

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 beta-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 as-polished 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.

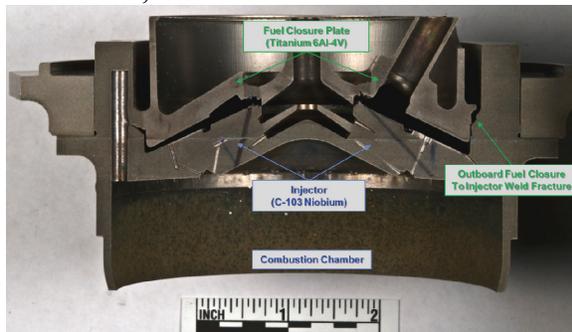


Fig 1. RCS Thruster Injector Cross-Section

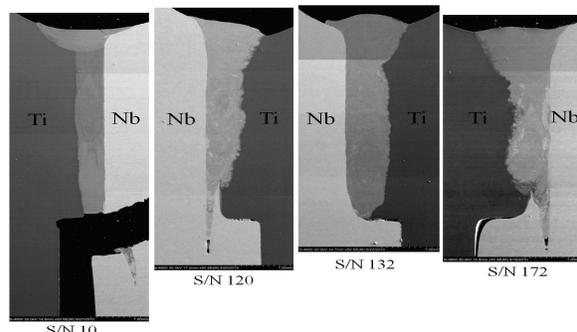


Fig 2. SEM BSE Comparison of 4 Injectors Outboard Fuel Closure Welds

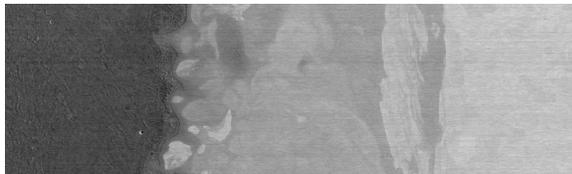


Fig 3. SEM BSE Image of Outboard Fuel Closure Weld

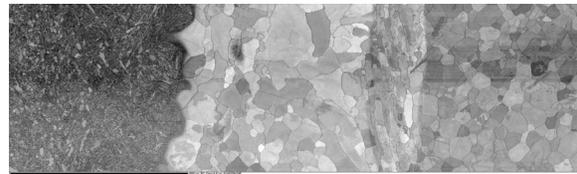


Fig 4. EBSD Pattern Quality Map of Outboard Fuel Closure Weld



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

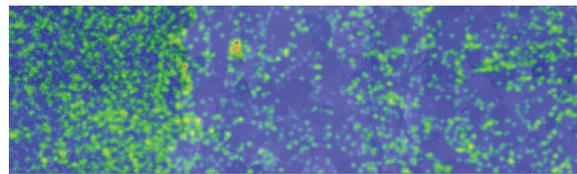


Fig 6. EBSD Strain Contouring Map of Outboard Fuel Closure Weld



Microscopy & Microanalysis 2011

**Symposium A11 – Effects of Metallographic and Other Preparation Techniques
Upon Microstructural Characterization**

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Background



- **On June 17, 2010, an Space Shuttle Reaction Control System (RCS) thruster (S/N 10) failed during a firing test at the NASA WSTF**
 - Firing test was being conducted to investigate a previous Oxidizer Valve Low Current
 - The event occurred during the 4th pulse fire test that day
- **Misfiring event generated a constraint to launch of STS-133**
- **Non-Destructive analysis of thruster found a number of cracks associated with the fuel closure plate/injector assembly**
 - “Blue Crack” – 45° Acoustic Cavity
 - “Red Crack” – Inboard Fracture between Fuel/OX Passages
 - “Yellow Crack” – Ox Tube EB Weld Crack
 - “Green Crack” – Outboard Fuel Closure EB Weld Fracture
- **JSC M&P was asked to evaluate portions of the “Green Crack” and “Red Crack”**

Misalignment of Flange/Plate

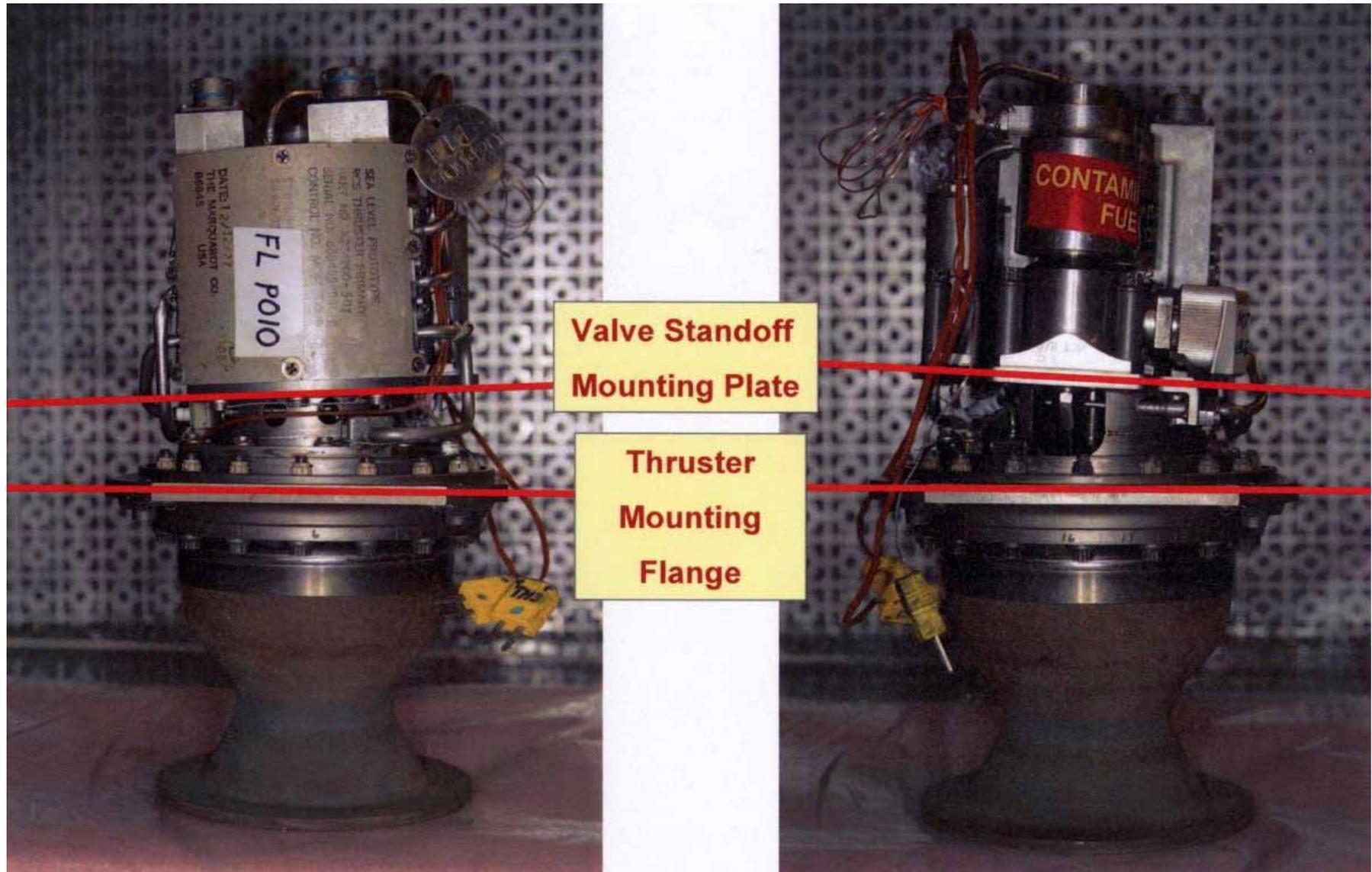


Image source: AE3654: L1L (P010) Investigation Status charts, E. Fitzgerald 08/11/2010, pp 7

Injector Cut-Away View

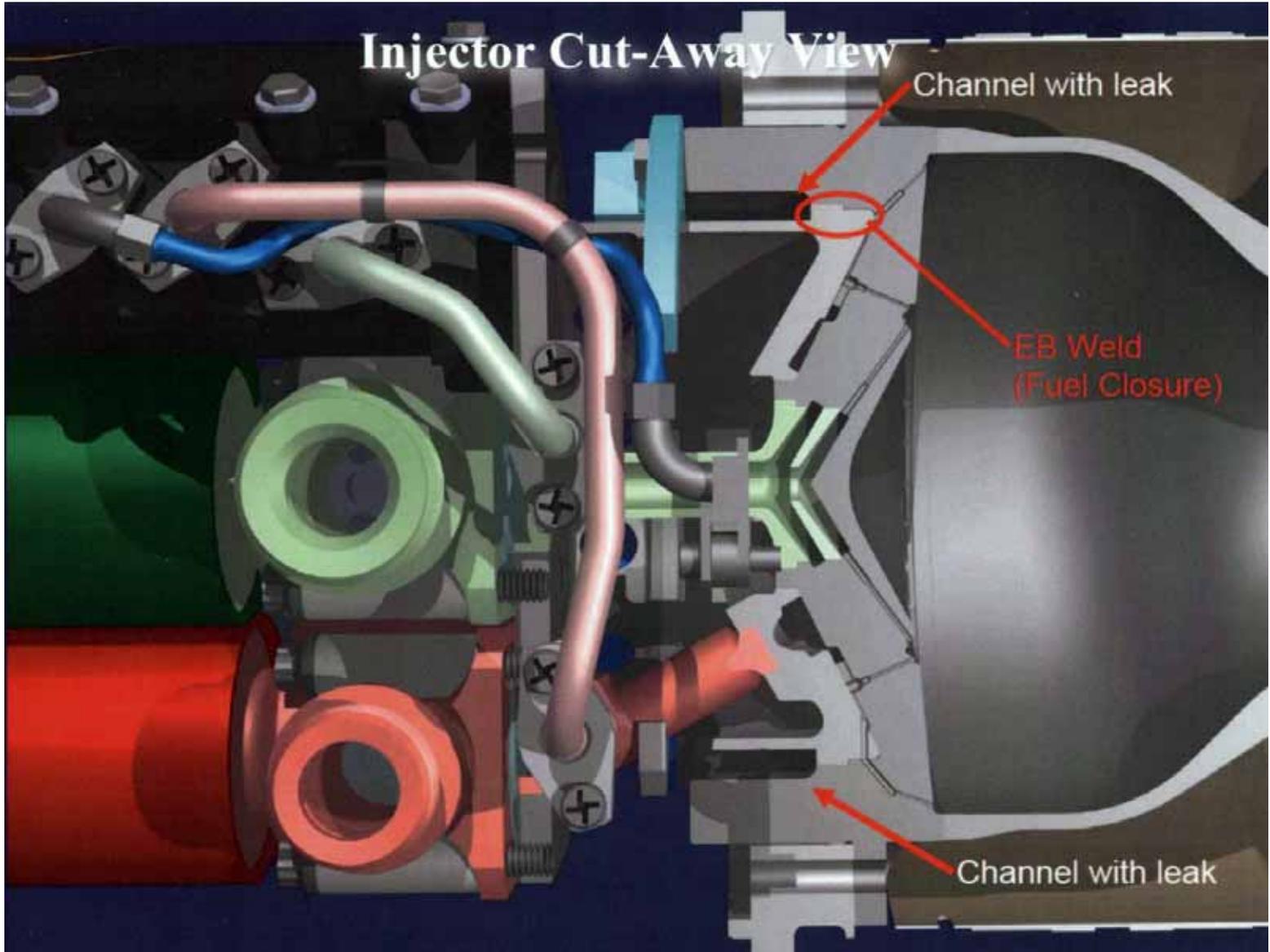
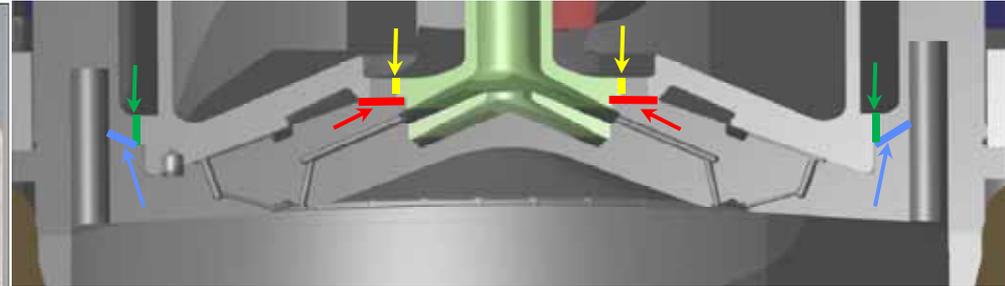
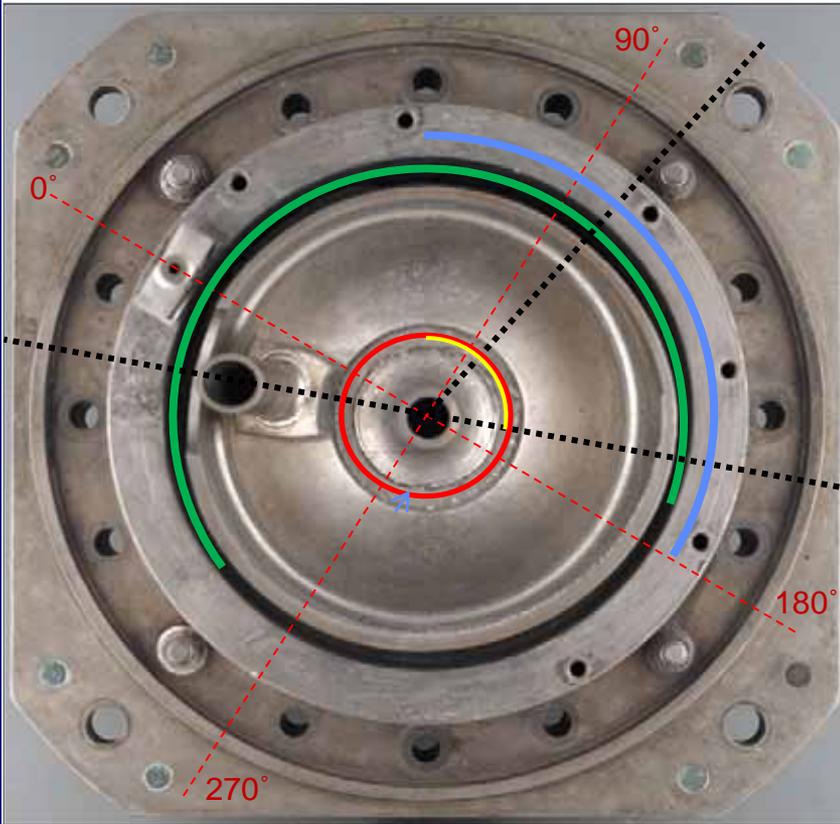
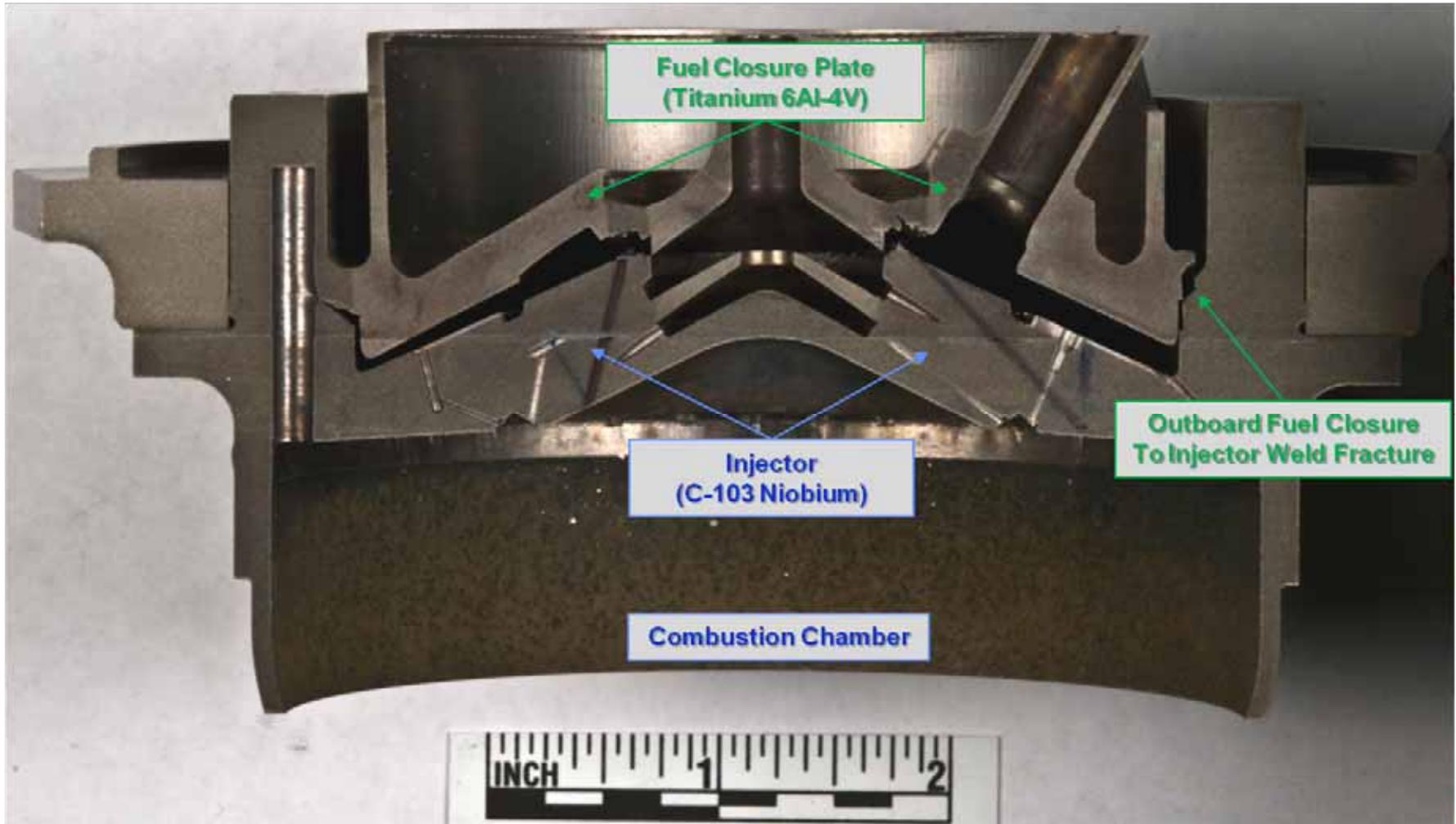


Image source: Orbiter RCS Thruster WSTF Test Stand Failure Investigation

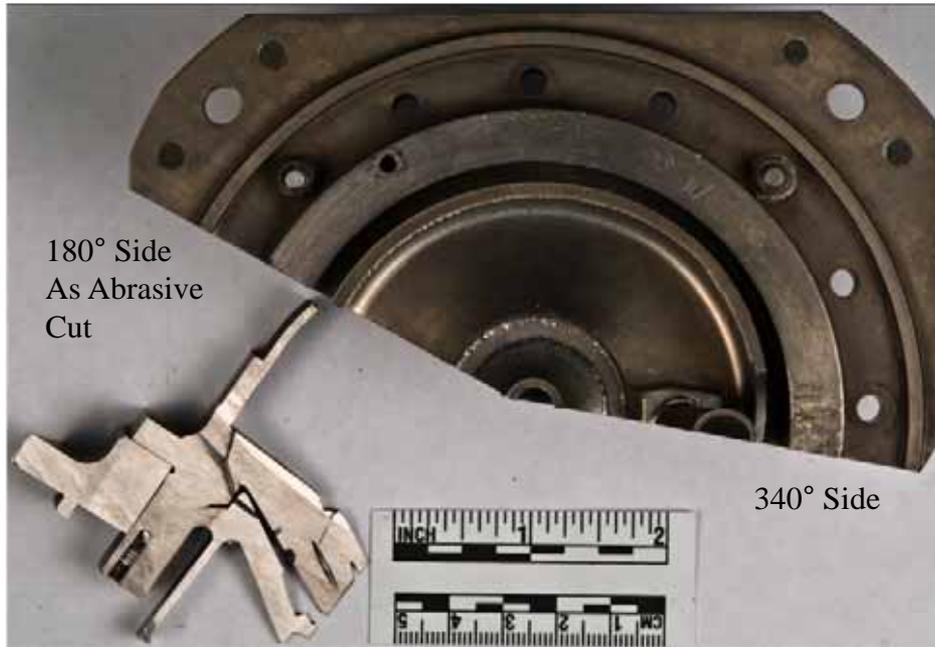


-  '45 Degree' Acoustic Cavity Cracks
(60 -180° CW)
-  Inboard Fracture between Fuel/OX Passages
(0 - 360°)
-  Ox Tube EB Weld Crack
(45 – 160° CW)
-  Outboard Fuel Closure EB Weld Fracture
(300 - 160° CW)
-  EDM Cut Paths

SN 10 RCS Thruster Cross-Section



SN 10 As Cut (1st) Photography

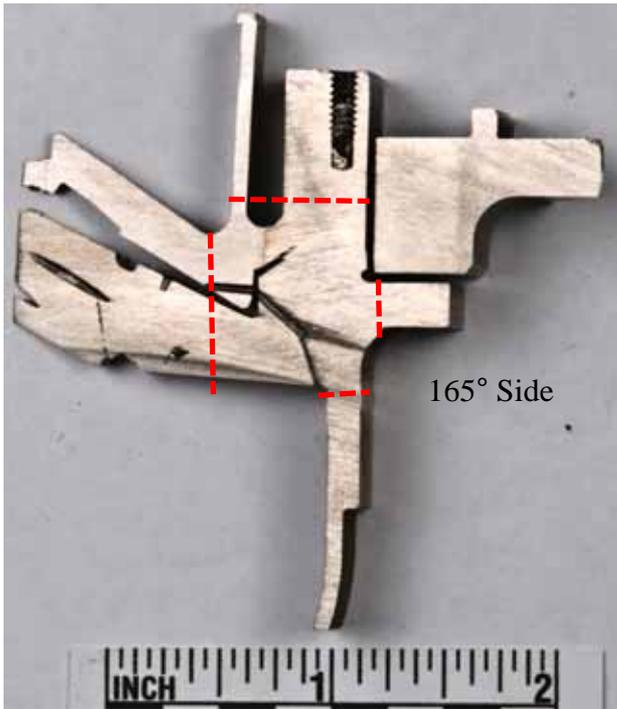


180° Side
As Abrasive
Cut

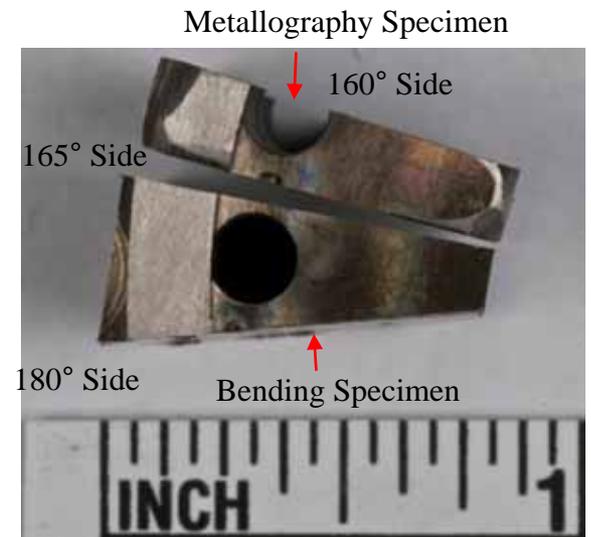


340° Side

SN 10 2nd Cut Photography

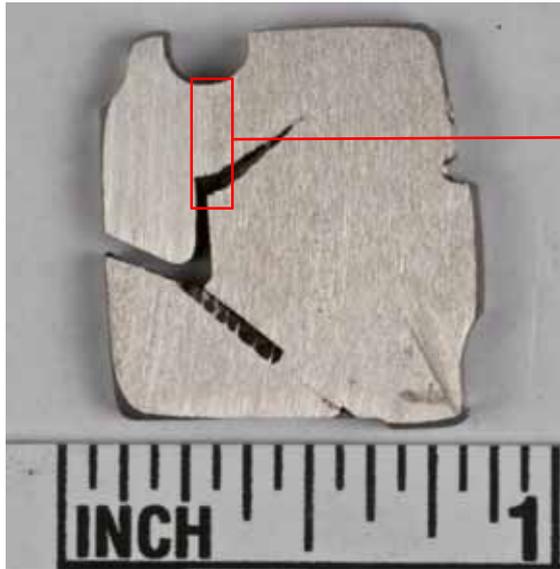


Metallography Specimen

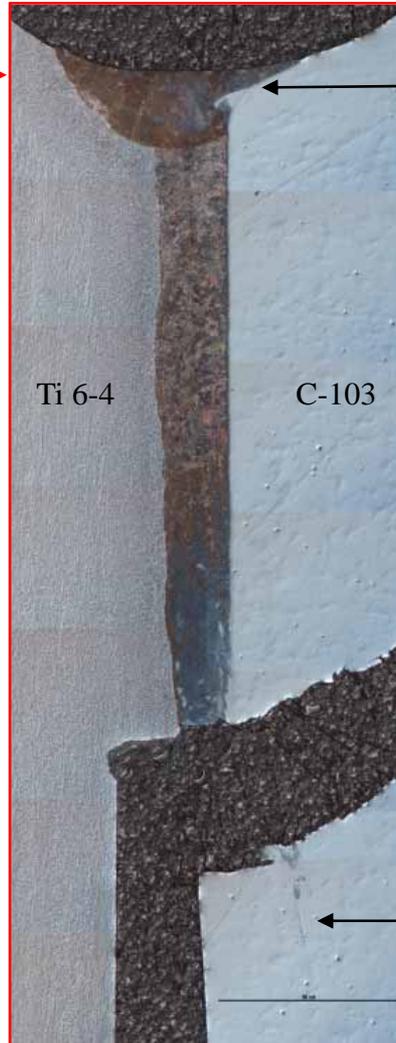




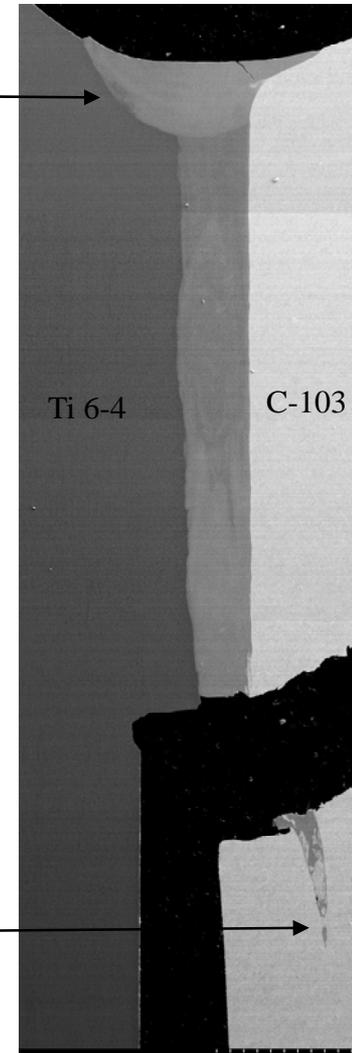
SN 10 OB Fuel Closure Weld Metallography



DIC Optical



BSE SEM

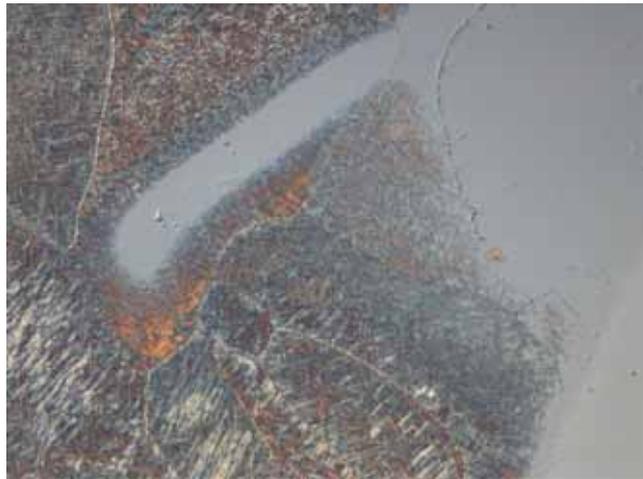


Cosmetic
Weld

EB Weld
Root



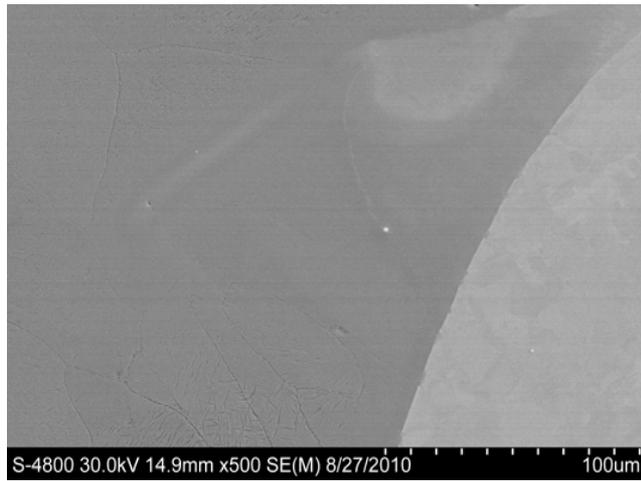
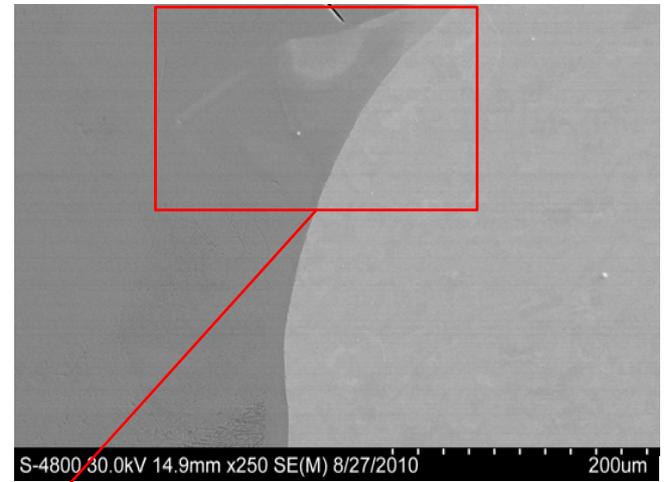
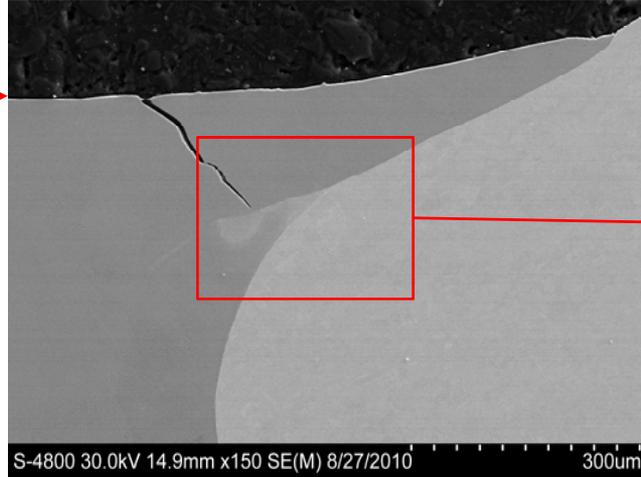
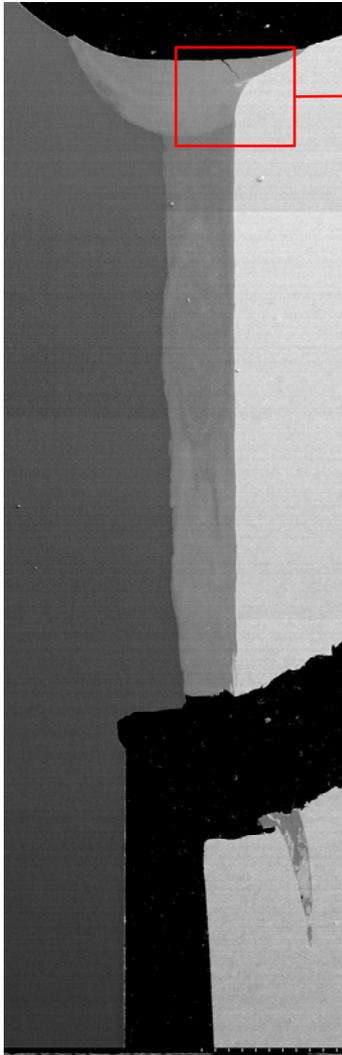
SN 10 OB Fuel Closure Weld Optical Micrographs



Kroll's Etchant
5 sec swab



SN 10 OB Fuel Closure Weld SEM Micrographs (165°)



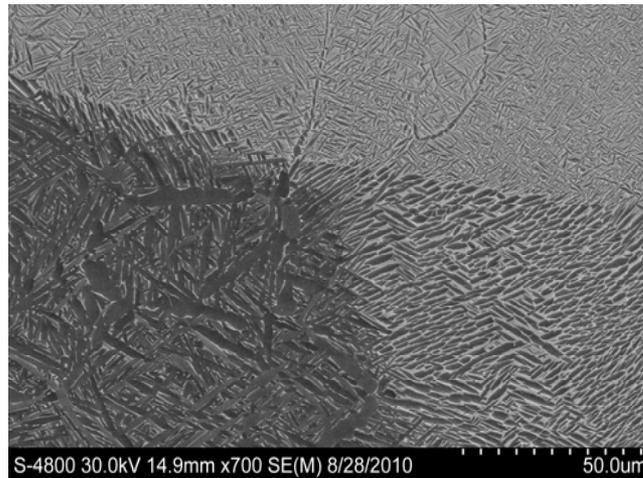
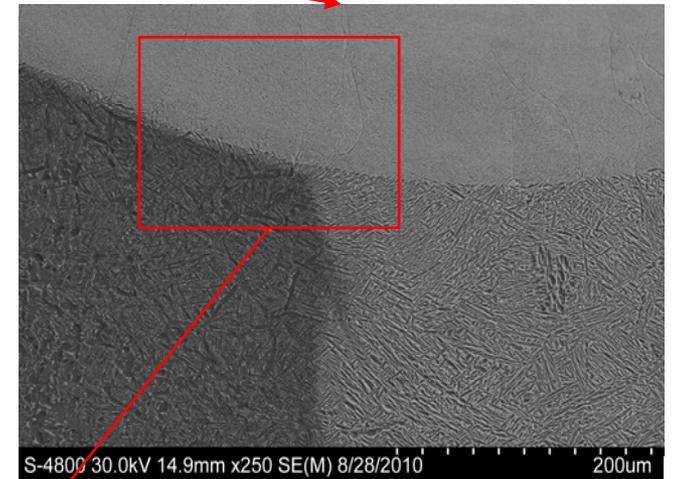
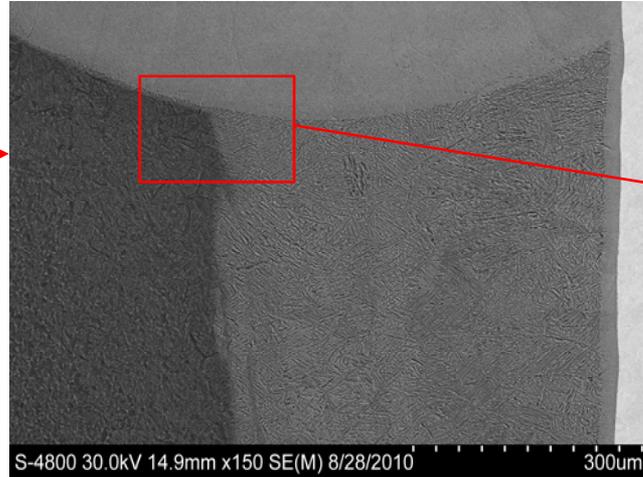
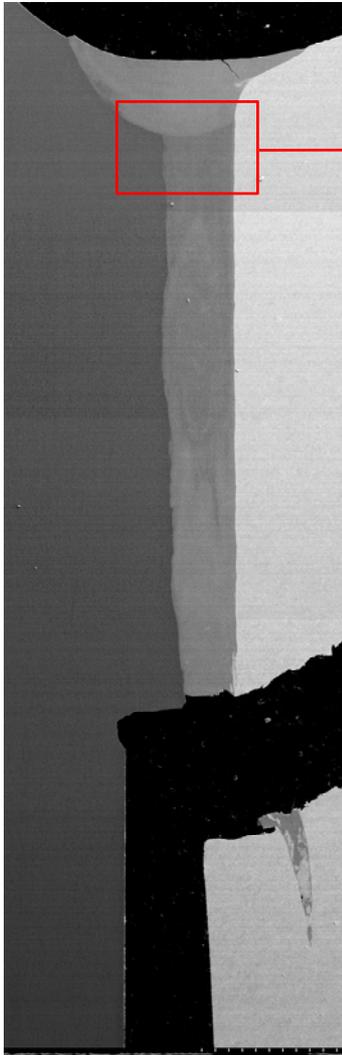


SN 10 OB Fuel Closure Weld Optical Micrographs (165°)



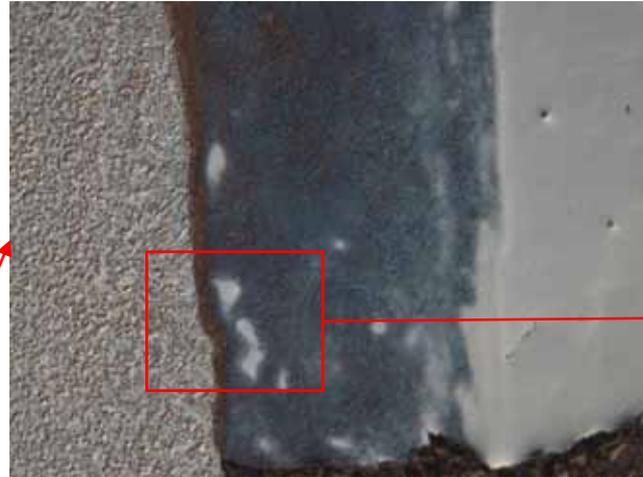


SN 10 OB Fuel Closure Weld SEM Micrographs (165°)



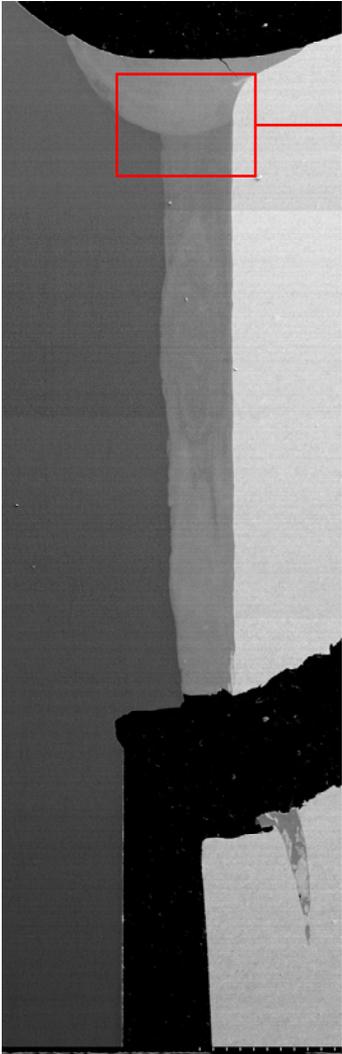


SN 10 OB Fuel Closure Weld Optical Micrographs (165°)



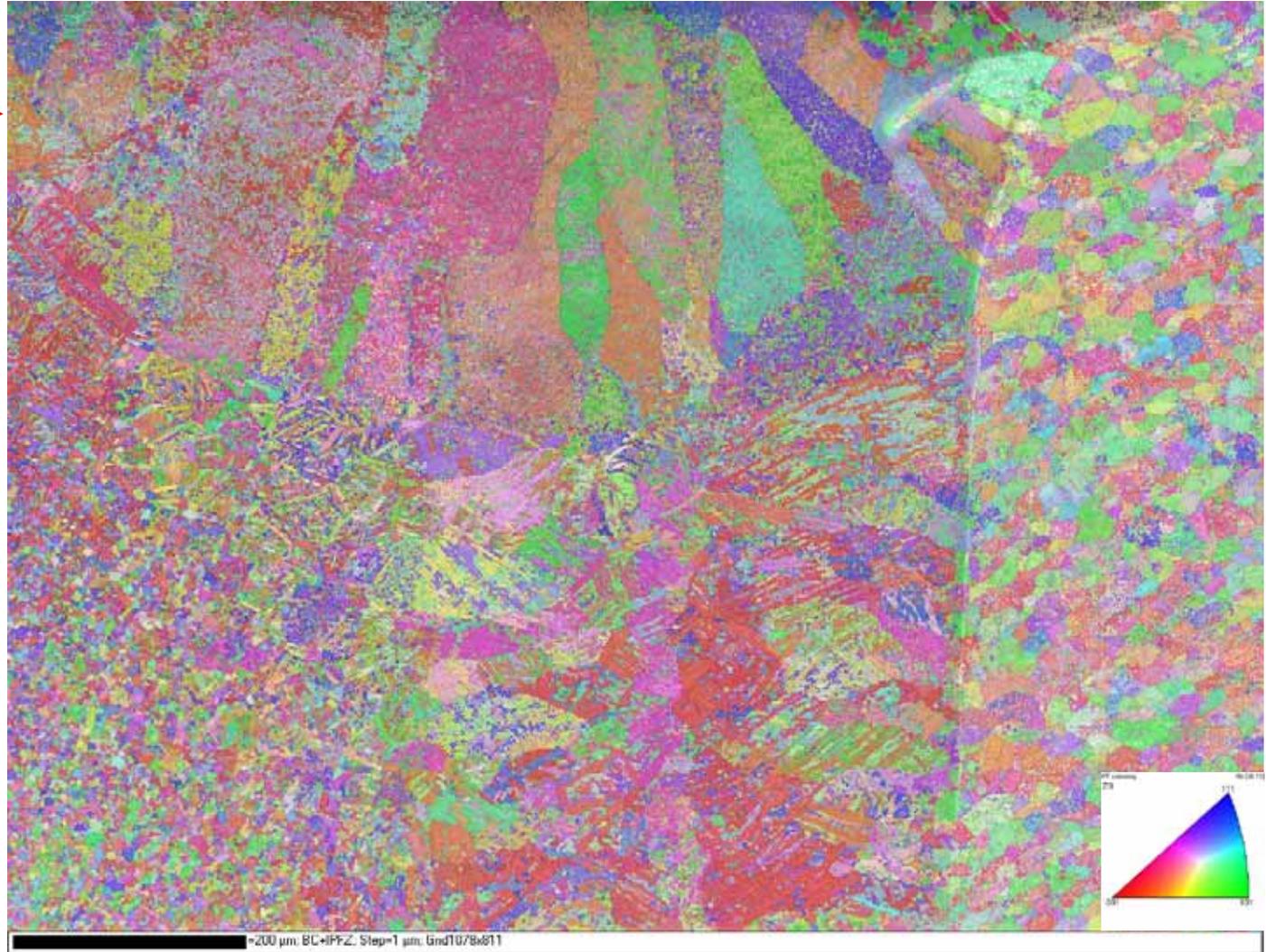
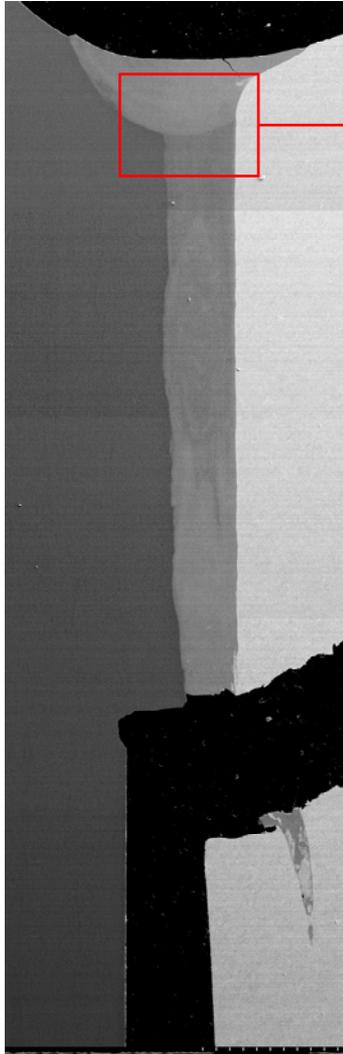


SN 10 OB Fuel Closure Weld EBSD Pattern Quality Map



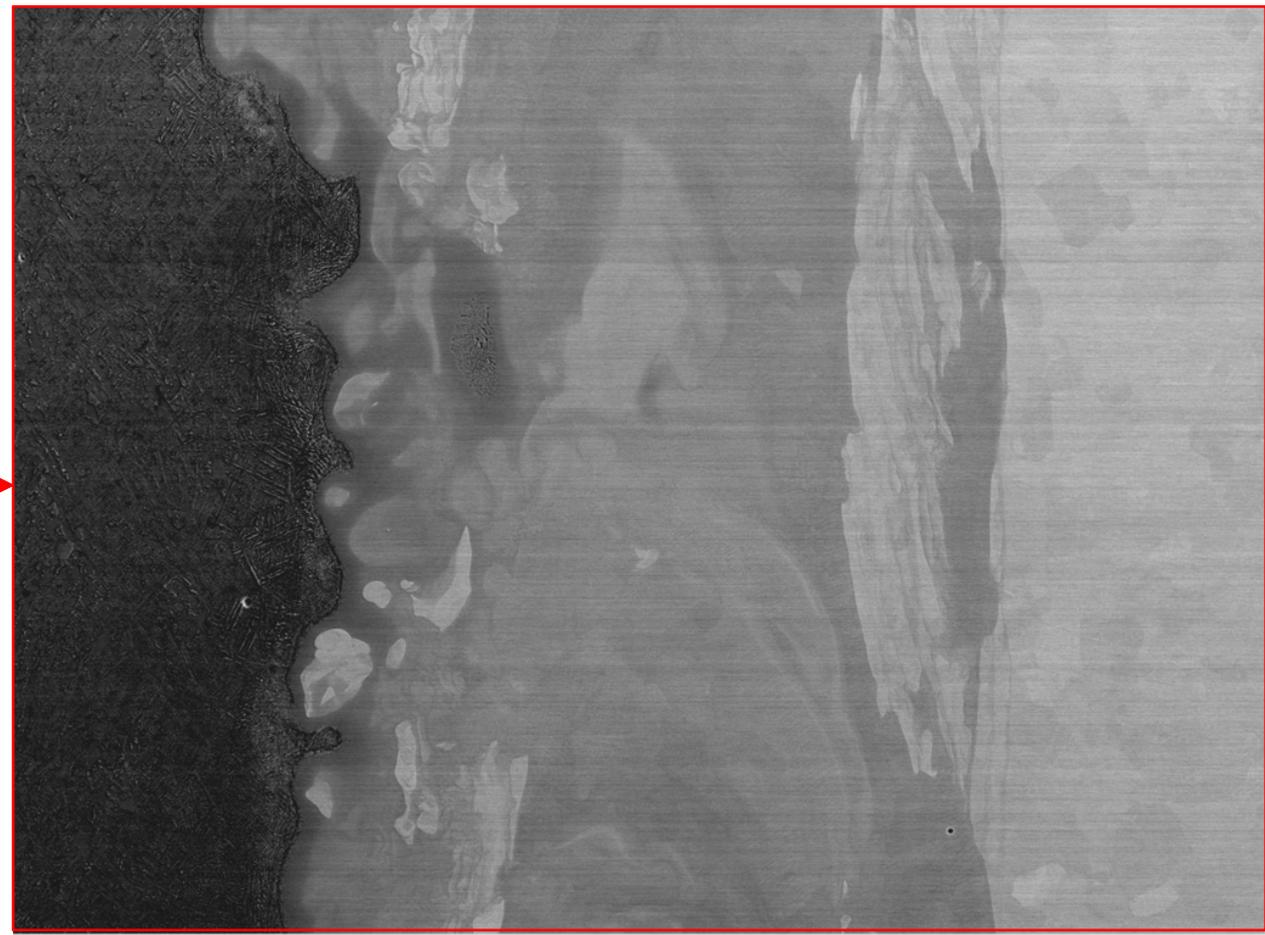
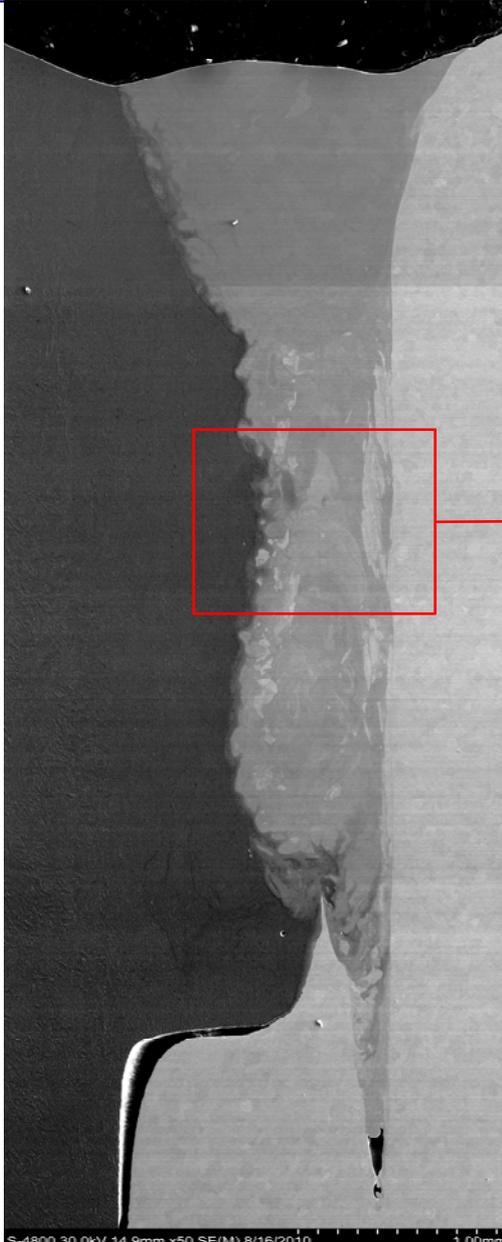


SN 10 OB Fuel Closure Weld EBSD Inverse Pole Figure Map





BSE SEM Micrograph



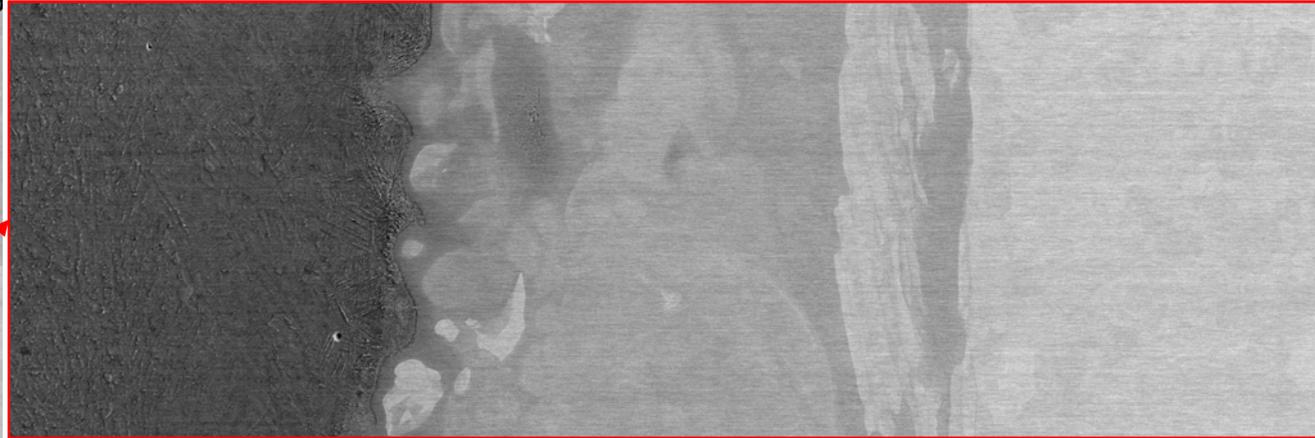
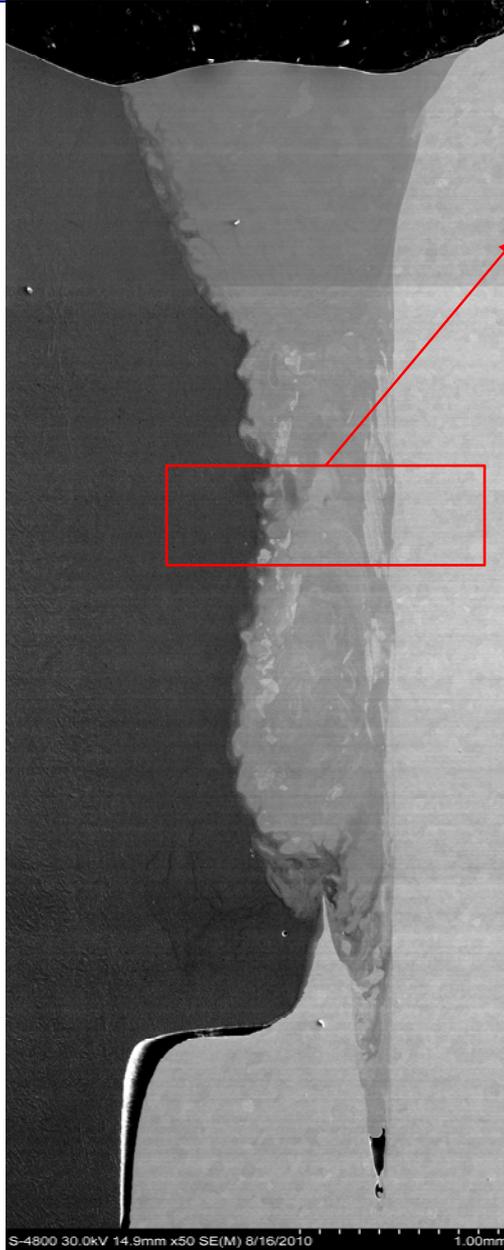
600μm

Electron Image 1

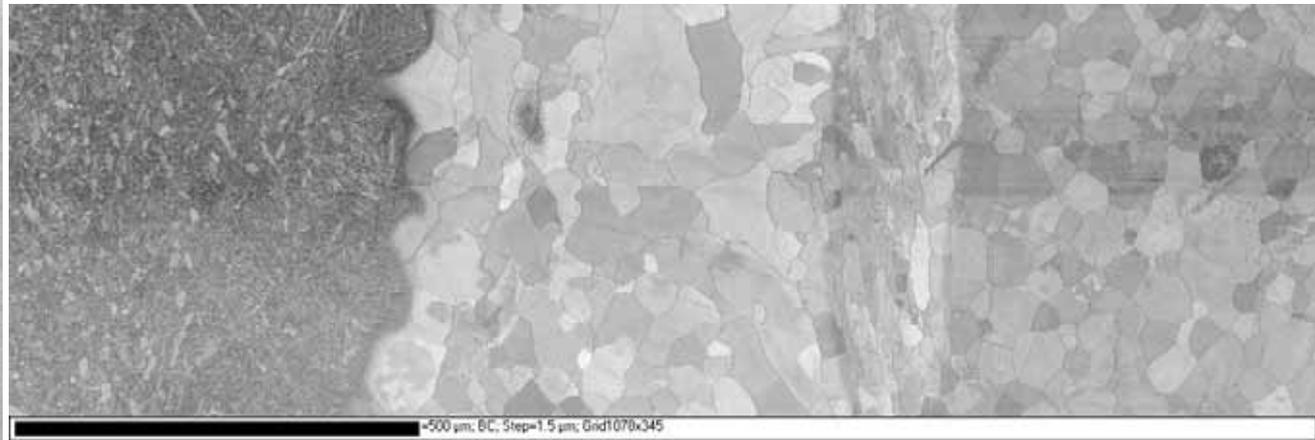


Electron BackScatter Diffraction (EBSD)

Band Contrast Map (Pattern Quality)

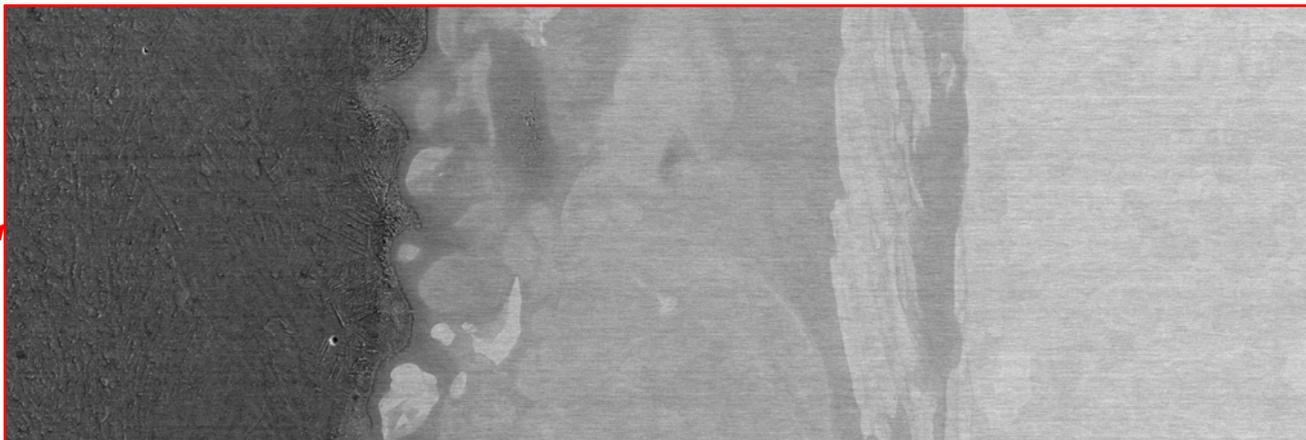
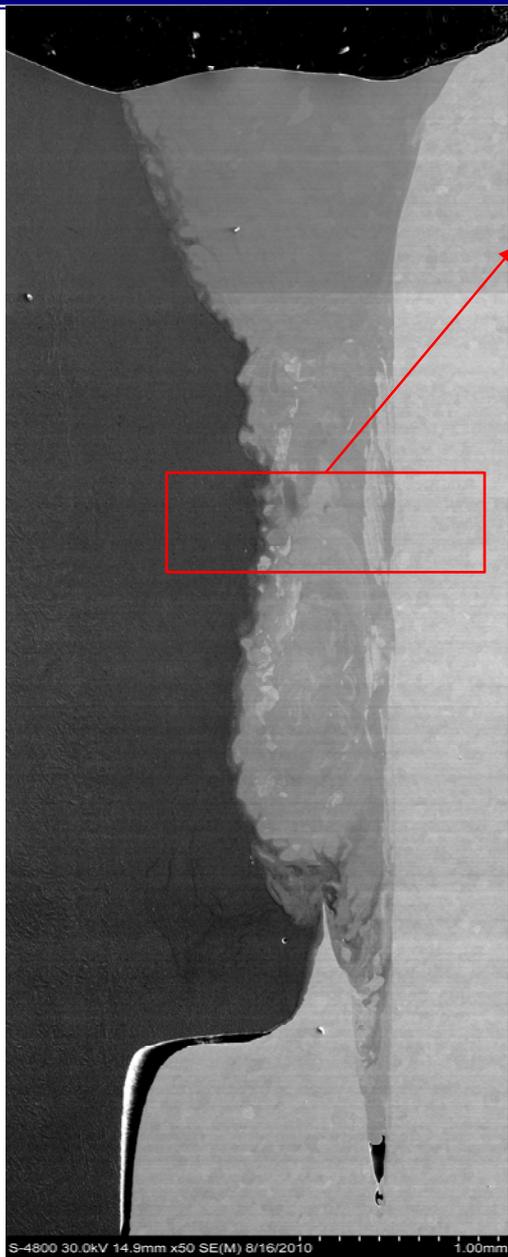


Scanning Electron Micrograph
Gray scale indicates atomic contrast



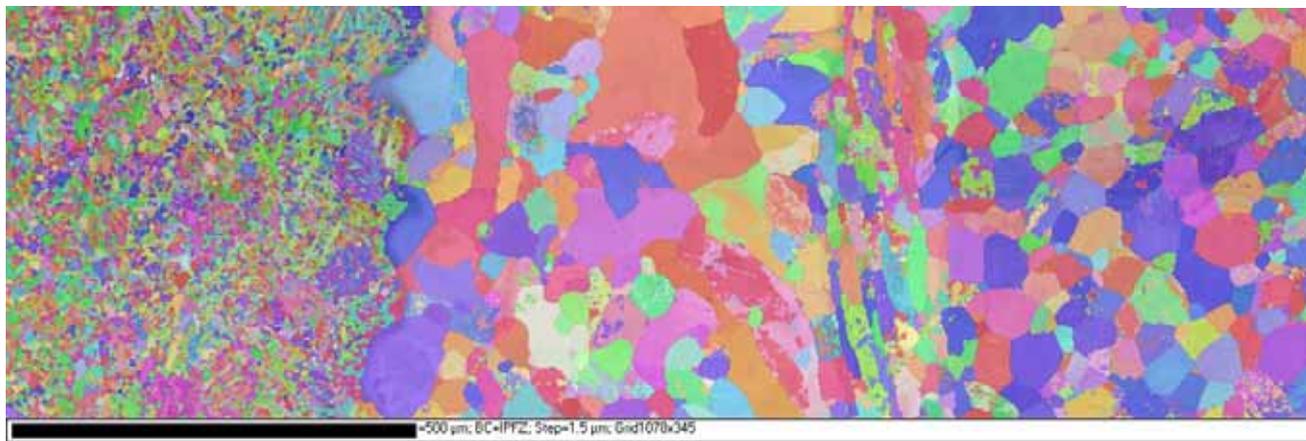
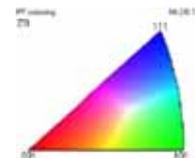
EBSD Band Contrast Map
Reveals pattern quality and grain boundaries

EBSD Inverse Pole Figure



Scanning Electron Micrograph

Gray scale indicates atomic contrast

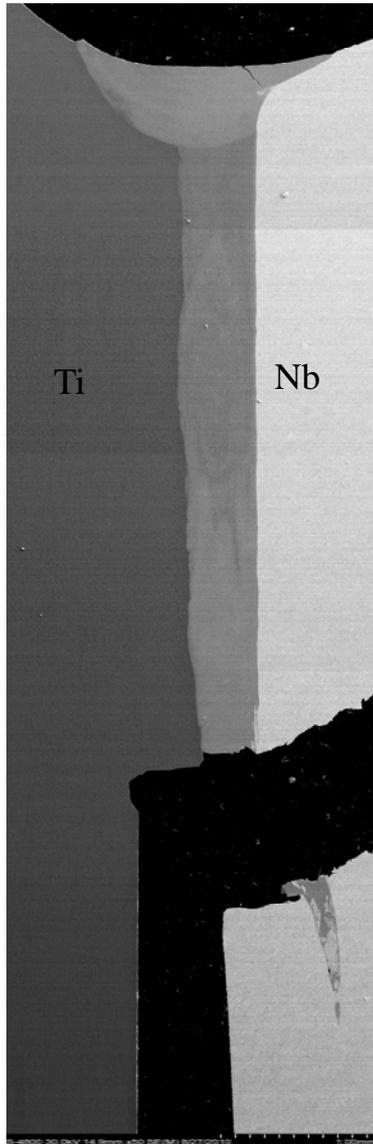


EBSD Inverse Pole Figure (IPF)

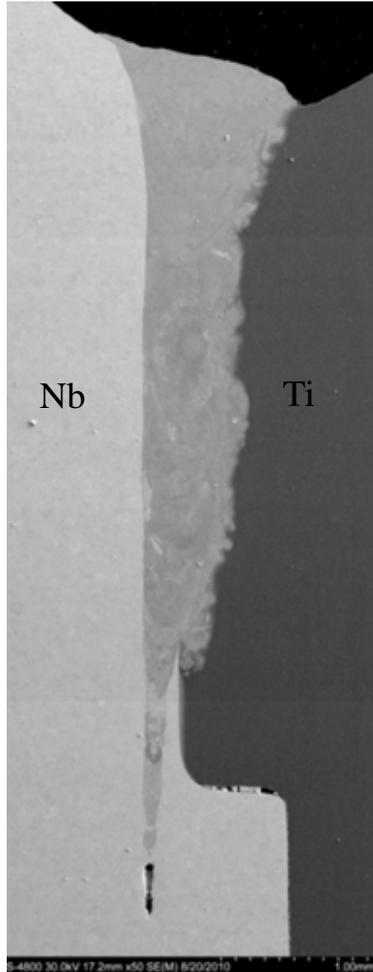
Reveals crystallographic “texture”



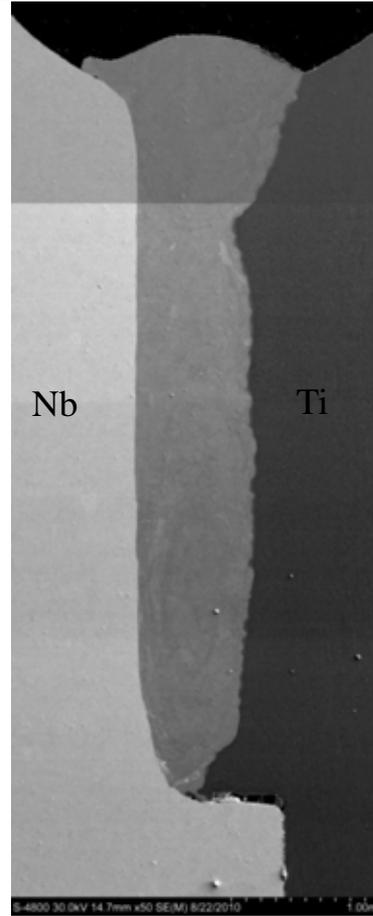
Correlative Scanning Electron Micrographs Outboard Fuel Closure Welds



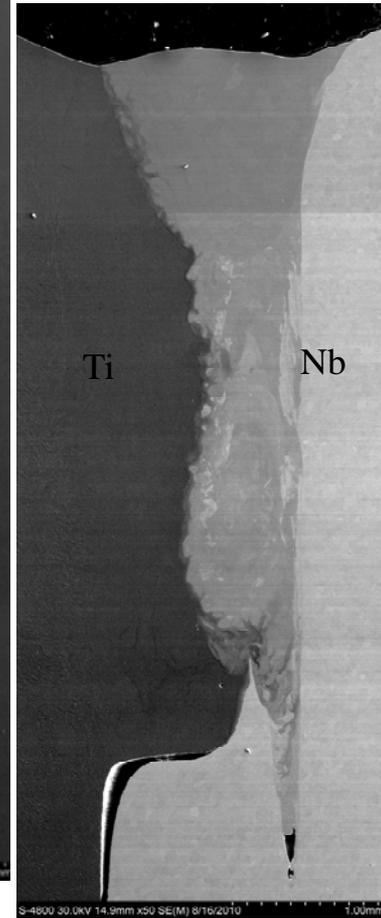
S/N 10



S/N 120



S/N 132



S/N 172



“Green Crack” Conclusions



- **No anomalous material condition associated with weld insufficiency was observed in the material surrounding the “Green Crack”**
 - **No pre-existing flaws were found in the fuel closure plate-to-injector welds of any of the four thrusters evaluated (SN 10, 120, 132, 172)**
 - **Adequate fusion took place during original EB welding process**
 - **Microstructure and microhardness (both of the parent materials and fuel closure plate) are consistent with specifications/expectations**
- **No evidence of fatigue, intergranular cracking (ICG) or other sub-critical mechanisms found**



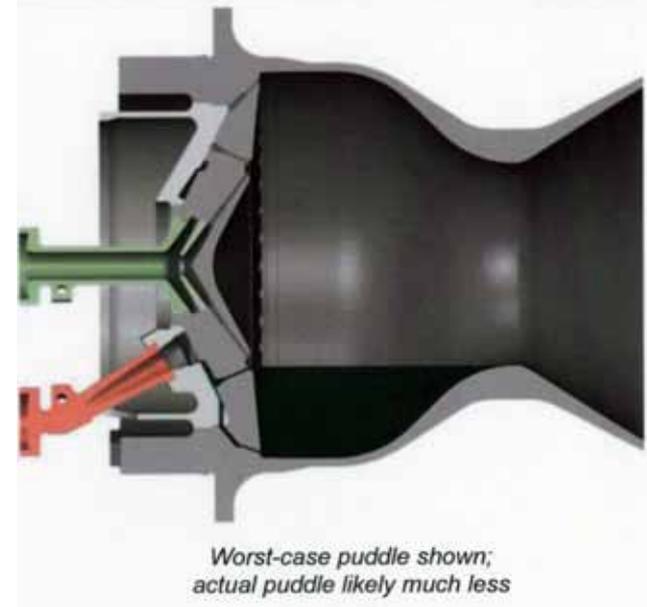
JSC/WSTF M&P Conclusions



- **Each of the four primary fractures within the injector (Red, Green, Blue, Yellow) was the result of a single event.**
- **High heat in conjunction with an oxidizing environment during the fracture ‘event’ resulted in the formation of a titanium-rich molten oxide that was deposited in a “recast” form on the Green and Blue Crack fracture surfaces.**
- **No evidence was found to indicate that a sub-critical cracking mechanism contributed to the ‘event’.**

Failure Scenario

- **Liquid Ox Collection**
 - Leaking Ox valve
 - Thruster orientation and gravity field allows puddle formation
 - Sufficient time with leak rate for puddling
- **Liquid OX Flows thru injector and boundary layer cooling holes into fuel injector manifold**
 - Sufficient puddle depth to go through holes
- **Sufficient OX in fuel manifold to produce detonation capable of causing injector damage**
- **Single firing to produce explosion**





Flight Rationale



- 1. Flight valves less susceptible to leakage than S/N 10.**
- 2. Flight conditions prevent liquid oxidizer intrusion into fuel manifold.**
- 3. Leak detectors and existing Flight Rules make firing a leaking thruster unlikely.**



References



- **Struers E-METALOG Method #1416 available at:**
http://www.struers.com/modules/emetalog/generic_view.asp?method=1416 (Feb 2011)
- **ASTM E407 Standard Practice for Microetching Metals and Alloys, Etchants Table 2, Etchants 129, 163, 161.**
- **The authors would like to gratefully acknowledge our colleagues D. Cone et al, NASA WSTF, Las Cruces NM.**
- **AE3654: L1L (P010) Investigation Status, E. Fitzgerald OMSS/RCS PRT, 8/11/2010**
- **RCS Development Thruster S/N 100 Failure Investigation Status, WSTF ARCS Test Article – L1L, E. Fitzgerald OPOTT 8/25/2010**
- **L1L S/N10 M&P Conclusions, Orbiter M&P PRT, D. Cone et al, NASA WSTF, Las Cruces NM. 09/15/2010**
- **RCS Development Thruster S/N 10 Failure Investigation Summary, ORB-BU-3, 10/25/2010**
- **Orbiter RCS Thruster WSTF Test Stand Failure Investigation, NESC Out-Briefing, Bud Castner & Jeremy Jacobs, 11/02/2010**



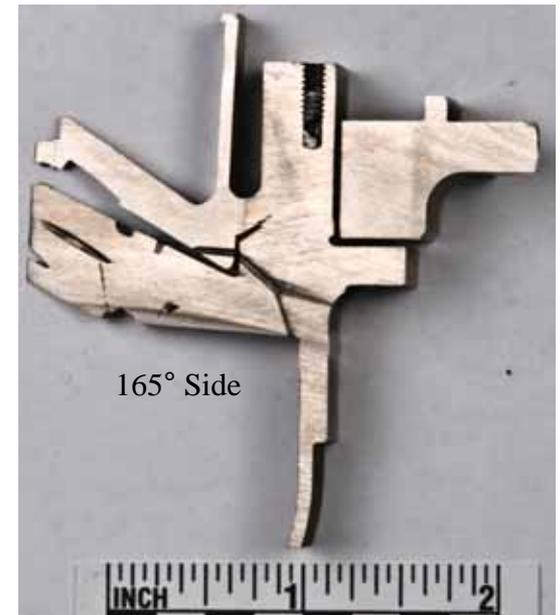
Back-Up Charts



SN 10 2nd Cut Photography

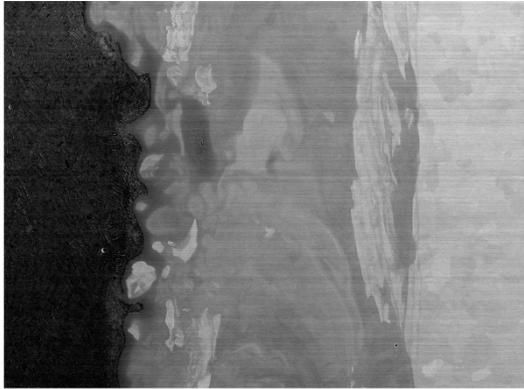


Diamond
Saw Cut

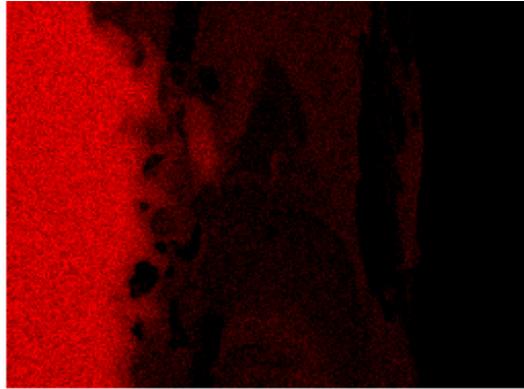




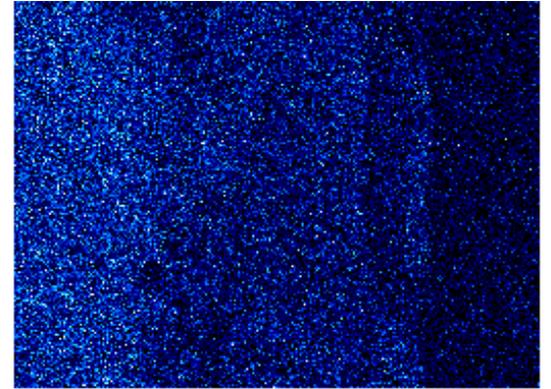
EDS Dot Map



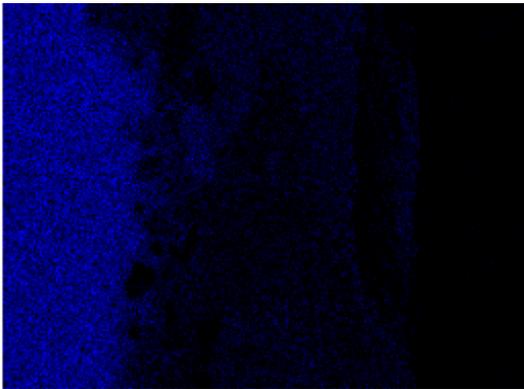
Electron Image 1



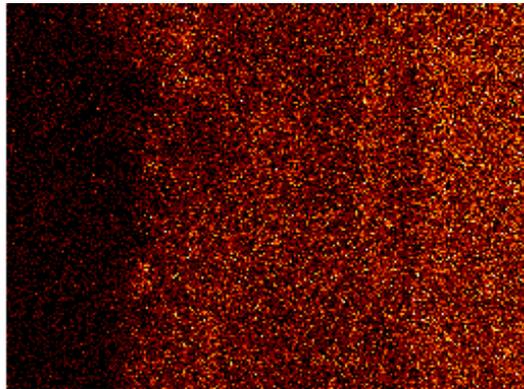
Ti Ka1



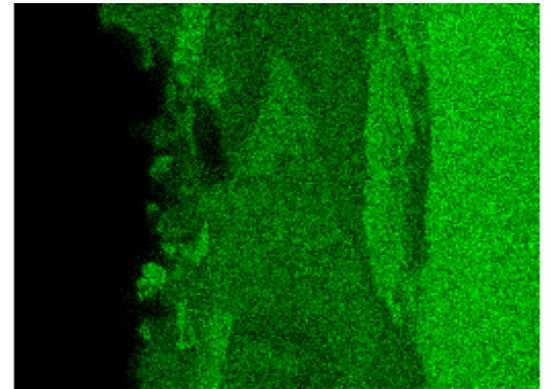
Al Ka1



V Ka1

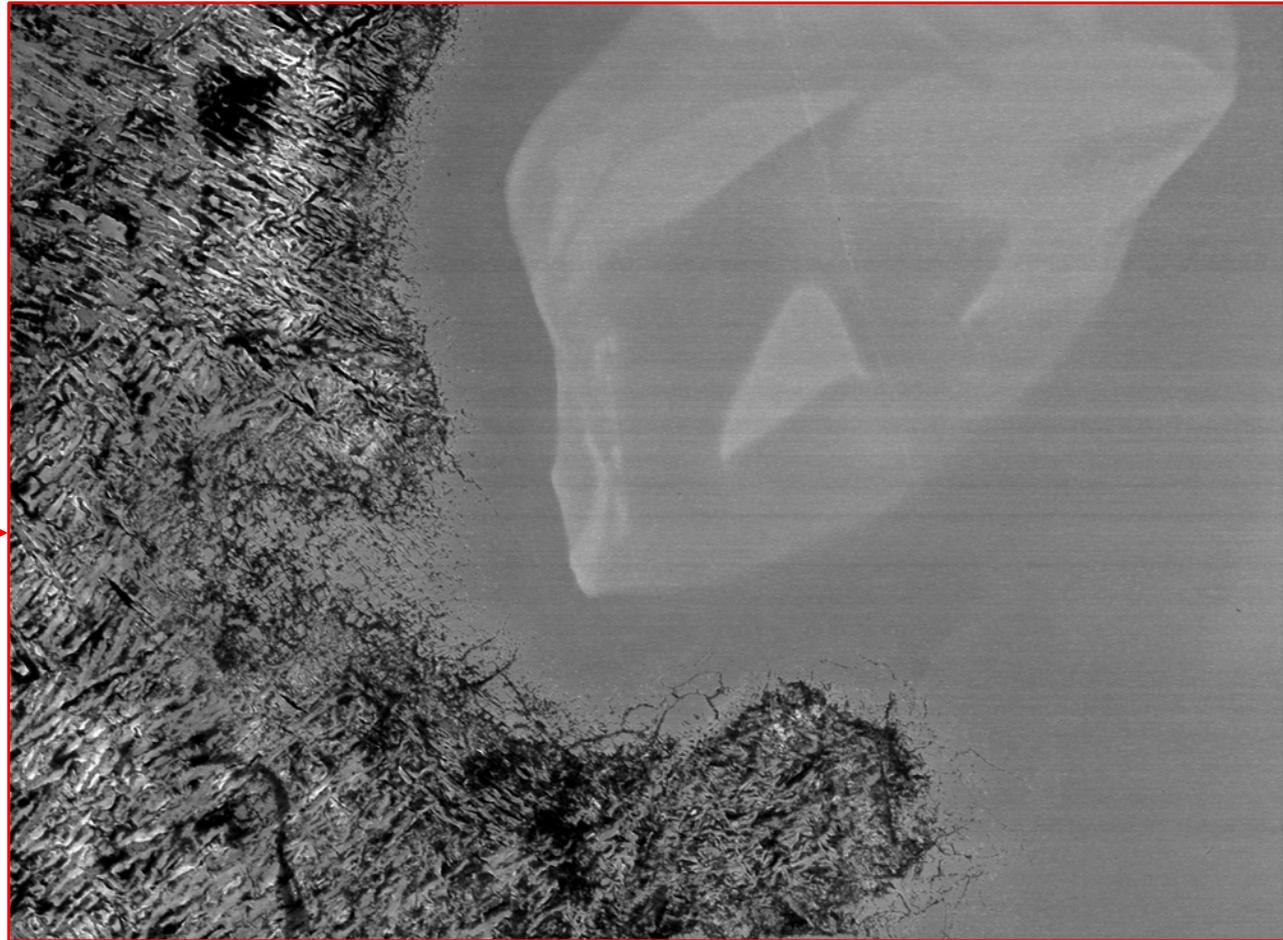
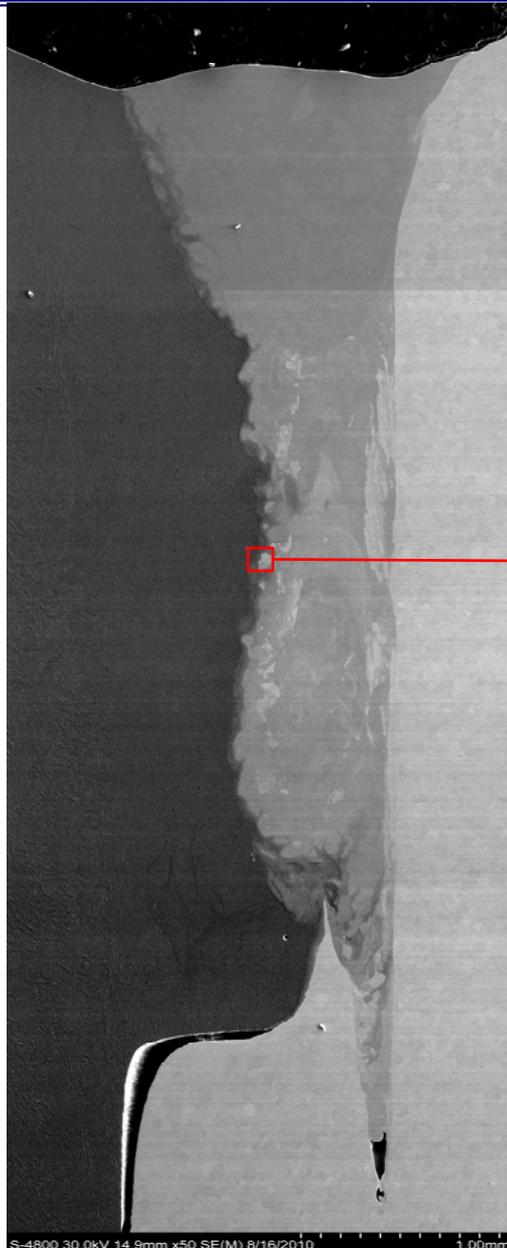


Hf La1



Nb La1

SEM Micrographs

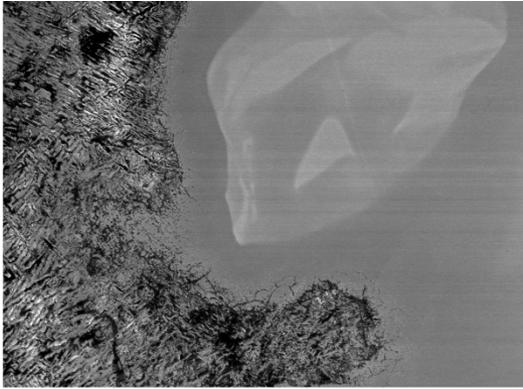


60μm

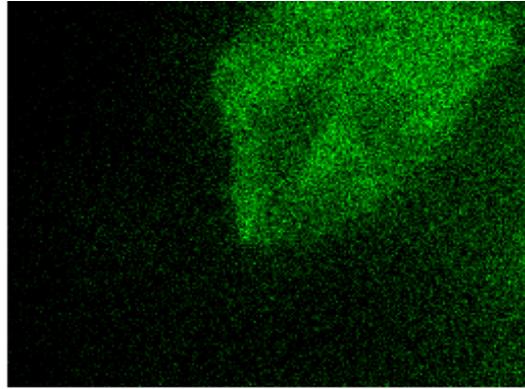
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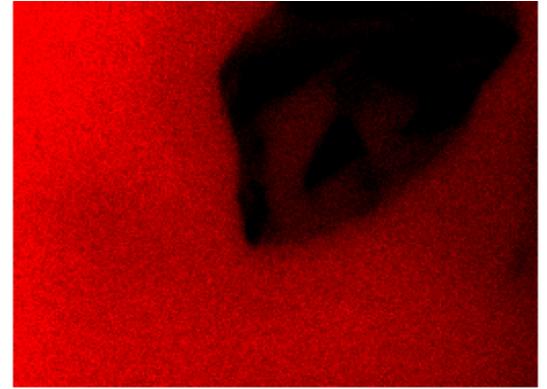
EDS Dot Map



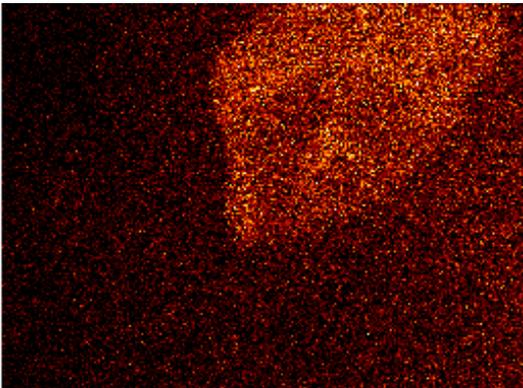
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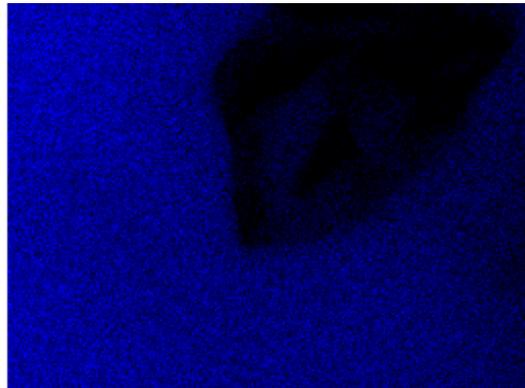
Nb Ka1



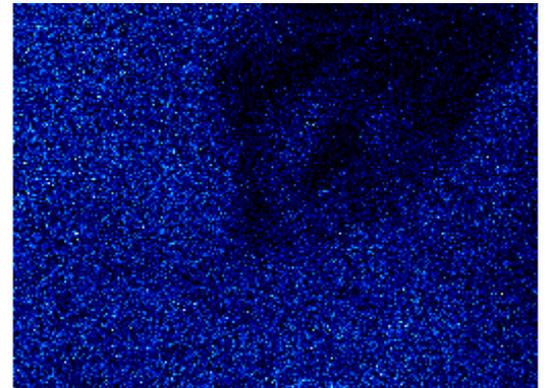
Ti Ka1



Hf La1



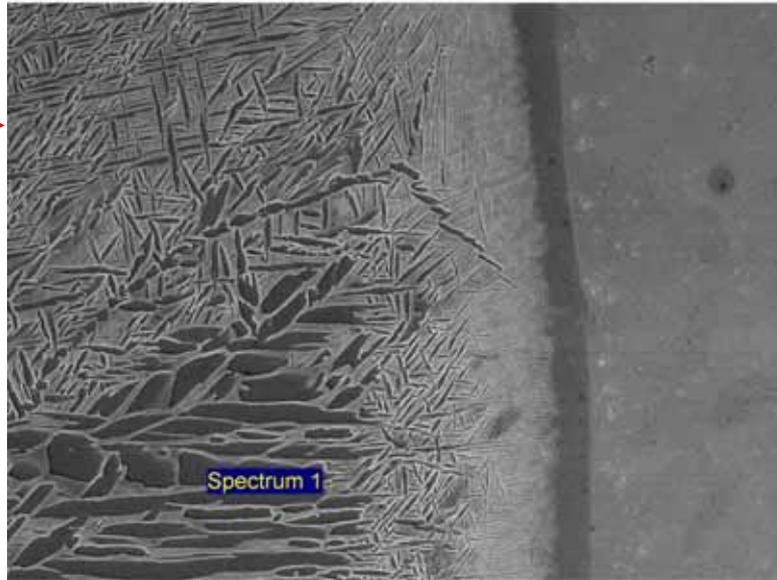
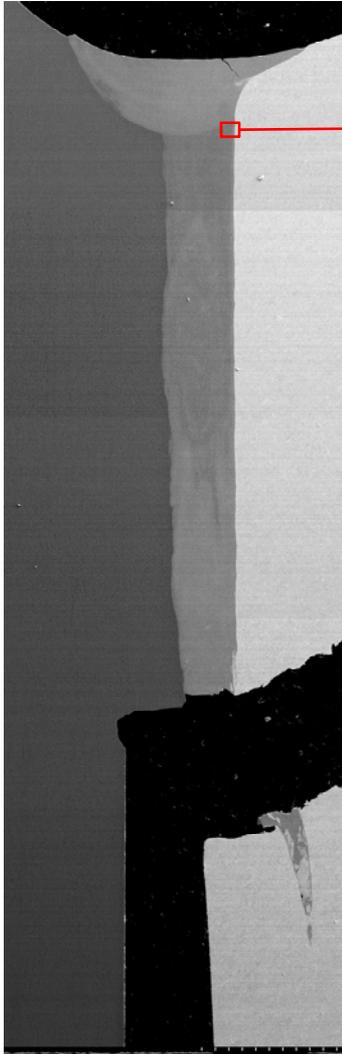
V Ka1



Al Ka1

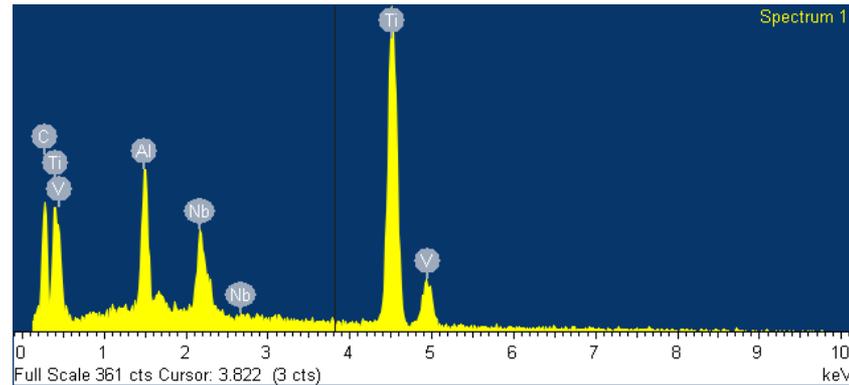


SN 10 OB Fuel Closure Weld EDS of Niobium/Weld Interface (165°)



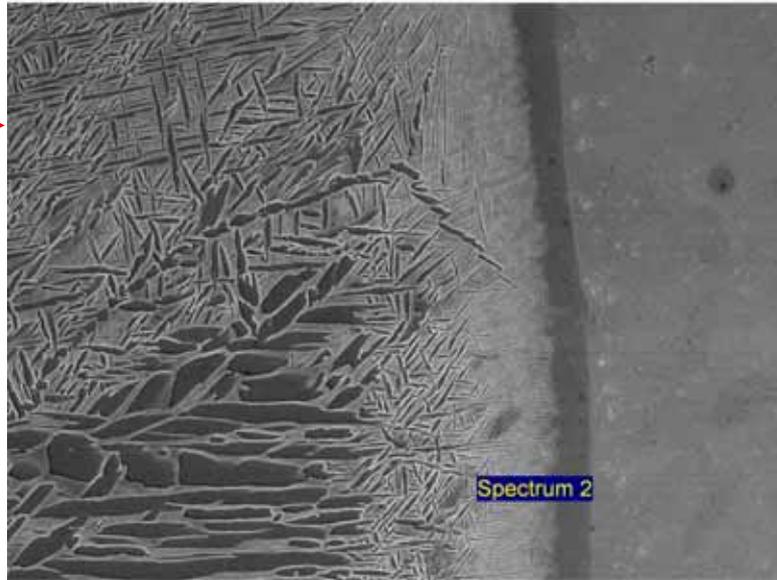
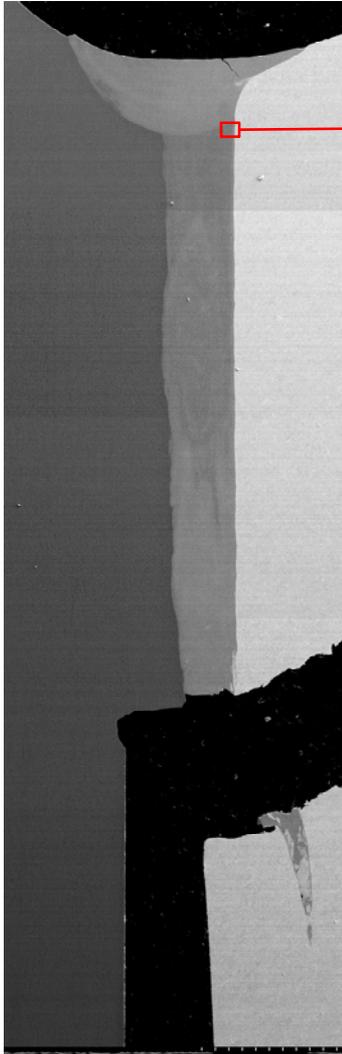
30µm

Electron Image 1



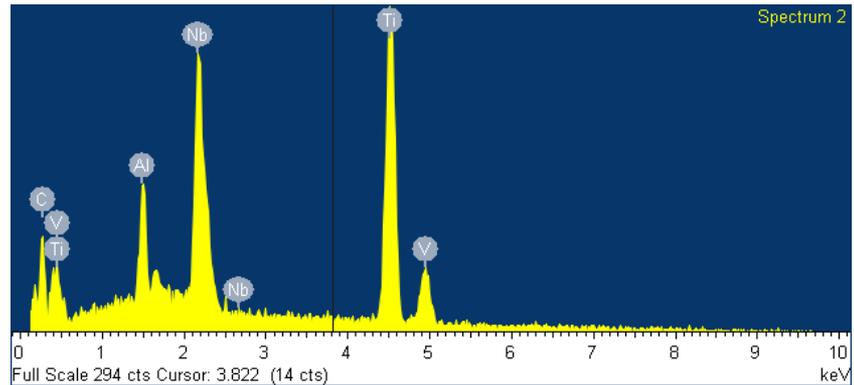


SN 10 OB Fuel Closure Weld EDS of Niobium/Weld Interface (165°)



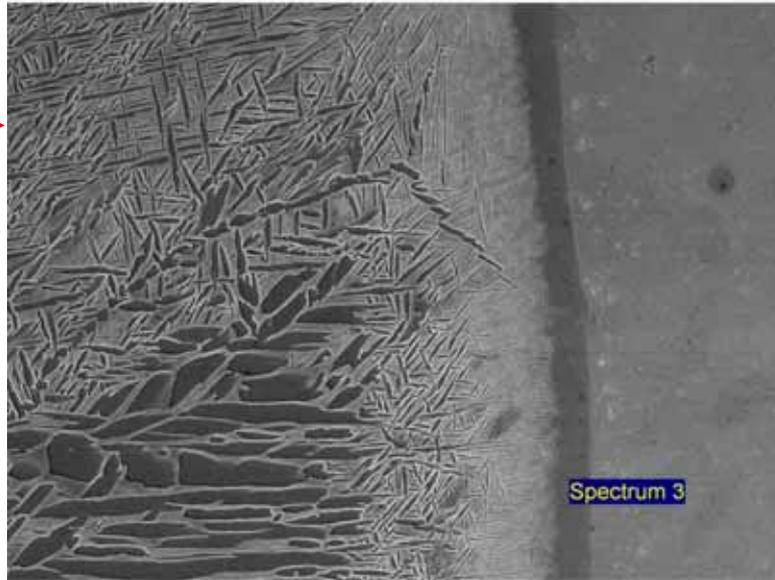
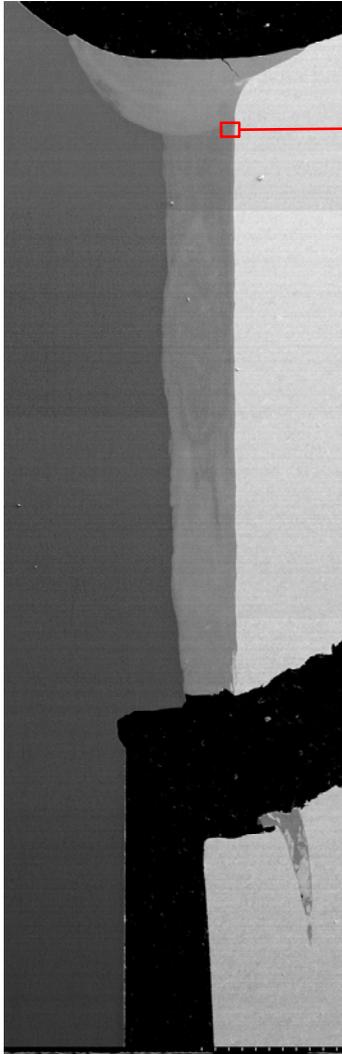
30µm

Electron Image 1



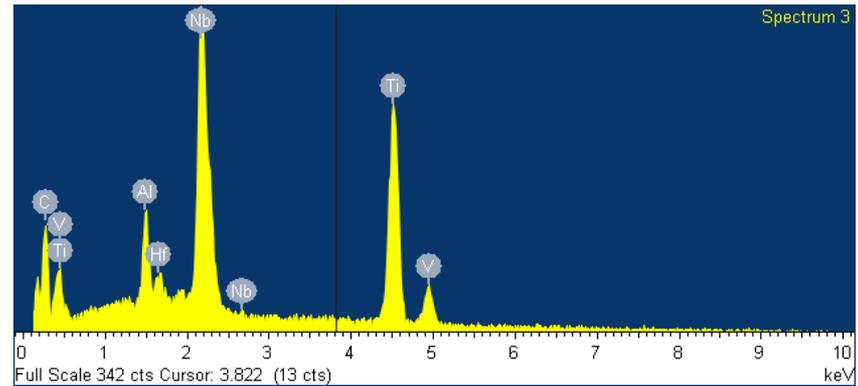


SN 10 OB Fuel Closure Weld EDS of Niobium/Weld Interface (165°)



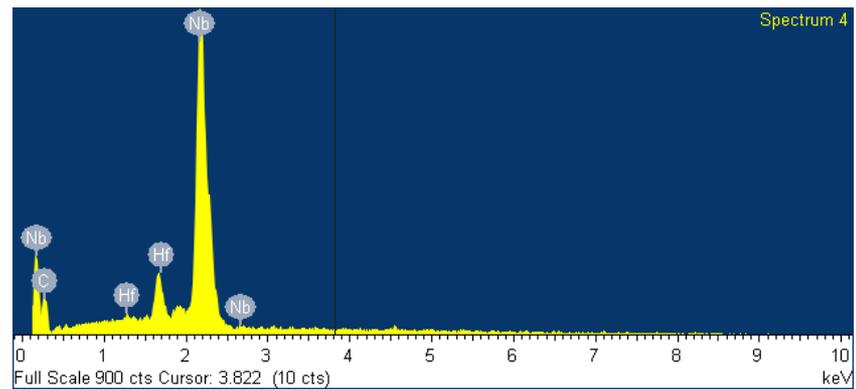
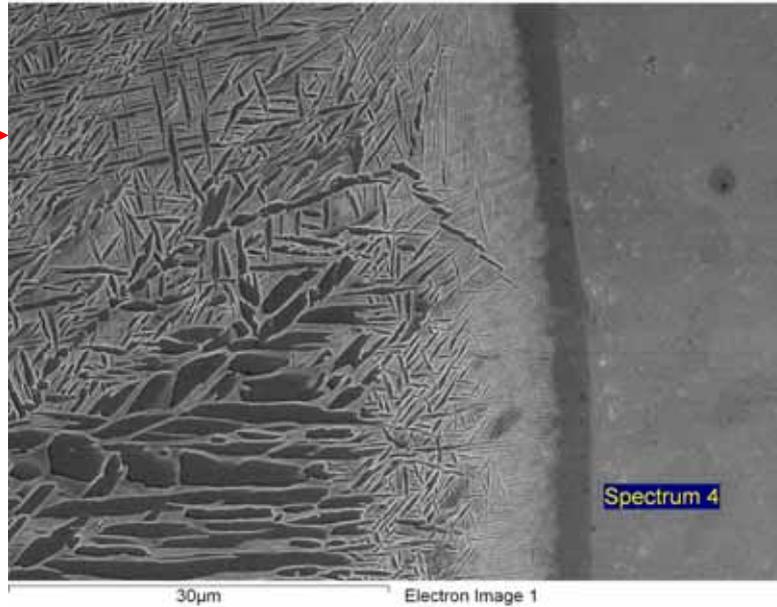
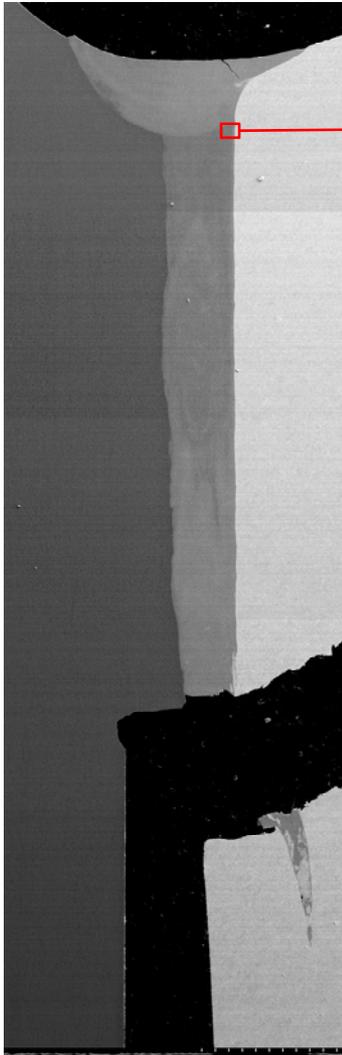
30µm

Electron Image 1





SN 10 OB Fuel Closure Weld EDS of Niobium/Weld Interface (165°)

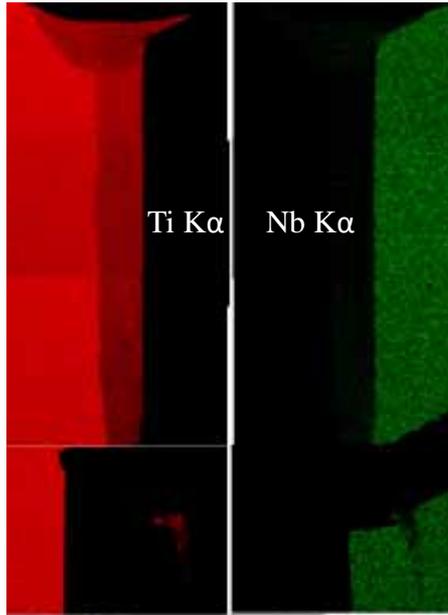




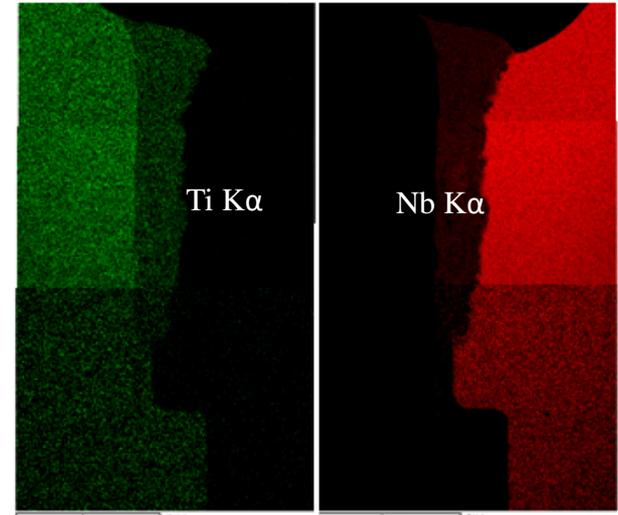
Correlative Energy Dispersive Spectroscopy Outboard Fuel Closure Welds



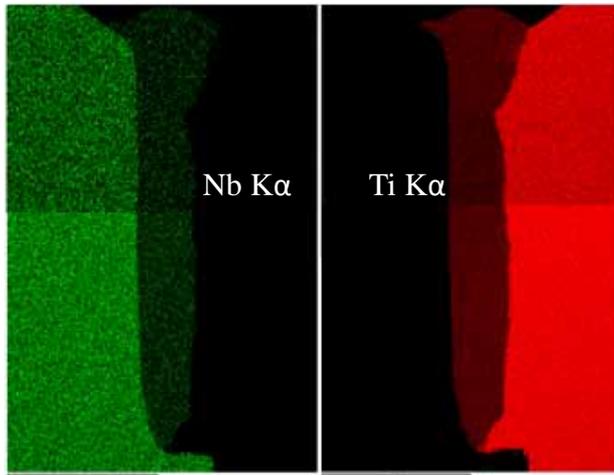
S/N 10



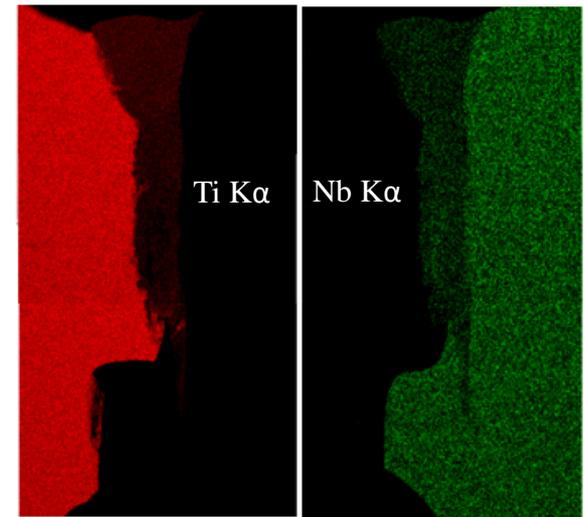
S/N 120



S/N 132



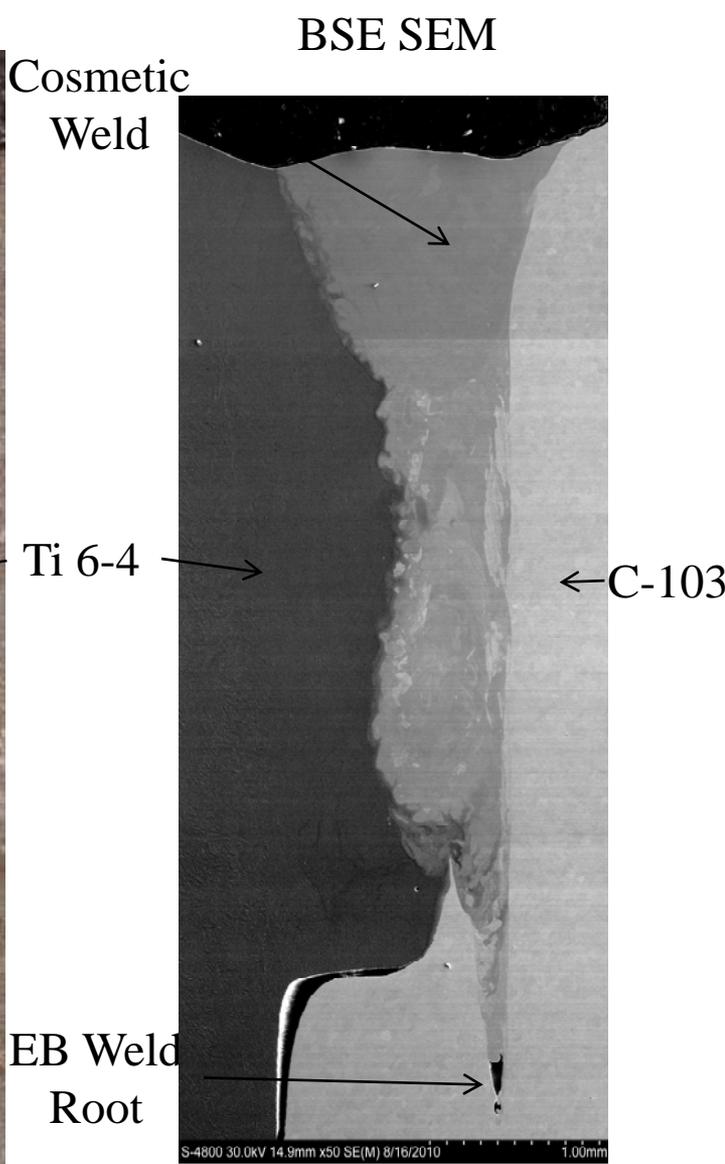
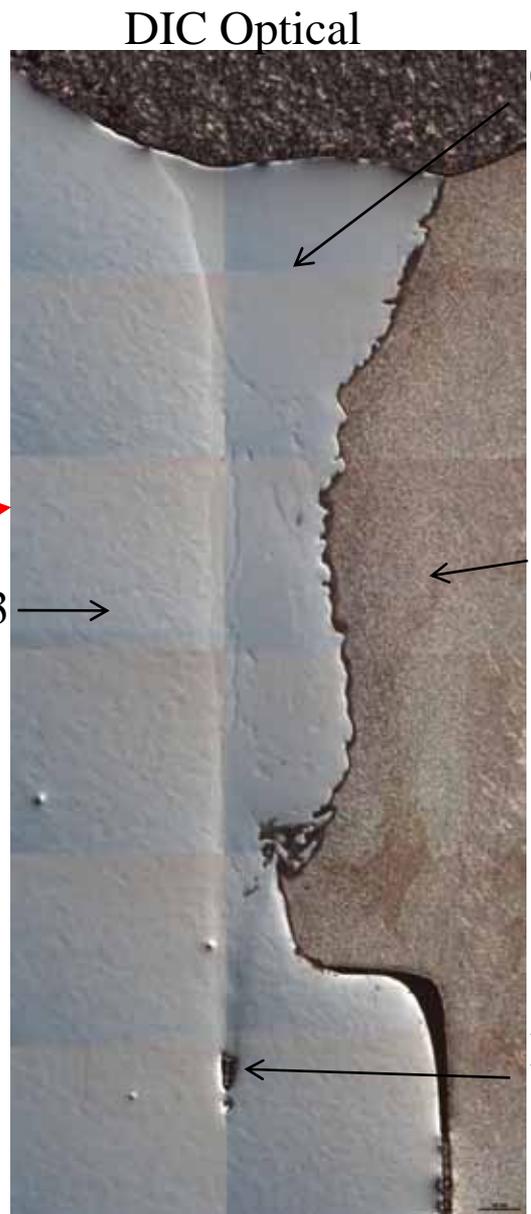
S/N 172



Montages not to scale

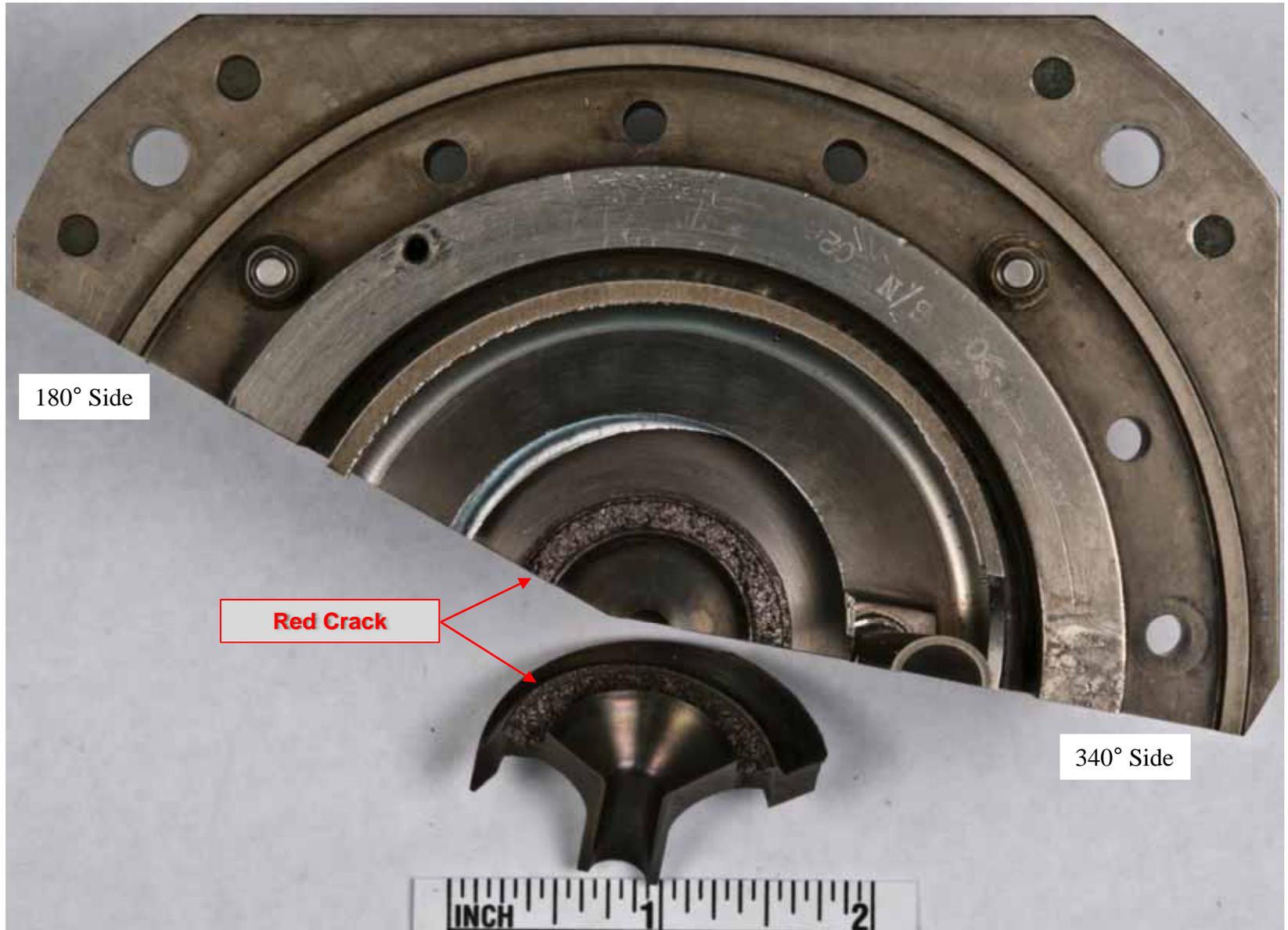


SN 172 DIC Optical & BSE SEM Montages





S/N: 10 Red Crack Exposed by End Milling (180° to 340° Side)



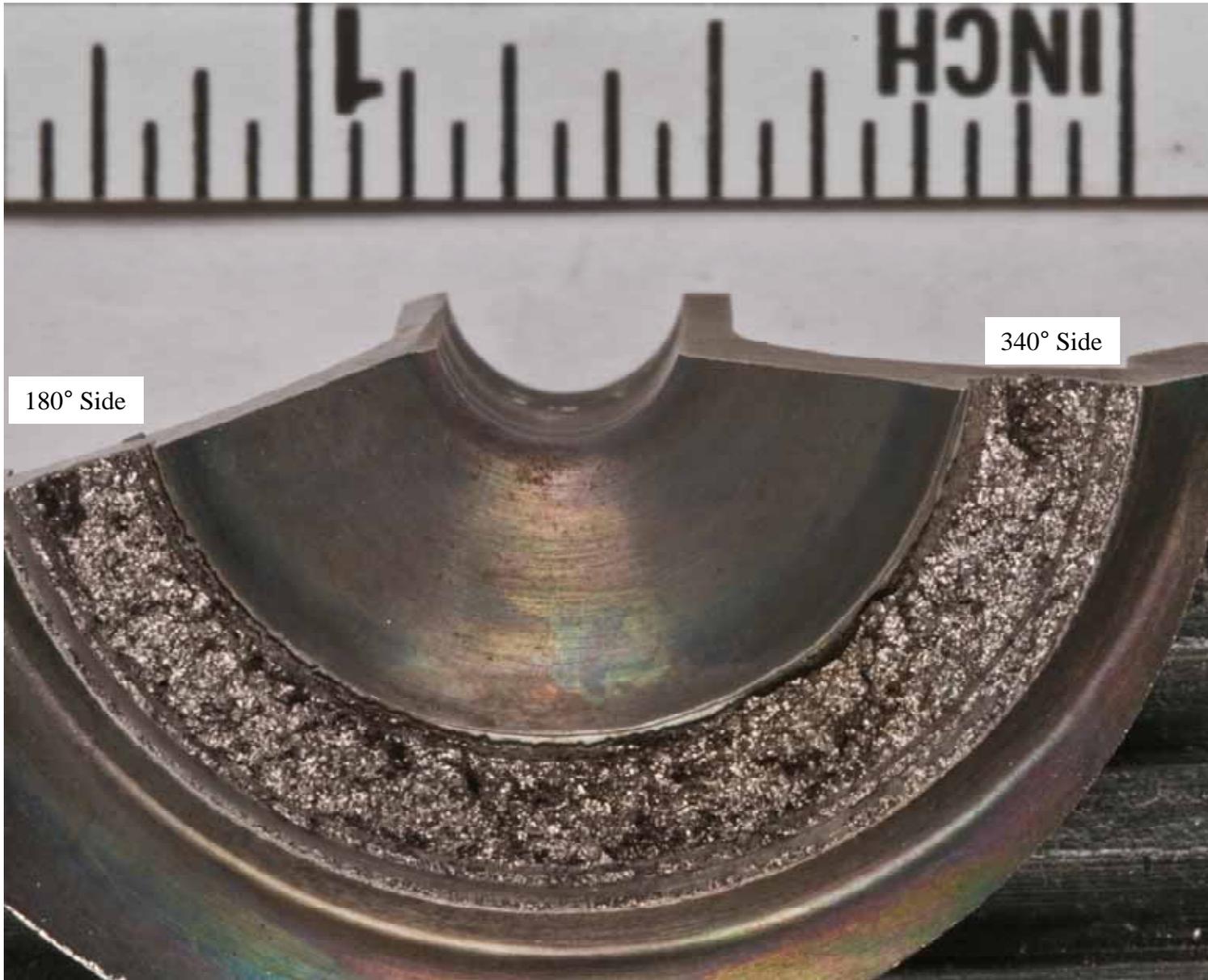


Isolated 'Red Crack' Region, Fuel Closure-Side (180-340° Side)





Isolated 'Red Crack' Region, Fuel Closure-Side (180-340° Side)





Isolated 'Red Crack' Region, Fuel Closure-Side (180-340° Side)



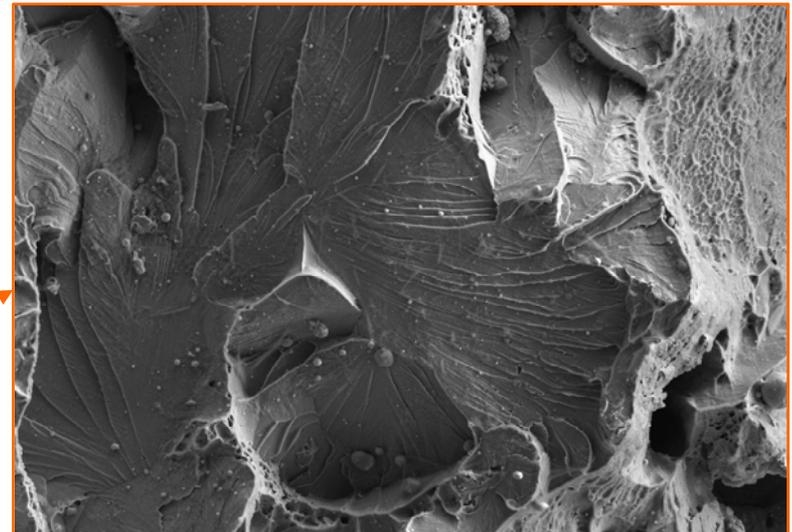
Scanning Electron Microscopy

Fuel Closure-Side

180-340°

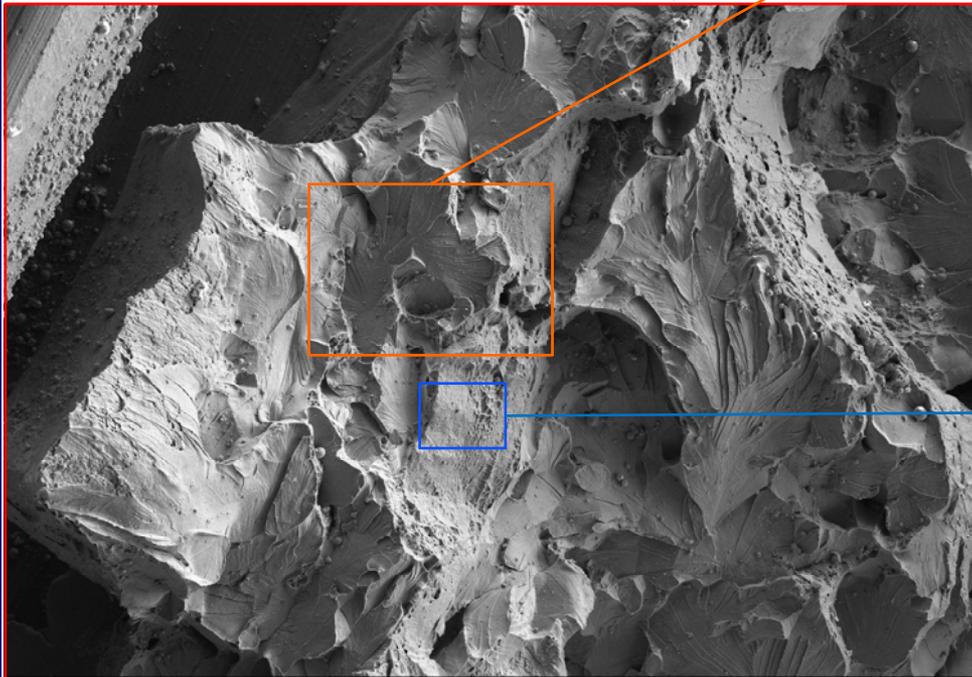
180° Side

340° Side



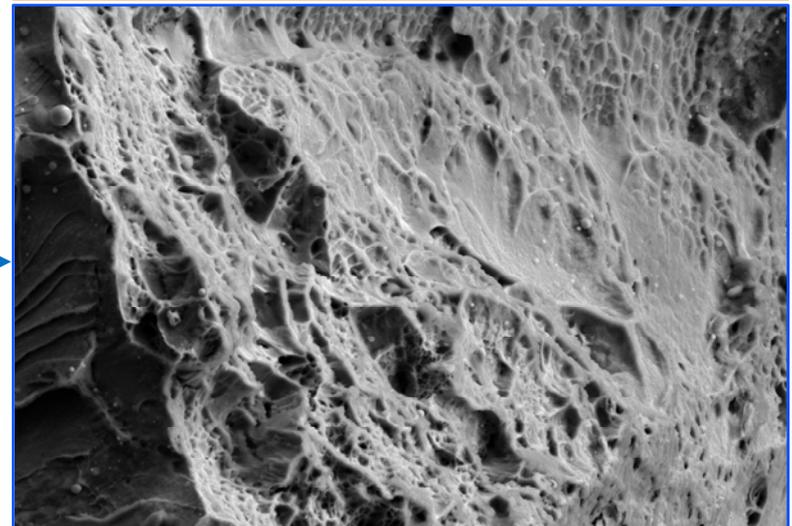
S-4800 20.0kV 5.6mm x500 SE(M) 9/7/2010

100um



S-4800 20.0kV 5.6mm x100 SE(M) 9/7/2010

500um



S-4800 20.0kV 5.6mm x1.00k SE(M) 9/7/2010

50.0um



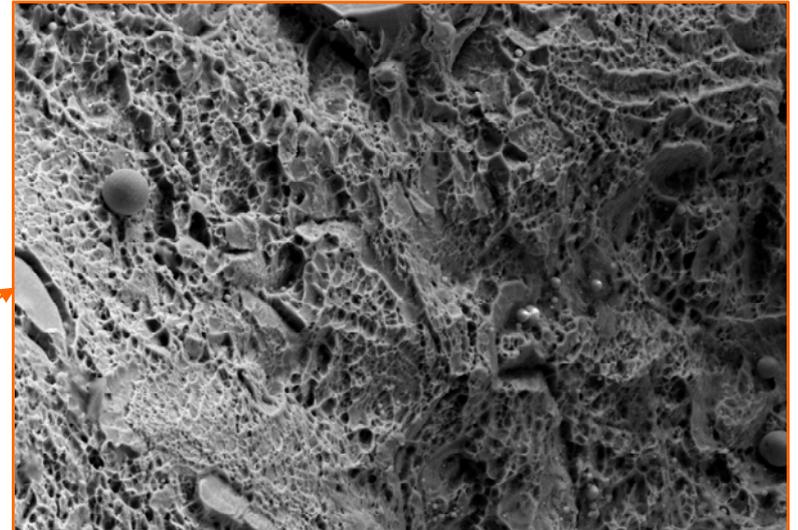
Scanning Electron Microscopy

Red Crack, Fuel Closure Side (180-340°)



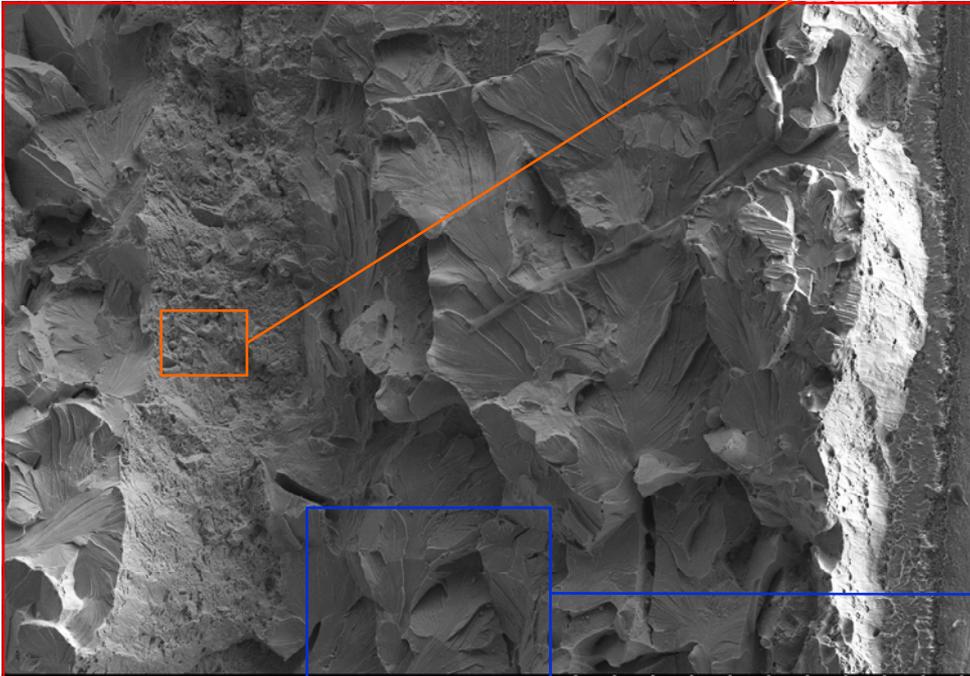
180° Side

340° Side



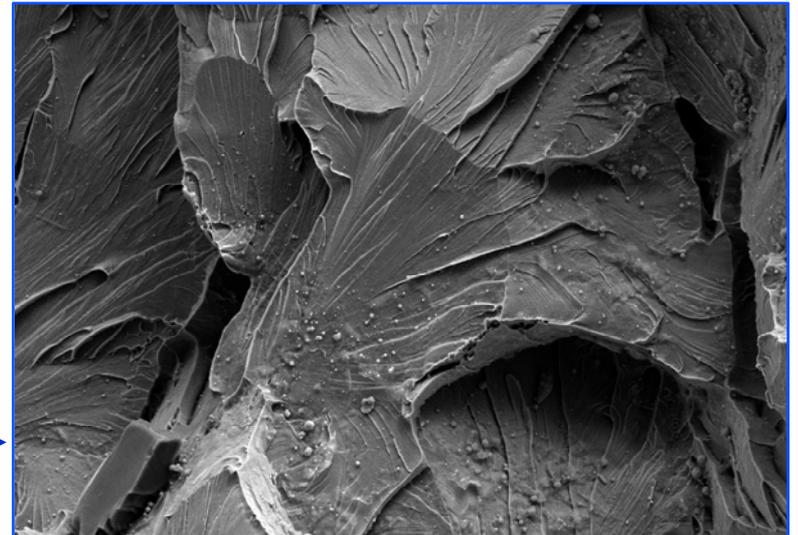
S-4800 20.0kV 5.6mm x1.00k SE(M) 9/7/2010

50.0um



S-4800 20.0kV 5.6mm x100 SE(M) 9/7/2010

500um



S-4800 20.0kV 5.6mm x500 SE(M) 9/7/2010

100um



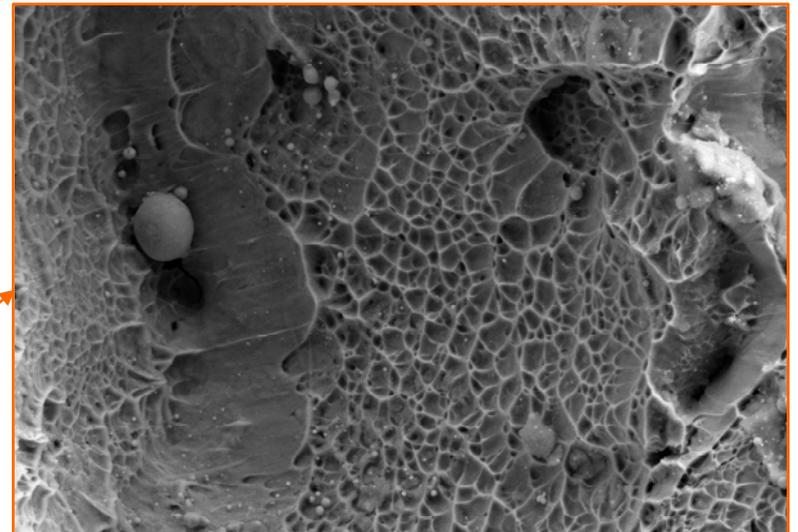
Scanning Electron Microscopy

Red Crack, Fuel Closure Side (180-340°)



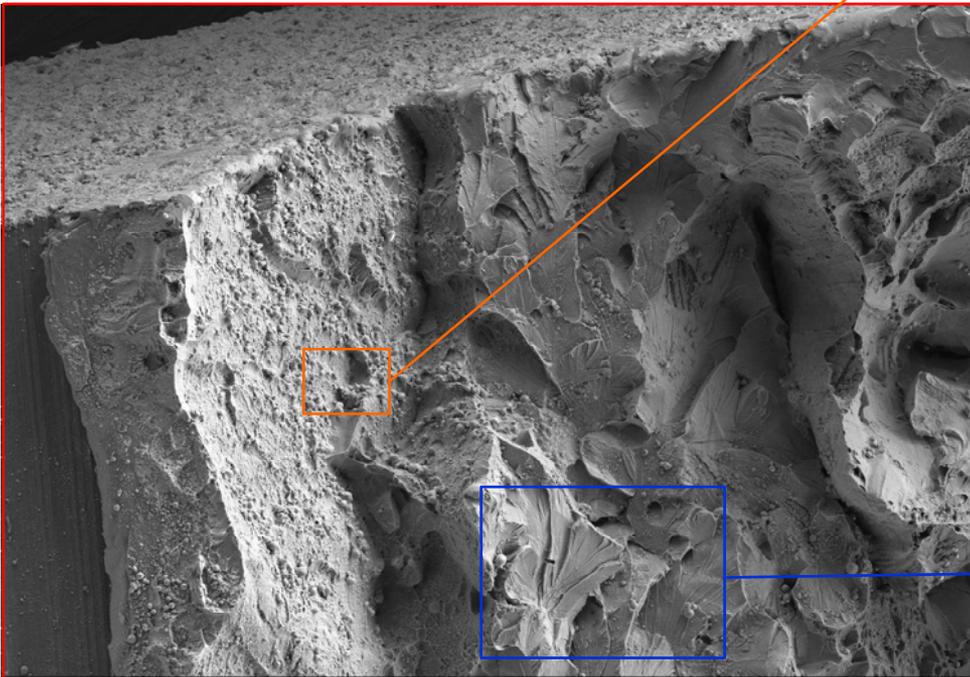
180° Side

340° Side



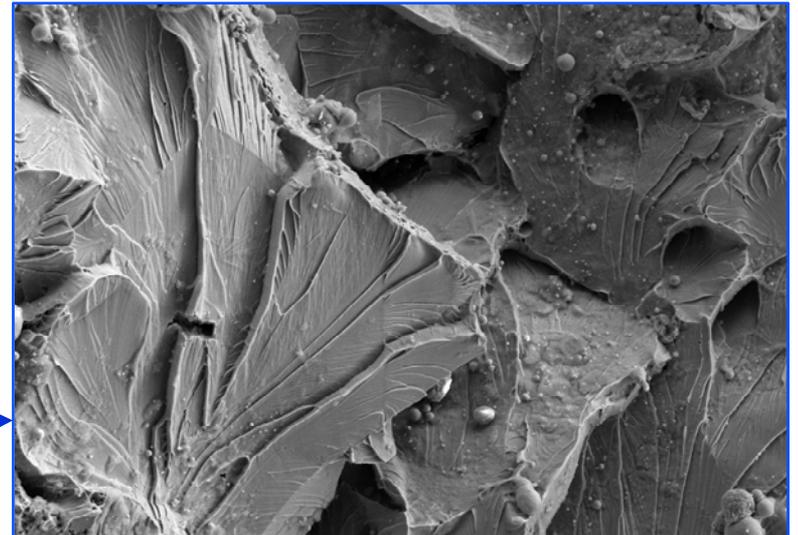
S-4800 20.0kV 5.6mm x1.00k SE(M) 9/7/2010

50.0um



S-4800 20.0kV 5.6mm x100 SE(M) 9/7/2010

500um



S-4800 20.0kV 5.6mm x500 SE(M) 9/7/2010

100um



“Red Crack” Summary



- **No thumbnails evident**
 - No conventional fatigue observed
 - No intergranular cracking observed
 - No unexplained discoloration
- **Continuous shear band evident along inboard and outboard welds**
 - Shear dimples evident along bands
 - No significant interruptions in shear bands
- **Fracture surface across niobium**
 - Mostly cleavage-like facets
 - Occasional shear dimples between facets