

## Degradation of a N06690 Borescope in a Radioactive Waste/Glass Melter System (U)

by

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## Degradation Of A N06690 Borescope In A Radioactive Waste/Glass Melter System

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### Abstract

Radioactive liquid waste from nuclear materials production processes will be vitrified in the Defense Waste Processing Facility (DWPF) melter. The melter borescope outer housing, fabricated from N06690, was severely degraded by the combined effects of corrosion and oxidation after only five months of non radioactive operation. The melter was idled and not being fed over 85% of the time during the cold run operations. The borescope was designed to perform in an oxygen rich, chloride containing environment with temperatures approaching 900°C (1650°F). The housing was designed for a minimum of two years of continuous service in the DWPF melter. Air and steam were purged through the borescope and swept over the optics assembly to keep molten glass and volatile gases from depositing on the lens cover. Upon exiting the borescope the air passes through a N06690 orifice and enters the melter. Severe oxidation was observed around the orifice. Extensive material loss was also observed on the side of the outer housing which protrudes through the dome of the melter. Redesign of the borescope is currently underway and will include a new set of optics that will allow the size of the orifice to be significantly decreased, thus reducing the amount of air necessary to keep the lens cover clean. Application of a duplex diffusion coating, consisting of chromium and aluminum, on the end of the borescope outer housing and the use of an inert gas purge are also being considered.

THROUGHOUT FORTY PLUS YEARS of operation at the Savannah River Site (SRS), high activity radioactive waste has been collected and temporarily stored in large underground storage tanks. In preparation for long-term disposal, the waste will be vitrified and encapsulated in stainless steel canisters at the Defense Waste Processing

Facility (DWPF). The stabilized radioactive waste will be stored at the SRS until a suitable repository for long-term storage is identified and approved. A total of 125 million liters (33 x 10<sup>6</sup> gal.) of liquid waste will be processed in the DWPF over the next 25 years.

Selection of materials for use in the DWPF included laboratory scouting tests, bench top tests and exposure in scale melter systems (1-3). The selection of N06690 as the primary material of construction for both internal and off gas melter components was made because of its resistance to molten glass attack, sulfidation and oxidation. The performance of N06690 was confirmed in the Integrated DWPF Melter System (IDMS), a 1/10 scale version of the DWPF. Although some of the IDMS melter components experienced minor oxidation and sulfidation attack, all appeared satisfactory for a two year life in the DWPF melter.

In preparation for radioactive start up of the DWPF later this year, "cold" runs have been initiated. Non-radioactive synthetic sludge (feed material) for the cold runs contained reference levels of all materials, including chlorides, sulfates, formates and nitrates. The synthetic sludge is first blended with a borosilicate glass frit and formic acid in the Feed Preparation System. After being refluxed and concentrated it is fed to the melter. Once in the melter, excess liquid is boiled off and the feed solution is melted at temperatures approaching 1150°C (2100°F) producing a homogeneous glass which is poured into canisters, fabricated from S30403 stainless steel. As this sludge frit mixture is heated, organics are evolved. To ensure complete combustion of these organics, air is injected into the melter through the backup off gas film cooler and the borescopes. Off gases containing chlorides and other halides, sulfates and entrained glass, exit the melter through the primary off gas system.

The melter is refractory lined and is approximately 3.3 meters (11 ft.) high with an internal diameter of approximately 1.8 meters (6 ft.) and an internal height of 2.1 meters (7 ft.). The outer shell is fabricated from S30403

stainless steel while all internal top head and off gas components, i.e. thermowells, level probe, borescopes, drain valve, feed tube, and film cooler, are fabricated from N06690, Figure 1.

Melter components must withstand the high temperature corrosive environment. During idle times, when the melter is not being fed, the vapor space temperature can approach 900°C (1650°F). During feeding, vapor space temperatures can be as low as 650°C (1200°F). In the past 5 months of cold run operation the melter has been idled over 85% of the time. Air was still injected through the film cooler and borescopes during this time.

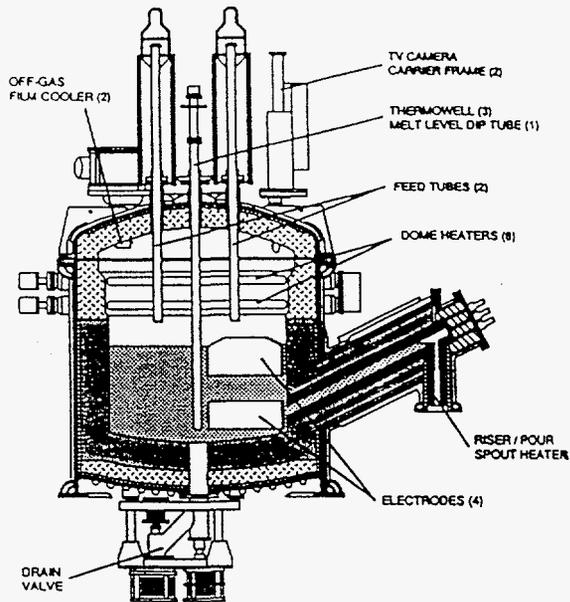


Fig. 1 - Schematic of DWPF melter.

### Borescope Design and Operation

Two borescopes are inserted through the top head of the melter and are positioned in such a fashion to allow remote viewing of the cold cap, feed tubes, and melter side wall. The borescope consists of a N06690 outer housing, 81 cm (32 in.) long by 8.9 cm (3.5 in.) with a nominal wall thickness of 0.953 cm (0.375 in.), and a central S30403 stainless steel optics assembly, with approximately 6.4 cm (2.5 in.) outer diameter, Figure 2. The stainless steel optics assembly is cooled internally with plant air. This cooling air is not discharged into the melter. Air is also continually injected between the optics assembly and the outer housing at approximately 0.20 m<sup>3</sup>/min (7 SCFM). This air sweeps over the end of the optics assembly and is intended to keep molten glass and volatile gases from depositing and/or condensing on the lens cover. Steam is also injected periodically for a duration of approximately two minutes to remove any deposits which may have built up on the lens cover. Originally the steam was to be used on a monthly basis; however, deposits built up faster than expected and

degraded the signal so the frequency of steam purges was increased to every half hour.

Both the air and steam exit the borescope and enter the melter through a 1.9 centimeter (0.75 in.) orifice. The orifice is designed with concentric steps to create turbulence and a slight back pressure. The turbulence was intended to minimize degradation of the lens cover by preventing aspiration of particles and hot gases back through the orifice.

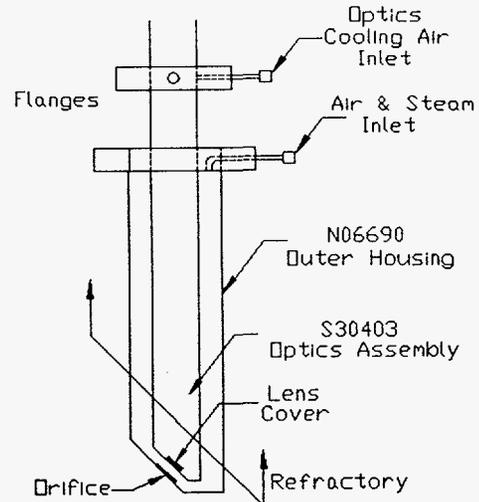


Fig. 2 - Schematic of borescope.

### Metallurgical Evaluation

Degradation of the borescope outer housing revealed numerous pits and heavy scales. Deposits of varying colors, black, red, yellow and white were observed, Figure 3. The most severe spalling of material appeared around the orifice where the air and steam exit. Cracks were observed emanating radially outward from the inner diameter of the orifice in all directions. The bottom of the borescope was covered mainly with black glassy deposits and heavy oxide scale, Figure 4.



Fig. 3 - Photograph of failed borescope.

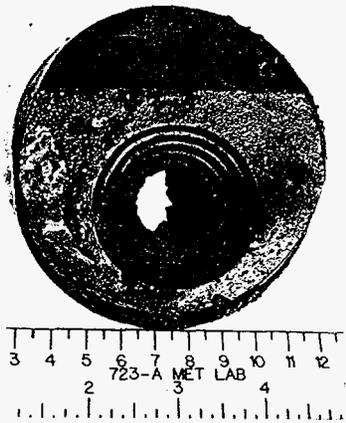


Fig. 4 - Photograph of orifice at end of the borescope outer housing.

The deposits and scale from around the orifice were identified using X-ray diffraction, and were found to be comprised mainly of chromium and nickel oxides and a sodium aluminum silicate sulfate, Figure 5. Significant internal oxidation was evident in this region. Chromium trioxide was not identified. A micrograph of material sectioned from around the orifice is presented in Figure 6.

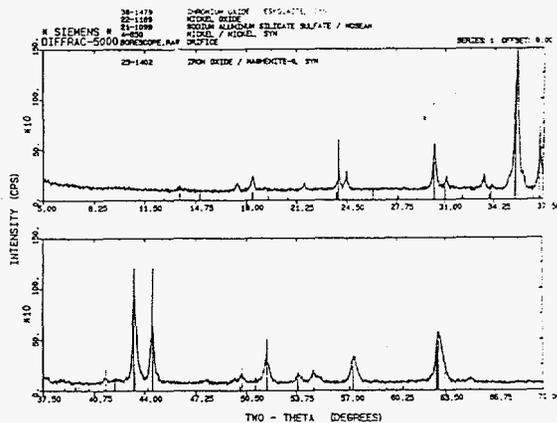


Fig. 5 - X-ray diffraction spectra of deposits and scale sampled from around the orifice. Results are superimposed over an iron spectra.



Fig. 6 - Micrograph of region around orifice exhibiting internal oxidation. Voids contain a chromium oxide layer and in some areas formed a continuous path to the surface.

A large but localized region of degradation, approximately 7.6 cm (3 in.) in diameter and over 0.38 cm (0.15 in.) in depth, was observed on the side of the outer housing facing the center of the melter. The affected region was near the point where the borescope passes through the refractory and enters the melter. The degradation exceeded 9.1 mm/yr (360 mpy). This would lead to a through wall penetration in just over one year. Chromium oxide, sodium chloride and potassium sulfate were identified by X-ray diffraction in the surface deposits. Intergranular attack and internal oxidation were not observed in this region. However, second phase precipitates decorated the grain and twin boundaries near the attacked surface, Figure 7. Energy dispersive spectroscopy (EDS) using a scanning electron microscope (SEM) revealed a significant depletion of chromium (from greater than 28 wt% to less than 10 wt%) and enrichment in nickel, Figures 8 and 9. The inner diameter of the housing, except the degraded region around the orifice, did not show any evidence of chromium depletion or oxidation. The original machining marks were still evident on all the exposed internal surfaces.



Fig. 7 - Micrograph of region sectioned from the severely degraded region on the side of the borescope. Upper left corner shows the outer diameter of the housing. No intergranular attack was observed.

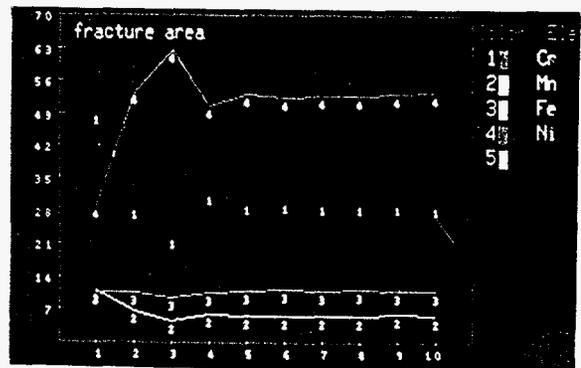


Fig. 8 - EDS spectra showing chromium depletion in region adjacent to the chromium oxide layer. A corresponding increase in the nickel concentration is also evident. First point of scan was taken on the chromium oxide layer on the surface.

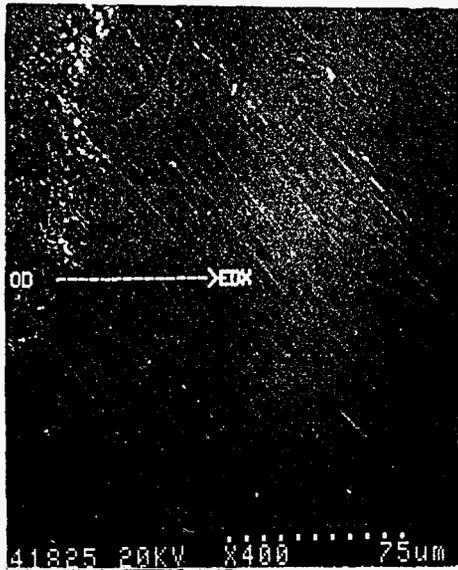


Fig. 9 - Scanning electron micrograph showing location EDS point scan on polished sample taken from around large pit.

The S30403 stainless steel optics assembly was in excellent condition. A thin brownish deposit had developed on the lens cover and on the lower eight inches of the optics assembly. Heat tinting gave a slight bluing to this region. The lenses and other internal components did not suffer any damage from the high temperature exposure.

### Discussion

Degradation of the DWPF borescope resulted from high temperature oxidation of the N06690 material surrounding the orifice. Metal temperatures would be expected to be higher around the orifice than in any other portion of the borescope since it is closest to the lid heaters and the molten glass. The severe internal void formation and spalling of material is characteristic of oxidation damage (4). Attack in this region ultimately led to the failure of this component.

The absence of a cold cap on the molten glass during idle mode resulted in excessively high temperature in the vapor space. The elevated temperature combined with the oxygen and chloride rich environment surrounding the borescope contributed to the severe degradation observed on the side of the housing. Thermal fluctuations of the borescope caused by frequent purges of steam may have also contributed to the degradation process by causing spalling of the protective chromium oxide layer, thus accelerating the oxidation process. Oxidation in this region was not anticipated because N06690 product literature shows that temperatures above 1000°C (1832°F) are required for such attack (5). Therefore, chlorides may have played a significant role in the degradation of this material. Evaluation of the effects of chlorides on the degradation of this material is continuing.

The S30403 optics assembly was not significantly affected by the elevated temperature exposure. The air

cooling was adequate to protect the optics and other internal components from degradation. However, deposits on the lens cover were of sufficient thickness to cause a noticeable degradation in the camera signal.

A redesign of the borescope was recommended to provide a reduction in the diameter of the orifice and a reduction in the volume of purge air required to sweep past the lens. Use of an inert gas purge is also being considered. A material change was not recommended due to concerns over sulfidation in the vapor space; however, a duplex diffusion coating, consisting of chromium and aluminum, will be applied to the lower portion of the one of the borescope outer housings. Performance of this component will be evaluated after one month of service.

### Conclusions

Results of the metallurgical investigation indicated that the borescope outer housing was severely degraded by the combined effects of corrosion and high temperature oxidation. The degradation process around the orifice resulted in a significant depletion of chromium in the near surface region, internal void formation and spalling of material from around the orifice. Cooling air was sufficient to protect the optics from elevated temperature exposure. A redesign of the orifice is required to minimize the amount of air and reduce the frequency of steam cleaning necessary to keep particulates from depositing on the camera lens cover.

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