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SPECIMEN PREPARATION OF IRRADIATED MATERIALS FOR EXAMINATION IN THE ATOM PROBE FIELD ION MICROSCOPE

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The atom probe field ion microscope (APFIM) is well suited to the characterization of the fine scale features and defects that are formed in materials due to exposure to neutron irradiation.[1-3] However, in order for the technique to be effective, suitable specimens are required. Atom probe field ion microscopy specimens are in the form of ultrasharp needles that are usually produced by a series of mechanical and chemical or electrochemical methods. These needles have a typical end radius of approximately 10 to 50 nm and a taper angle of between 1 and 5°. The small dimensions mean that the specimens are extremely fragile and difficult to handle and do not easily lend themselves to remote operations in a hot cell. The small size and mass of the APFIM specimen has the advantage that the amount of material required is minimal.

A concept in working with irradiated materials is to keep exposure to the operator "as low as reasonably achievable," (ALARA). Basic protective measures used to reduce exposure during the preparation of specimens include minimizing the time in the field of radiation, minimizing the amount of radioactive material, maximizing the distance from the source of radiation, and using appropriate shielding whenever possible. Time is minimized by preplanning the entire process so that the number of steps is reduced to a minimum. Variables such as the optimum solutions and electropolishing conditions are fully developed on unirradiated material, and all necessary items are in place before the radioactive samples are introduced to the work area. Measures designed to maximize distance include making all controls (such as on the electropolishing power supply) remote, using tweezers to handle the specimens, and using special tools for standard operations such as crimping the specimen in the copper support tube. The operation is conducted within a hood where shielding is provided by the use of lead bricks, two pairs of gloves, safety glasses and protective clothing.

The low-level radioactive specimen blanks to be electropolished are generally in the form of bars measuring approximately 0.5 mm x 0.5 mm x 10.0 mm. These blanks may be cut from samples (such as surveillance samples) that have been irradiated in various fission reactors. However, a better approach is to design the irradiation capsules with pre-made specimen blanks so that all cutting operations are performed prior to the irradiation. In the case of specimens taken from the pressure vessel of the nuclear reactor, the radiation is β/γ with ^{60}Co being the dominant isotope. In terms of the radiation field, a specimen blank exceeding 3 mrem/h at 30 cm is considered to be too radioactive to be handled without a remote hot cell facility. The first stage of the process is to crimp the specimen blank in a copper support tube using a modified vise. The normal two-stage electropolishing process is then performed with a Fischione Model 770 Tip Polisher. In the first stage, a thin layer of electrolyte (typically a 25% solution of perchloric acid in acetic acid) is floated on a denser inert liquid (Galden HT-70 electronic fluid), as shown in Fig. 1. This stage removes material rapidly and produces a necked region in the center of the blank. Polishing is continued in the second stage (a solution of 2% perchloric acid in 2-butoxyethanol) until the weight of the lower half of the specimen blank is too heavy to be supported by the thin neck. The lower half is recovered and crimped into another copper support tube. Thus, two specimens are prepared from each specimen blank thereby maximizing the number of specimens produced from a blank. During electropolishing, approximately 50 percent of the blank is dissolved in the electropolishing solution, mostly in the first electropolishing stage. It should be noted that because of the amount of specimen that is dissolved, the solutions become a significant source of

radiation and therefore require the same considerations as the specimens. Since the electropolishing solutions and methanol used for rinsing must be disposed of as low-level radioactive waste, it is important to minimize the amount of waste generated. However, this has the adverse side effects of making the radioactivity of the solutions higher. The amount of material handled may also be reduced with the use of a micropolishing technique to repolish blunt or fractured specimens as shown in Fig. 2. In this method, the end of the specimen needle is inserted into a bubble of electrolyte suspended in a wire loop while the electropolishing voltage is applied. By moving the needle in and out of the bubble, the taper angle and the sharpness of the specimen may be adjusted. This method may be applied to specimens produced after the standard electropolishing process or after the specimen has been examined in the atom probe.

Another area that requires more attention than in standard specimen preparation is the control of possible spills and contamination. Control of contamination is achieved through the use of trays placed in the hood to catch dropped specimens and small drops of contaminated liquids that might be accidentally spilled during polishing. It should be noted that blotter paper cannot be used to absorb perchloric solutions as such paper would then be a potential fire/explosion hazard and create a mixed waste problem. Examples of the effectiveness of this method of specimen preparation of irradiated materials may be found elsewhere.[2-4]

References

1. M.K. Miller and G.D.W. Smith, *Atom Probe Microanalysis: Principles and Applications to Materials Problems*, Pittsburgh, Materials Research Society (1989).
2. M.K. Miller, M.G. Hetherington, and M.G. Burke, *Metall. Trans.*, 20A (1989) 2651.
3. M.K. Miller and M.G. Burke, *J. Nucl. Mater.*, 195 (1992) 68.
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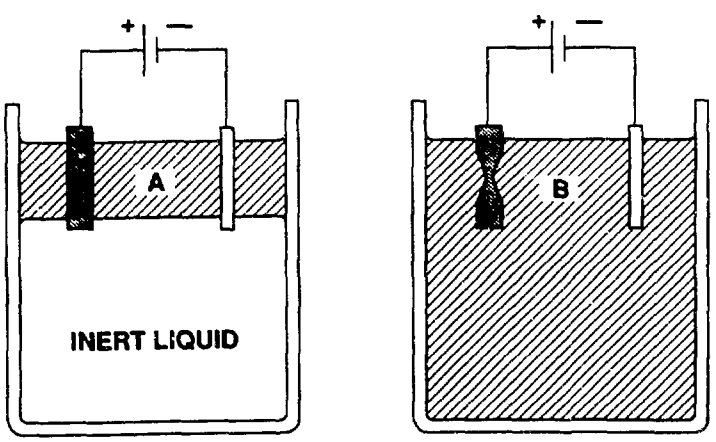

1 STAGE 1 STAGE 2

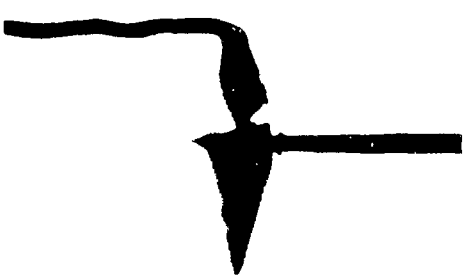

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FIG. 1. -- Schematic diagram of electropolishing. In the first stage, a thin layer (5-7 mm thick) of electrolyte is floated on an inert liquid. This stage produces a neck in the specimen blank. The second stage is continued until the lower part separates thereby producing two specimens.

FIG. 2. -- Micropolishing a blunt of fracture specimen by moving the needle-shaped specimen within a bubble of electrolyte suspended in a wire counter electrode.