TECHNIQUES USED IN THE REMOTE METALLOGRAPHY
OF IRRADIATED PLUTONIUM

Pu
C. C. Burwell, W. E. Ferguson and F. H. Newbury

University of California
Los Alamos Scientific Laboratory
Los Alamos, New Mexico

INTRODUCTION

Hot Cell metallography installations are quite common today. The Los Alamos Wing 9 Facility^{1,2} has two hot metallography trains, one each for U and Pu. The two systems do not differ greatly from other installations around the country. However, because of the special nature and requirements of Pu handling, a few new techniques have been successfully included in the Pu metallography train. These ideas, which are discussed in detail in the sections that follow, have been especially interesting to visitors, engaged in remote work, who have toured the facility.

A. Vibratory Polisher Remotization

Cloth changing on a vibratory polisher normally consists of stretching the polishing cloth, wrinkle-free, over the hold-down ring and clamping it in place with some type of band. The remote handling problems associated with this procedure are not trivial.

The cloth changing technique used at Wing 9 consists of sealing the polishing cloths to a 1/8" thick lucite disc with chloroform. The sealing is done outside the cell to guarantee that the cloth nap has not been sealed to the disc, and that the cloth surface is wrinkle free. The disc is slotted on the side opposite the cloth at three places. The slots serve two purposes: 1) they permit any liquid trapped between the disc and the bowl to be squeezed to the center or edge of the disc and 2) they weaken the

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disc so that it can be easily broken into six pieces with the manipulators. The six pieces may be placed in gallon paint cans for easy disposal. The cloth disc has a 3/4" diameter hole in the center which also serves two functions: 1) it permits liquid trapped beneath the disc to flow into the main liquid volume above the disc and 2) a lifting lug may project through the hole to facilitate handling of the cloth disc.

The remote phase of cloth changing consists of removing the old disc and dropping a new one into the polisher bowl. The hold down ring is lowered onto the new disc. Finally a dust cover is placed on top of the hold-down ring, and the whole assembly (bowl, disc, hold-down ring and dust cover) is locked together with three spring loaded toggle clamps.

The polisher bowl dust cover is also a lucite disc. It is one of the polisher parts which is clamped to the bowl during polishing. Since the dust cover must be in place to lock the hold-down ring to the polishing cloth, the polisher will not operate properly without it. Therefore an advantage of the system is that it insures that the dust cover is always used. When it is clamped in place, it seals the system and thus minimizes liquid spillage from the bowl. Built into the dust cover is a paddle which projects into the bowl and which may be rotated by an external handle. Its purpose is to assist the vibratory rotating action of the polisher when necessary (which may be inadequate due to the deep nap of a new cloth, for example).

The various parts of the vibratory polisher are shown in Fig. 1. One other minor innovation not shown is the use of ball plungers in the place of set screws to hold and center the mounted specimens in the mount weights.

B. "Eye-Wash" Chemical Etching

Figure 2 shows a diagram of a chemical etching technique used at Los Alamos. It consists of passing the etchant across the face of a metallographic mount in a semi-sealed system. The driving force may be gravity,

mechanical pump or suction system. The acid wash is immediately followed by a water wash flowing in the same line and thus rinsing the acid out of the system. By metering the acid volume the etching time can be closely controlled. Proper choice of line size permits etching with a minimum amount of acid. A major advantage of the system is that acids are never stored in the cell, and acid fumes in the cell are minimized (with respect to the quantity generated due to etching and with respect to the time the cell is exposed to the fumes).

If the etchant has a long shelf life so that quantities of it may be prepared in advance, it may be stored external to the cell in safe containers and connected to the "eye-wash" etcher by a suitable valve system.

C. Alpha Sealed Metallograph Viewing

Decontamination of alpha contaminated equipment is complicated by the low permissible exposure limits and the difficulty of detection. These problems, added to the intricacies of a metallograph and the limited decontamination procedures suitable for use on a metallograph, led to the alpha-sealed metallograph viewing technique in use at Wing 9.

Figure 3 shows the alpha seal system. A large diameter (15") convoluted boot is sealed to the alpha box ring with a hose clamp. The smaller diameter hole, 3", in the center of the boot is similarly sealed to a metal ring. The boot is made of thin cast PVC. The convolutions of the boot accommodate the x and y motions of the metallograph stage over which the boot is centered. The small diameter seal ring (clamped to the center hole of the boot) has two "O" ring grooves machined on its inside diameter surface. The two "O" rings provide the seal between the mount holder and seal ring and yet permit the mount holder to rotate within the seal ring.

Figure 3 also shows the construction of the mount holder. The mount holder has two holes drilled into the bottom, 180° apart. These holes engage two identically positioned pins which are attached to the metallograph stage. The stage pins serve as the "spanner wrench" which rotates the mount holder (and specimen) in unison with the stage while the alpha box with boot attached remain stationary.

The fit between the mount holder and the metal seal ring is critical since metal surface roughness or excessive "O" ring squeeze make the stage rotation action too stiff. In practice the outside surface of the mount holder is given a polished finish, and the "O" ring squeeze on 0.070" cross section "O" rings is 0.005".

The transparent viewing window is glued to the perimeter of the hole in the bottom of the mount holder. It is 1-1/4" in diameter and 0.004" to 0.006" thick. Several materials have been tried for this service including transparent PVC, mylar, cellulose acetate and microscope slide cover glass. Both the acetate film and cover glass are optically satisfactory, and have been used successfully. Both have drawbacks due to radiation damage. The acetate film becomes brittle with time-due primarily to irradiation. The cover glass browns with radiation and by its nature is brittle. The windows are relatively easy to replace and last for about a month. Thin Homalite film is on order and, if optically satisfactory, should significantly increase the life of the window beyond that presently expected.

At magnifications greater than 50x surface scratches or mars on the window material are not in focus and do not interfere with the metallographic examination (although immersion oil is sometimes used between the specimen and the window to improve optical contact). A thin film is essential to accommodate the short working distance of high power objectives.

An additional advantage of the seal between the specimen and the metallograph is that chemical etching may be performed in the same alpha-gamma box that serves the metallograph without damage to the equipment and lenses from acid fumes.

D. Specimen Recovery from Mounting Plastics

Plutonium metallography at Wing 9 to date has been in support of the LAMPRE³ reactor which uses concentrated Pu alloy fuels. Consequently a single metallographic specimen may contain 10 - 15 grams of plutonium. Recovery of this much material is mandatory.

The recovery technique in use consists of dropping the specimens in hot (350°C) oil for about 1/2 hour. Under these conditions the epoxy mounting material soaks up oil, swells, and becomes rubbery, yet somewhat brittle. With a little probing the specimen pops out leaving the mounting plastic behind. It should be noted that the mounting plastic will separate easily from the specimen only while it is hot. If allowed to cool, the plastic becomes hard and must be reheated in oil in order to recover the specimen.

Cooking oils, such as Mazola and Wesson Oil, have sufficiently high flash points (400°C) to meet the 350°C required by the process. The oil may be reused several times, and the entire process may be performed in an open beaker without fire hazard. The epoxy materials release noxious fumes which should be directly drawn off in a fume hood to prevent deposition of the vapors on surrounding equipment.

Of the epoxy materials thus far tested, viz., Maraglas, Epon 815 and Hysol, all are susceptible to the technique. Bakelite, of course, remains relatively unaffected by exposure to oil at this temperature.

E. Sample Holding Fixture for Use with an Abrasive Cut-Off Wheel

The abrasive cut-off wheel in combination with the holding fixture

provides an easy method for properly preparing small odd-shaped ceramic and other specimens for metallography with a minimum of polishing and grinding.

Figure 4 shows a holding fixture used at Wing 9 in conjunction with a water cooled abrasive cut-off wheel. It mounts to a vise which is part of the basic cut-off machine. The fixture consists of two identical halves which grip the sample to be cut on both sides of the cut. The cut-off wheel passes between the two halves when cutting through the sample. After cutting, both sample pieces (even ones as short as 1/16") are still gripped by the fixture, and thus catching the sample after the cut is not a problem. "Stops" on the fixture locate it properly in the vise so that the cutting wheel automatically cuts at the correct location. The capacity of the fixture is 1-1/4" diameter and up to 1-1/4" long. The size corresponds to the metallographic specimen mount size in use at Wing 9.

The cut-off machine vise is designed to handle relatively large, regularly shaped specimens. Since there are frequent requirements for cutting a variety of odd-shaped small samples for metallographic examination, the specimens are mounted for metallography and cut in the exact zone of interest by the cut-off wheel before the start of polishing and grinding. This is accomplished by properly positioning the mount in the fixture with the calibrated screw thread. The metallographic mount serves the function of converting small odd-shaped specimens into a size and shape which is relatively easy to handle.

1. Lilienthal, James R., "Los Alamos Alpha-Gamma Cells," Proceedings of the Seventh Conference on Hot Laboratories and Equipment, pp 292 - 304, April 7 - 9, 1959.
2. Burwell, C. C., Schulte, J. W. and Wilson, M. T., unpublished report.

FIG. 1 REMOTIZED VIBRATORY POLISHER

FIG. 2 "EYE WASH" ETCHER

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FIG.3 ALPHA SEALED METALLOGRAPH VIEWING SYSTEM

FIG. 4 SAMPLE HOLDING FIXTURE

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