasive.

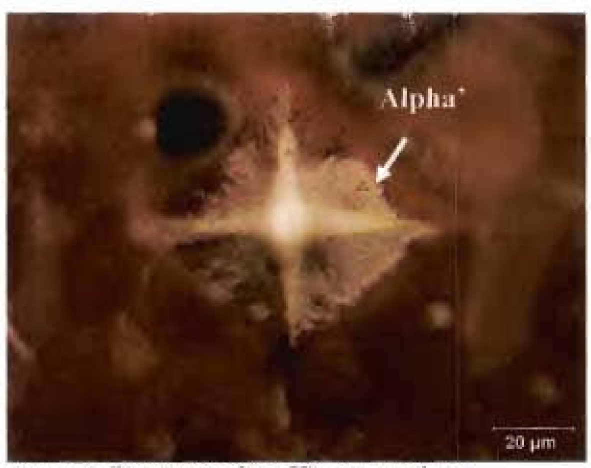

Figure 6. The stress of the Vickers hardness indentation has transformed the delta (dark phase) to alpha' (light phase).