## Metallographic Preparation of Space Shuttle Reaction Control System Thruster Electron Beam Welds for Electron Backscatter Diffraction

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A Space Shuttle Reaction Control System (RCS) thruster failed during a firing test at the NASA White Sands Test Facility (WSTF), Las Cruces, New Mexico. The firing test was being conducted to investigate a previous electrical malfunction. A number of cracks were found associated with the fuel closure plate/injector assembly (Fig 1). The firing test failure generated a flight constraint to the launch of STS-133. A team comprised of several NASA centers and other research institutes was assembled to investigate and determine the root cause of the failure. The JSC Materials Evaluation Laboratory was asked to compare and characterize the outboard circumferential electron beam (EB) weld between the fuel closure plate (Titanium 6Al-4V) and the injector (Niobium C-103 alloy) of four different RCS thrusters, including the failed RCS thruster.

Several metallographic challenges in grinding/polishing, and particularly in etching were encountered because of the differences in hardness, ductility, and chemical resistance between the two alloys and the bimetallic weld. Segments from each thruster were sectioned from the outboard weld. The segments were hot-compression mounted using a conductive, carbon-filled epoxy. A grinding/polishing procedure for titanium alloys was used [1]. This procedure worked well on the titanium; but a thin, disturbed layer was visible on the niobium surface by means of polarized light.

Once polished, each sample was micrographed using bright field, differential interference contrast optical microscopy, and scanning electron microscopy (SEM) using a backscatter electron (BSE) detector. No typical weld anomalies were observed in any of the cross sections. However, areas of large atomic contrast were clearly visible in the weld nugget, particularly along fusion line interfaces between the titanium and the niobium. This prompted the need to better understand the chemistry and microstructure of the weld (Fig 2). Energy Dispersive X-Ray Spectroscopy (EDS) was used to confirm the chemical composition of the variations in contrast in these areas.

Niobium alloys generally require exposure to more aggressive chemical reagents than titanium alloys for etching because of niobium's chemical resistance; therefore, the titanium portion of the sample was etched first. A five second immersion in Kroll's reagent revealed a general microstructure on the titanium portion of the sample; however, the titanium heat affected zone closest to the weld, was over-etched due to higher concentrations of refined grains and an increase in βeta-phase. The Kroll's etchant also revealed some microstructure in the weld nugget itself; the niobium portion of the sample remained unetched.

Several niobium etchants were tried on a test piece of niobium C-103, with ASTM E407-161 yielding the best results [2]. The ASTM E407-161 etchant was then used on the thruster EB welds with good results for the niobium; the titanium was significantly over-etched.

To provide investigators with a continuous view of general microstructure across all three distinct regions (weld and parent alloys), the sample was then reground, repolished, lightly etched using Kroll's reagent, and vibropolished in preparation for Electron Back-Scatter Diffraction (EBSD) mapping. EBSD provided an excellent, continuous view of the general microstructure in the aspolished condition. It also revealed crystallographic texture, giving more insight into the solidification patterns, grain size, strain contours, and heat flow direction of the weld during cooling (Figs 3-6).

By combining data collected using optical microscopy, SEM-BSE, EDS and EBSD mapping, investigators were better able to characterize these technically challenging metallographic samples. Ultimately, this correlative microscopy provided the investigators with sufficient evidence to conclude that no pre-existing flaws were present in the fuel closure plate-to-injector welds. Analyses performed in parallel by other members of the investigation team systematically eliminated all other plausible branches of a fault tree, ultimately determining the root cause as a leaking fuel valve upstream of the injector [3]. Combined with additional rationale, the flight constraint was lifted.

## References

- [1] Struers E-METALOG Method #1416 available at: http://www.struers.com/modules/emetalog/generic\_view.asp?method=1416 (Feb 2011)
- [2] ASTM E407 Standard Practice for Microetching Metals and Alloys, Etchants Table 2, Etchants 129, 163, 161.
- [3] The authors would like to gratefully acknowledge our colleagues D. Cone et al, NASA WSTF, Las Cruces NM.

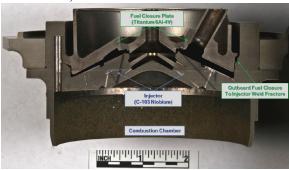


Fig 1. RCS Thruster Injector Cross-Section



Fig 3. SEM BSE Image of Outboard Fuel Closure Weld



Fig 4. EBSD Inverse Pole Figure Map of Outboard Fuel Closure Weld

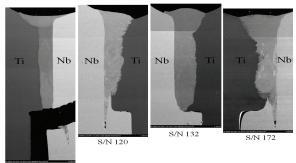


Fig 2. SEM BSE Comparison of 4 Injectors Outboard Fuel Closure Welds



Fig 4. EBSD Pattern Quality Map of Outboard Fuel Closure Weld

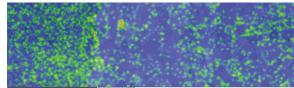


Fig 6. EBSD Strain Contouring Map of Outboard Fuel Closure Weld