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Summary Report
ORNL/TM-2002/276

SEM Examination of Uranium Oxide/Substrate Interfaces

DOE Project No. ERD96XJ130

1. General Information and Purpose

GE Reuter Stokes (Twinsburg OH), a manufacturer of radiation detectors and nuclear power plant instrumentation, has requested examination by scanning electron microscopy (SEM) and microprobe of components used in their fission chamber product. The goal is to image the uranium oxide coating and the interface with its substrate. Evaluation of the extent of any interpenetration or mixing between substrate and the coating constituents is of particular interest.

Twelve specimens were supplied, in the form of 0.5-in.-long segments of 0.18-in.-diam. titanium tube with an . 1-μm-thick uranium oxide layer on the inner tube surface.

2. Work Performed

The specimens were epoxy-potted into four specially-prepared, three-hole metallographic mounts. Remote (in-hot-cell) sample preparation began with fine grinding (400 and 600 grit SiC) followed by vibratory (Syntron) polishing using successively 15 μm, 6 μm, and 3 μm diamond compound on Streuers DP-Pan cloths.

Limited optical metallography (of a few of the specimens) was conducted at 500x and 1000x magnifications. All four mounts then received an extremely thin conductive gold coating, applied by sputtering, to permit their examination in our Jeol JXA-840A SEM/microprobe.

Different operational parameters were employed for the SEM versus electron microprobe applications, other than a common use of 20 kV for the primary electron beam. Relatively high resolution imaging using secondary electrons (SE) or back-scattered electrons (BE) were conducted using a close working distance (10 mm), small objective aperture (30 μm) and low beam current (0.06 to 0.2 nA) which together permit a fine probe size and spatial resolution of about 0.02 μm.

However for generating maps of chemical distributions, the microprobe mode requires a longer working distance (39 mm), large (100 μm) aperture, and higher beam currents (6 to 10 nA) which degrade even the SE and BE imaging resolutions [when acquired along with x-ray maps] to the order of 0.1 μm , and since x-rays for the various elements are generated from an enlarged volume below the surface (owing to electron scattering within the material), the x-ray mapping spatial resolution is coarser yet.

For this work, the light element x-ray signals (oxygen Kα line at 0.525 keV, carbon Kα line at 0.277 keV) were analyzed using an LDE-2 diffracting crystal, while a PET crystal was used for the titanium $K\alpha_1$ (4.508 keV) and uranium $M\alpha_1$ (3.170 keV) x-ray emissions. These crystals were installed in the two Wavelength-Dispersive Spectrometers (WDS) that are components of our JXA-840A instrument.

3. Results

A summary of all the images obtained is provided in Table 1, “SEM and Microprobe Data Obtained per Specimen”, For SEM imagery of the coatings’ physical condition, two locations (“a” and “b”, generally on opposite sides of the tube) were examined for each specimen. Usually four magnifications (300x, 800x, 2000x, and 5000x) were employed and both SE and BE images were acquired at each magnification. Secondary electron (SE) images offer the finest detail and depth representation, whereas the back-scattered electron (BE) signal varies by chemical composition – brighter for heavier (greater Z) elements.

Microprobe examination, conducted on three designated specimens at three or four additional locations (“c” through “f”), was done at 2000x and 5000x magnifications. Typically an SE or BE image was obtained simultaneously with a high-Z (uranium) and a low-Z (oxygen) element-map generated by the two WDS spectrometers. A few maps showing the location of the substrate (titanium) and the mounting medium (carbon-based) were also obtained.

Frequently, it was also possible to generate an elemental “profile plot” which semi-quantitatively represents the signal intensities for one or two elements at a horizontal traverse across the middle of the corresponding map-image. This data has been translated into Excel data files (.xls type) and presented as line graphs.

The results were transmitted by e-mail during the course of the project [images in “.jpg” format and profile plots in “.xls” format]. That data, together with the original image files in “.tif” and profile plots in “linescan” (“.lns”) formats, are all collected on a CD-ROM data disk, to accompany this report.

Telephone discussion on 5 September 2002 with the sponsor affirmed that these SEM and elemental map images have provided an answer to the main question of coating behavior for the various preparation conditions represented by the submitted samples. That is, the SEM images

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documented the physical condition of the different coatings, while the elemental maps and profiles consistently indicated the co-location of both uranium and oxygen within the coatings and negligible penetration of either element into the substrate.

Table 1. SEM and Microprobe Data Obtained per Specimen

SEM Images

Mount No.	GE-RS spec.no.	location	no. of images
6190	25	a	7
		b	7
"	40	a	7
		b	7
"	WRNM	a	7
		b	7
6191	3	a	8
		b	8
"	5	a	8
		b	8
"	11	a	8
		b	8
6192	9	a	8
		b	8
"	46	a	8
		b	8
"	4R6	a	8
		b	8
6193	27	a	5

Microprobe Element-Map Data

Mount No.	GE-RS spec.no.	location	no. of images	profile plots
6191	5	c	7	6
		d	6	4
		e	8	6
		f	10	8
"	11	c	12	8
		d	9	5
		e	10	6
6192	9	d	7	-
		e	9	8
		f	9	8
"	4R6	c	8	10

		b	7
“	33	a	4
		b	7
“	37	a	5
		b	7