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FISSION GAS RE-SOLUTION RATES

Quarterly Report No. 4
April 1 to June 30, 1971

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M. O. MARLOWE

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M. O. Marlowe

ABSTRACT

This fourth quarterly report describes progress in experimental programs and studies to determine the re-resolution rates of fission gases in oxide nuclear fuels under irradiation. An intermediate specimen thinning technique with abrasive blasting was developed for use with the radioactive specimens. Three as-irradiated specimens were thinned by the abrasive blasting technique followed by ion milling in preparation for transmission electron microscopy. Five irradiated specimens were annealed at temperatures in the range 1300 to 1600°C. These specimens were pre-thinned to approximately 100 μm thick by metallographic polishing.

INTRODUCTION

This is the fourth quarterly progress report on the status of the experimental program and studies being conducted to determine the re-resolution rates of fission gases in oxide nuclear fuels. A technique and equipment for thinning the irradiated specimens in the intermediate state from approximately 100 μm to approximately 10 μm thickness with an abrasive-blast was developed. The thinning of three as-irradiated specimens by that technique and the subsequent thinning by ion milling is described. The annealing of five irradiated specimens and subsequent thinning by metallographic techniques is presented.

ABRASIVE BLASTING TECHNIQUE FOR INTERMEDIATE SPECIMEN THINNING

To minimize contamination of the ion milling machine, personnel exposure, and specimen preparation time during final thinning of specimens to electron transparency (less than 1000 Å) an intermediate thinning operation is required. That operation is used to reduce the specimen thickness from approximately 100 μm obtained by metallographic polishing techniques to approximately 10 μm , a suitable thickness for thinning by ion milling. Initial attempts at developing an intermediate thinning procedure were previously described.^{1,2} A technique that has proved successful for the intermediate thinning step is as follows.

A stainless steel washer 0.008 cm thick (0.15 cm inside diameter by 0.32 cm outside diameter) is sealed to a brass cylinder which has been machined to accommodate the specimen and light source used in the abrasive thinning step with water soluble cement (polyvinyl alcohol). This seal prevents the light path from being filled with the fine abrasive powder during the thinning operation as well as providing for ease in handling the specimen. The approximately 10 μm thick UO_2 specimen is then attached to the upper surface of the stainless steel washer with a pre-formed ring (approximately 50 μm cross-sectional diameter) of thermal softening adhesive* by heating to approximately 160°C.

An SS White Airbrasive blasting unit is used to thin the specimens. The unit is operated at 80-psi argon and 10 μm alumina is used as the abrasive. Specimen thickness is monitored by watching for the appearance of transmitted red light through the specimen; the light indicates the sample is in the desired thickness range. The eroded fuel and the contaminated abrasive particles are entrained in an air stream generated by a small vacuum cleaner which exhausts through an absolute filter.

* Crystal Bond 509, Aremco Products, Inc., Briarcliff Manor, New York.

The brass mounting block is inserted in a sample holder. This holder includes the nozzle for the abrasive blast at a predetermined distance and position from the specimen surface (approximately 8 cm). The thinning fixture is shown schematically in Figure 1 and in the process of being loaded in Figure 2. Lead bricks and sheet are placed around the sample during the thinning operation for shadow shielding. The thinning fixture, vacuum cleaner, and filter are shown in Figure 3.

Abrasive blasting times of a few minutes (less than 5) are required to thin uranium dioxide specimens over the thickness range noted above. A telescope for observing a magnified image of the specimen during thinning aids in early detection of the red light transmission. Moderate air flow rates through the thinning device are required to avoid fragmentation of the specimen on continued thinning. Reduced flow is achieved by speed control of the vacuum cleaner motor.

After thinning until the red light is observed, the brass block containing the specimen is removed from the thinning device and the water soluble cement is dissolved to free the stainless steel washer and the specimen. The specimen and the stainless steel washer are left bonded together for the final thinning process (ion milling).

To date several unirradiated and three irradiated uranium dioxide specimens have been thinned through the intermediate stage with this technique. The irradiated specimens were the samples designated as N-5, 10-5, and 25-5.

ION MILLING FOR FINAL SPECIMEN THINNING

The final thinning of the unirradiated and irradiated specimens is being performed by using a Commonwealth Scientific IMMI III ion milling machine. Modifications have been made to the specimen holder to facilitate loading of the highly radioactive specimens and to prevent excessive shadowing of the specimen from the ion beam. The specimen is bombarded from both sides by an argon ion beam with the specimen tilted at 15 degrees to the beam. The specimen thinning time has been in the range of 18 to 36 hours.

Several unirradiated and the three irradiated specimens noted above were thinned by the ion milling technique and appear to be suitable for transmission electron microscopy.

ANNEALING OF IRRADIATED SPECIMENS

Five of the irradiated specimens were annealed as follows:

<u>Specimen Designation</u>	<u>Anneal Temperature (°C)</u>	<u>Annealing Time (hour)</u>
10-1	1300	16
10-11	1300	16
10-2	1600	1
10-12	1575	0
25-2	1600	1

The specimens were annealed in a flowing N₂-6% H₂ atmosphere. Failure of a circuit breaker caused termination of the annealing run on specimen 10-12 just short of the desired annealing temperature (1600°C). That specimen was allowed to cool and will be used to obtain a measure of the relative fission gas bubble growth during the heatup portion of the annealing cycle.

The annealed specimens are being thinned by metallographic techniques to approximately 100 µm thick as were the unannealed specimens.

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FIGURE 1. ABRASIVE THINNING DEVICE

FIGURE 2. MOUNTING BLOCK AND SAMPLE HOLDER DURING LOADING OPERATION

FIGURE 2. THINNING DEVICE WITH VACUUM CLEANER AND FILTER ASSEMBLY

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