Laboratory Reproduction and Failure Analysis of Cracked Orbiter Reaction Control System Niobium Thruster Injectors

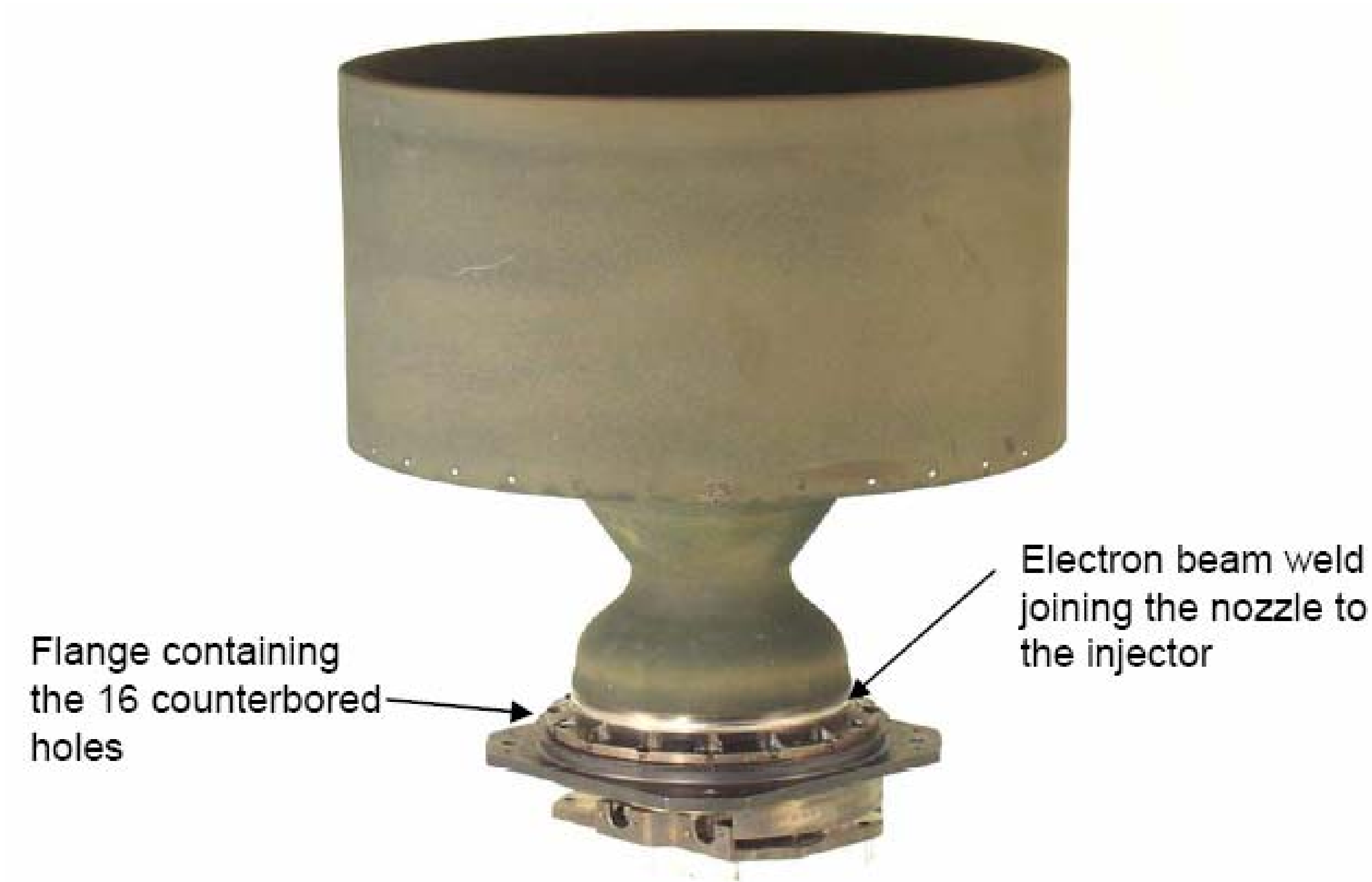
Jeremy B. Jacobs
&
Willard L. Castner

Presentation Outline

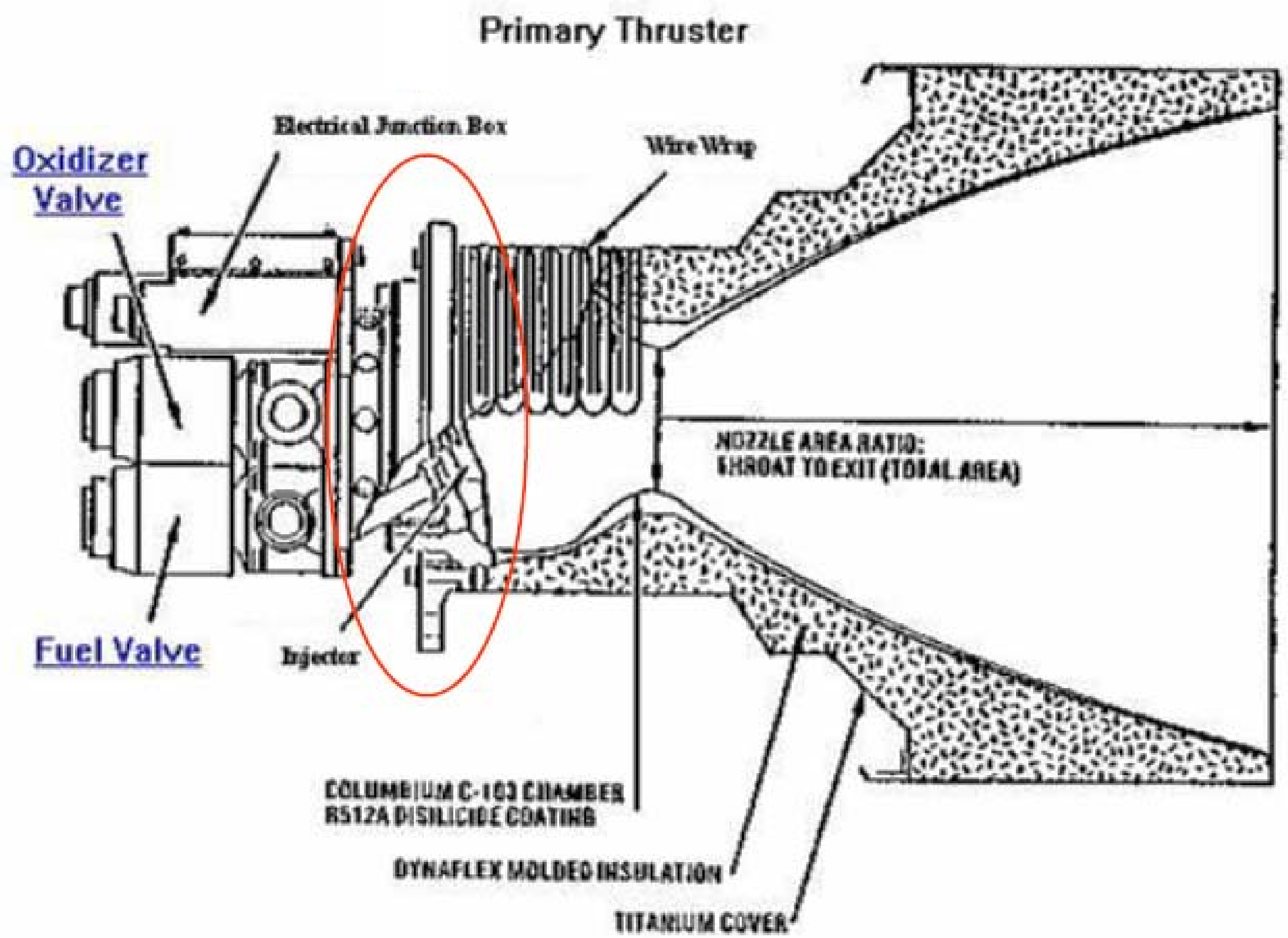
- Description of RCS Thruster
  - Thruster/Injector Photos & Cross Sections
  - Injector Crack Descriptions
- History of Injector Cracking
- Reproduction of Injector Cracking
  - Brownfield Specimen
  - Hydrofluoric Acid Tests
    - Specimen Loading Arrangement
    - Specimen #3 Results
    - Specimen #5 Results
    - Test Matrix
  - Krytox/Brayco Tests
    - Specimen Loading Arrangement
    - Specimen #13 Results
    - Test Matrix
- Conclusions/Recommendations

Endeavour STS-113 Landing

RCS Thruster

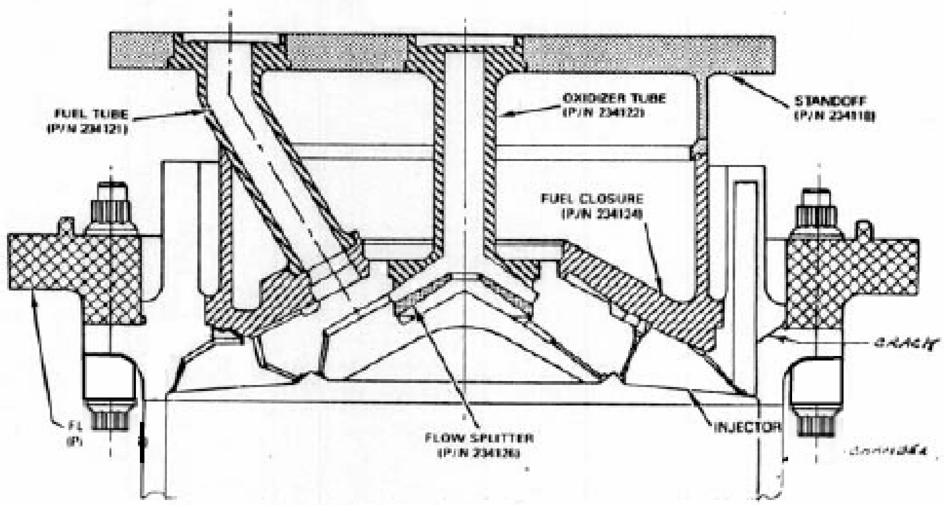


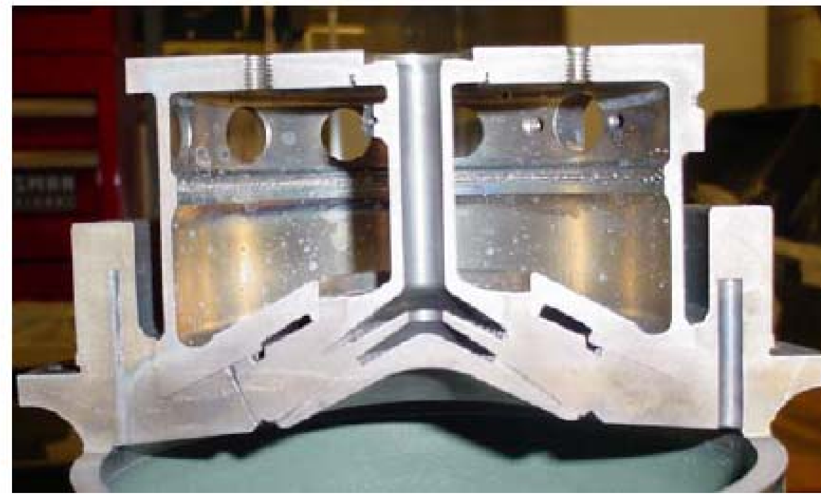
Thruster Cross-Section



RCS Injector

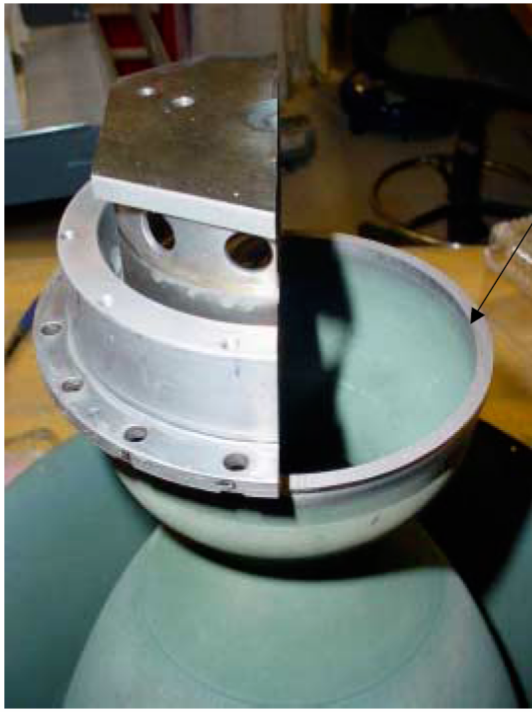
Injector Cross Section





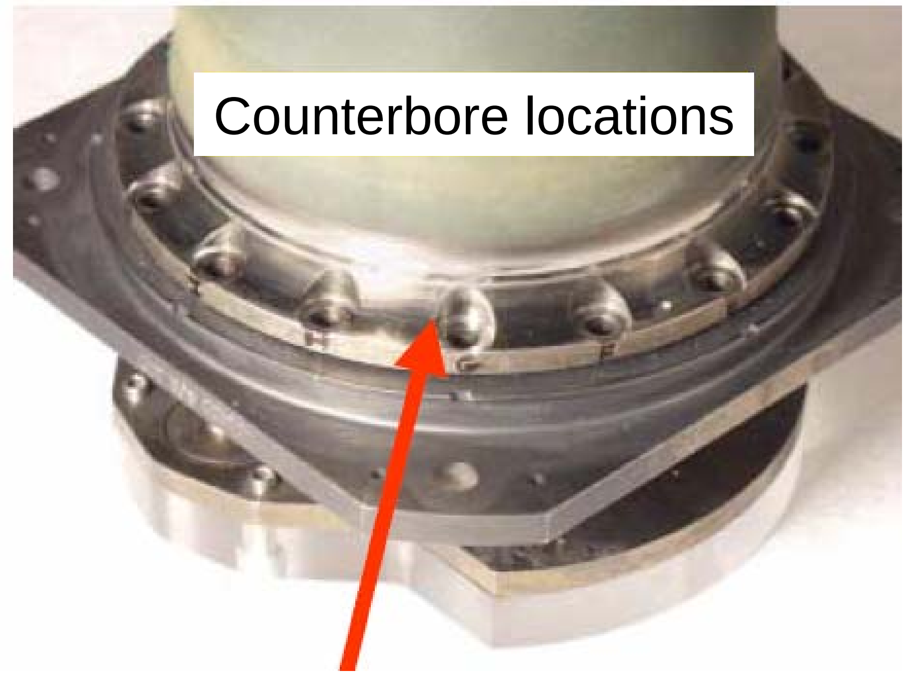
Sectioned S/N 120

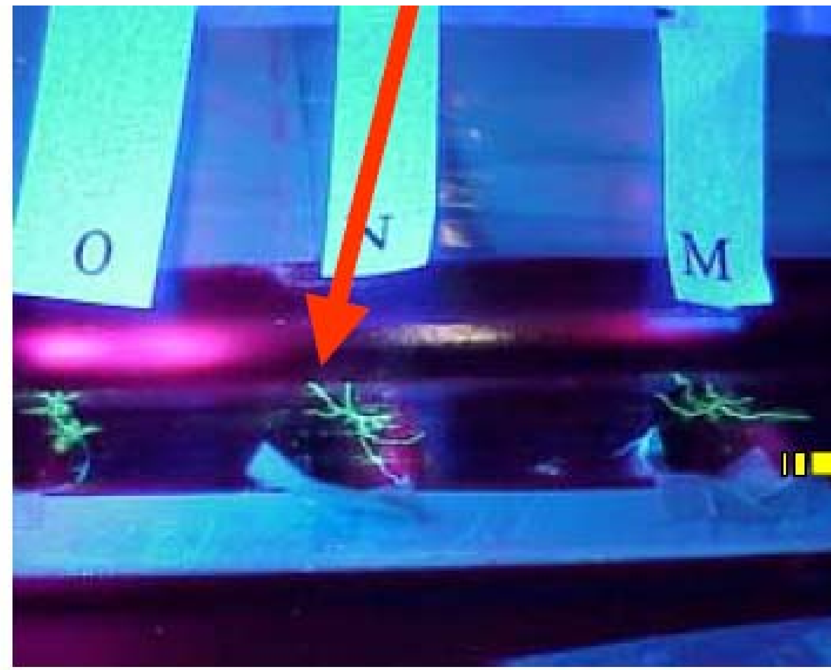
RCS Thruster, S/N 120



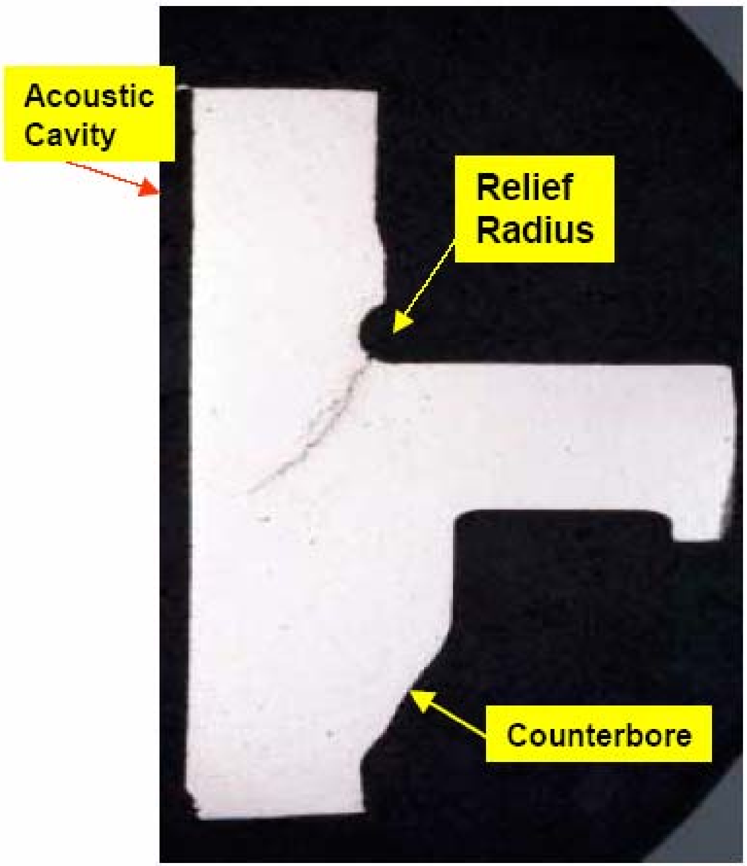
Thruster was sectioned through the EB weld using EDM

Counterbore Cracks

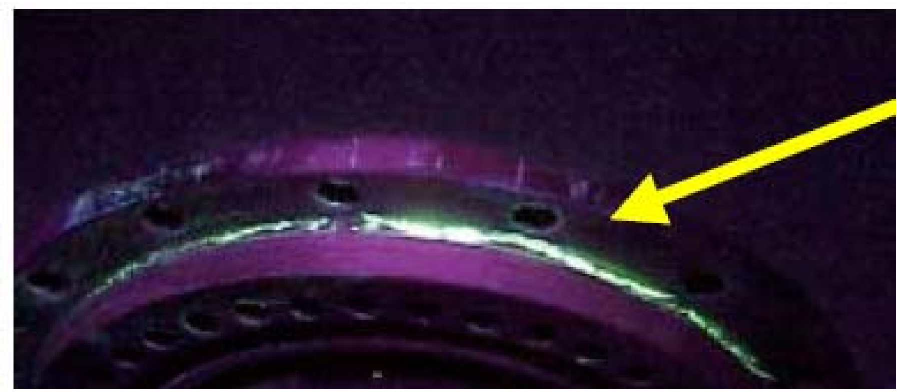




Relief Radius Cracks



Crack extending from ***relief radius*** towards acoustic cavity



Penetrant indication

RCS Thruster Cracking History

- **Three groupings of cracked thrusters**
- **1979 - SN 128, 130, 132**
  - No Direct cause of cracking found
    - Corrective actions
      - Developed ultrasonic inspection technique
      - Accomplished one time ultrasonic inspection of fleet
      - Added ultrasonic inspection to manufacturing flow (pre bake-out)
      - Completed 100 mission Qual test with SN 130 & 132
- **1982 - SN 322, 415, 416, 433**
  - Cracking attributed to HF etchant and 600 F insulation bake-out
    - Corrective actions
      - Eliminate etchants from manufacturing process
      - Added ultrasonic inspection immediately after bake-out cycle
- **2004 - SN 120**
  - Counter bore cracks found near new nozzle EB weld
  - First time such cracks found, discovered incidentally (penetrant inspection)
  - Relief radius cracks similar to 1979/1982 subsequently found

Thruster Manufacturing Timelines

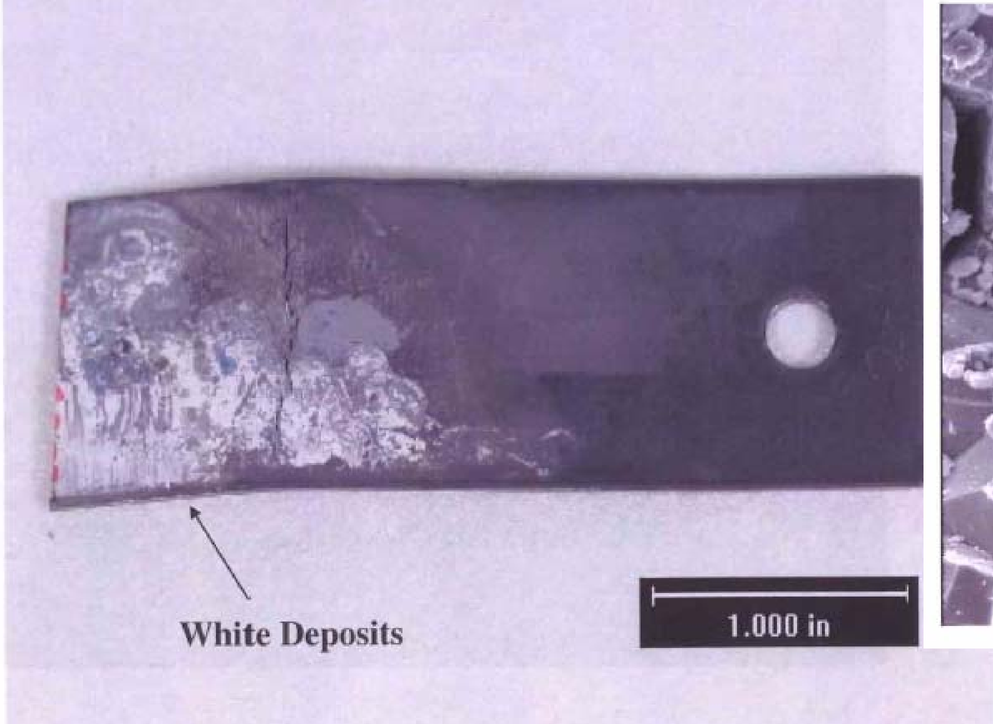
	1979			1982				2004
Thruster SN	128	130 Qual test	132 Qual test	322	415	416	433	120 High usage
Prebake ultrasonic	n/a	n/a	n/a	1/26/82 1/28/82 No Cracks found	No Cracks found	No Cracks found	No Cracks found	n/a
Leak Test	2/12/79	3/1/79	11/15/78	1/30/82				
Torque Bolts	2/13/79	3/2/79	11/16/78	1/30/82				
Insulation Bake	2/28/79			2/7/82				
Post bake Ultrasonic (One time fleet insp. after 128 found)	n/a	4/14/79 Cracked @ MAC	4/12/79 Cracked @ MAC	n/a	n/a	n/a	n/a	4/29/79 No cracks found
Post bake Ultrasonic (Mfg. Insp.Initiated after 322 found)	n/a	n/a	n/a	2/11/82 Cracked per Special Ultrasonic	12/14/82 Cracked	12/15/82 Cracked	12/7/82 Cracked	n/a
Post bake Leak test	3/14/79 Cracked	o.k.	o.k.	2/11/82 Cracked	n/a	n/a	n/a	o.k.
Penetrant Inspection @ WSTF	n/a	n/a	n/a	n/a	n/a	n/a	n/a	4/2/04 Cracked

Laboratory Reproduction of Injector Cracking

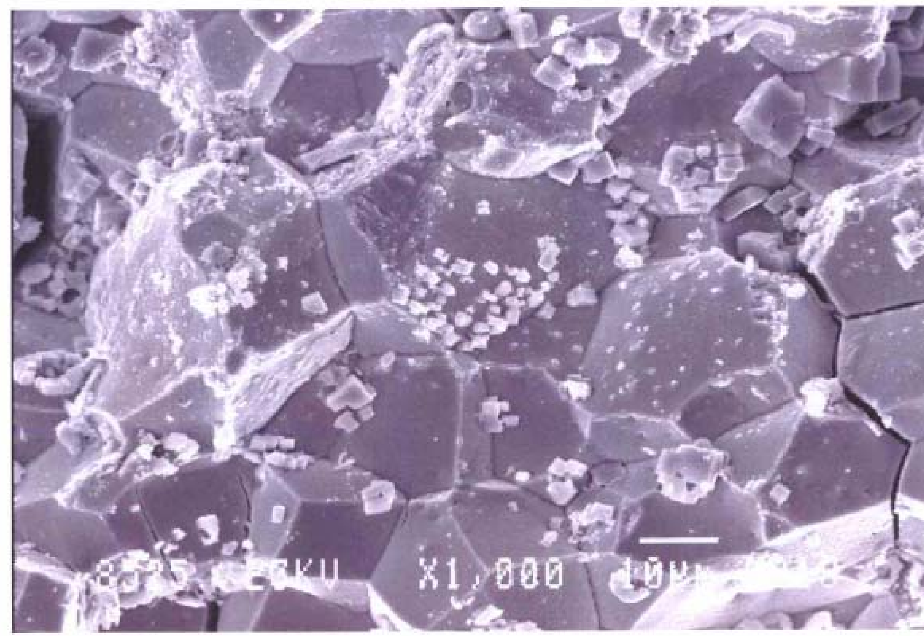
- Test Purpose
  - Identify Offending Species
  - Identify Conditions for Cracking
  - Establish Conditional Thresholds
    - Temperature
    - Stress Level
    - Time, etc.
- Test Protocol
  - Followed Guidance in 1982 CAR
    - Apply HF Etchant to C103
    - Cover with Titanium
    - Stress to 30 KSI
    - Heat to 600F for 48 hrs
- Laboratory Reproduction of Cracking Was Enhanced
  - When 1982 Cracked “Brownfield” Specimen Was Found
  - When Brownfield Fracture Surface Was Same As Thruster’s

The “Brownfield” Specimen…

OVERALL VIEW OF BEND SPECIMEN

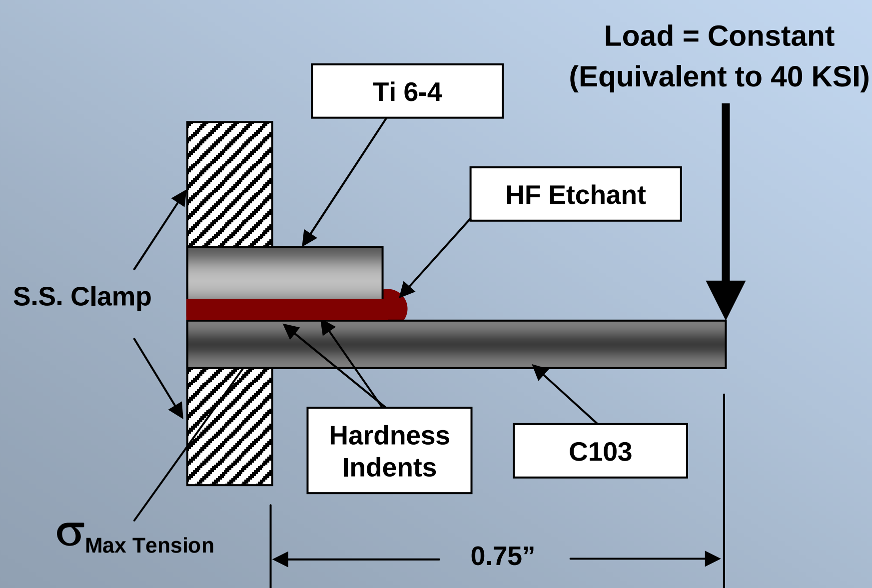


SEM OF SLOW GROWTH AREA



HF Etchant Tests/Specimen Loading

Cantilever Test Configuration



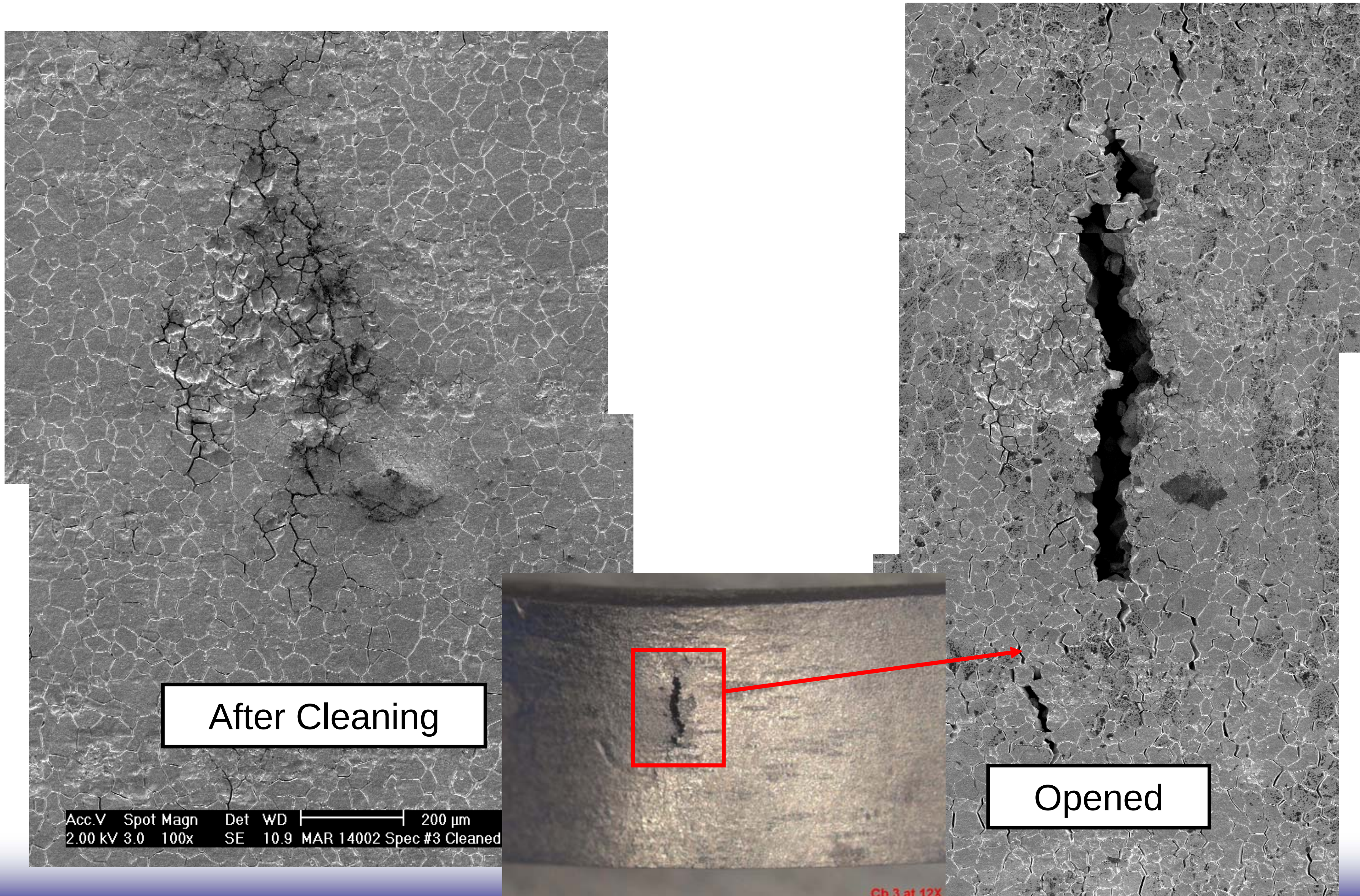
Variables = T, t, HF Concentration

- **Specimen Dimensions**
  - ≈ 1” X 0.2” X 0.040”
- **DMA Test Equipment**
  - Trend Load, Deflection, Temp, Time
  - Dry Air Purge

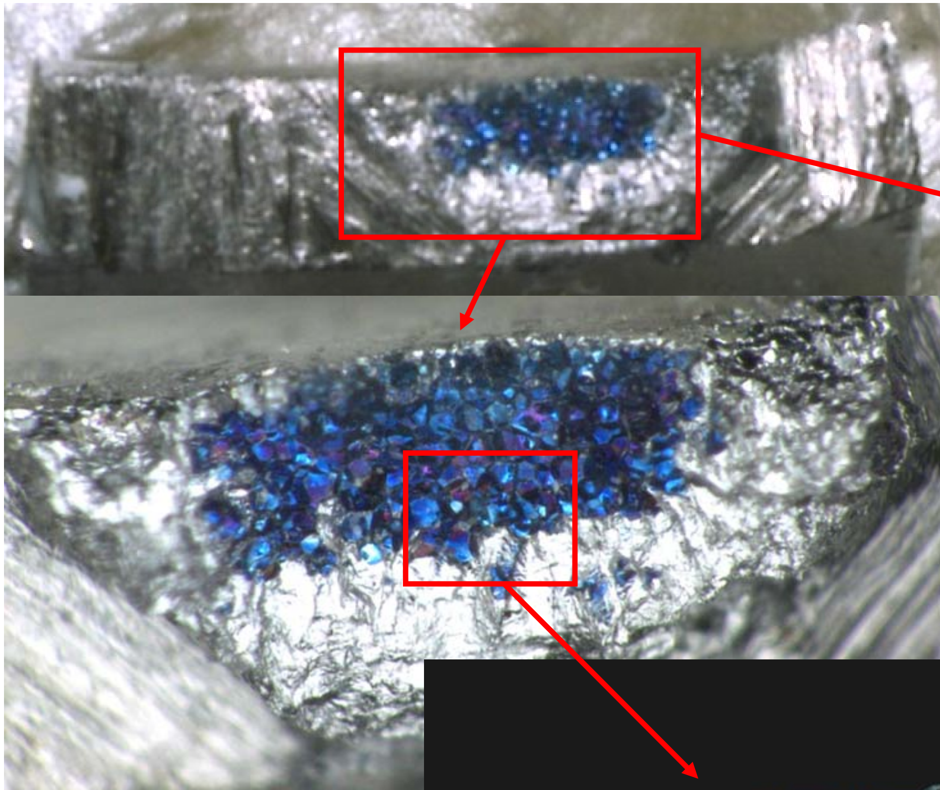
Generic Test Protocol:

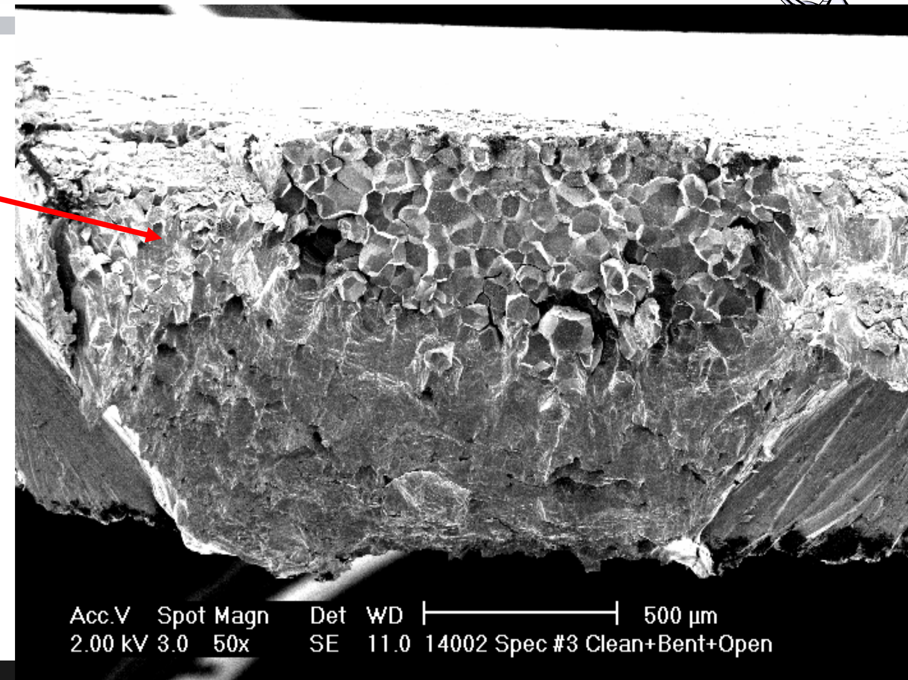
1. Cut & Prep C103 Test Sample
2. Place Hardness Indentations at Max Tensile Stress Location
3. Cover With Titanium, Clamp, & Apply Constant Load (Yield Strength / 40 KSI)
4. Apply HF Etchant to Crevice
5. Heating Profile, 400F-600F for 48 Hours
6. Remove from Test, Clean, Examine Under Microscope / SEM
7. Bend to Open Cracks
8. Examine Fracture Surface Morphology to Establish Failure Mode

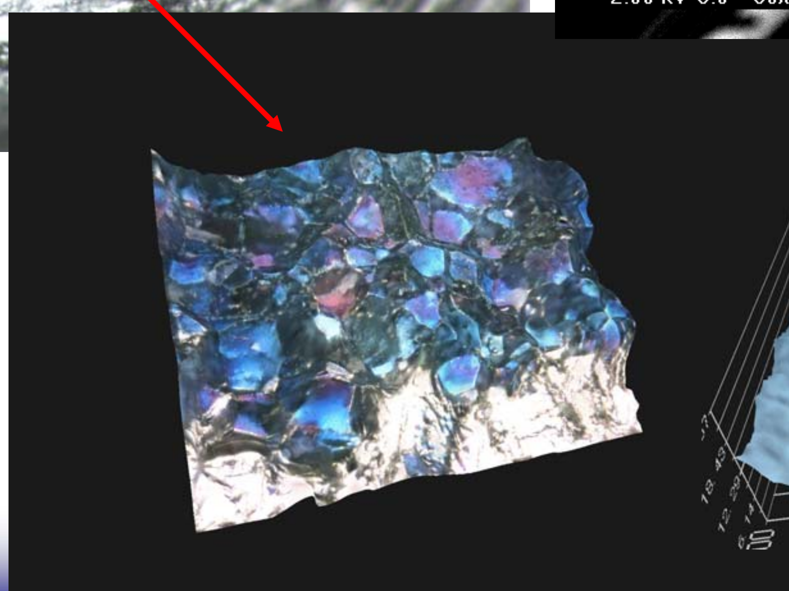
Specimen #3 – HF + 600F

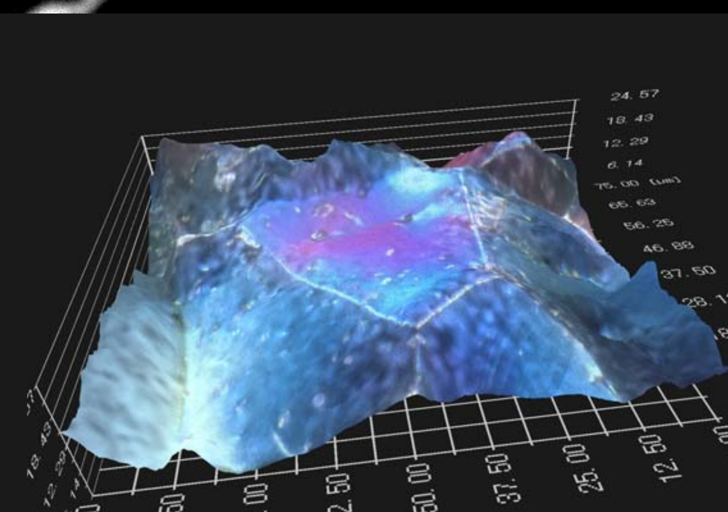


Specimen #3 – IG Fracture



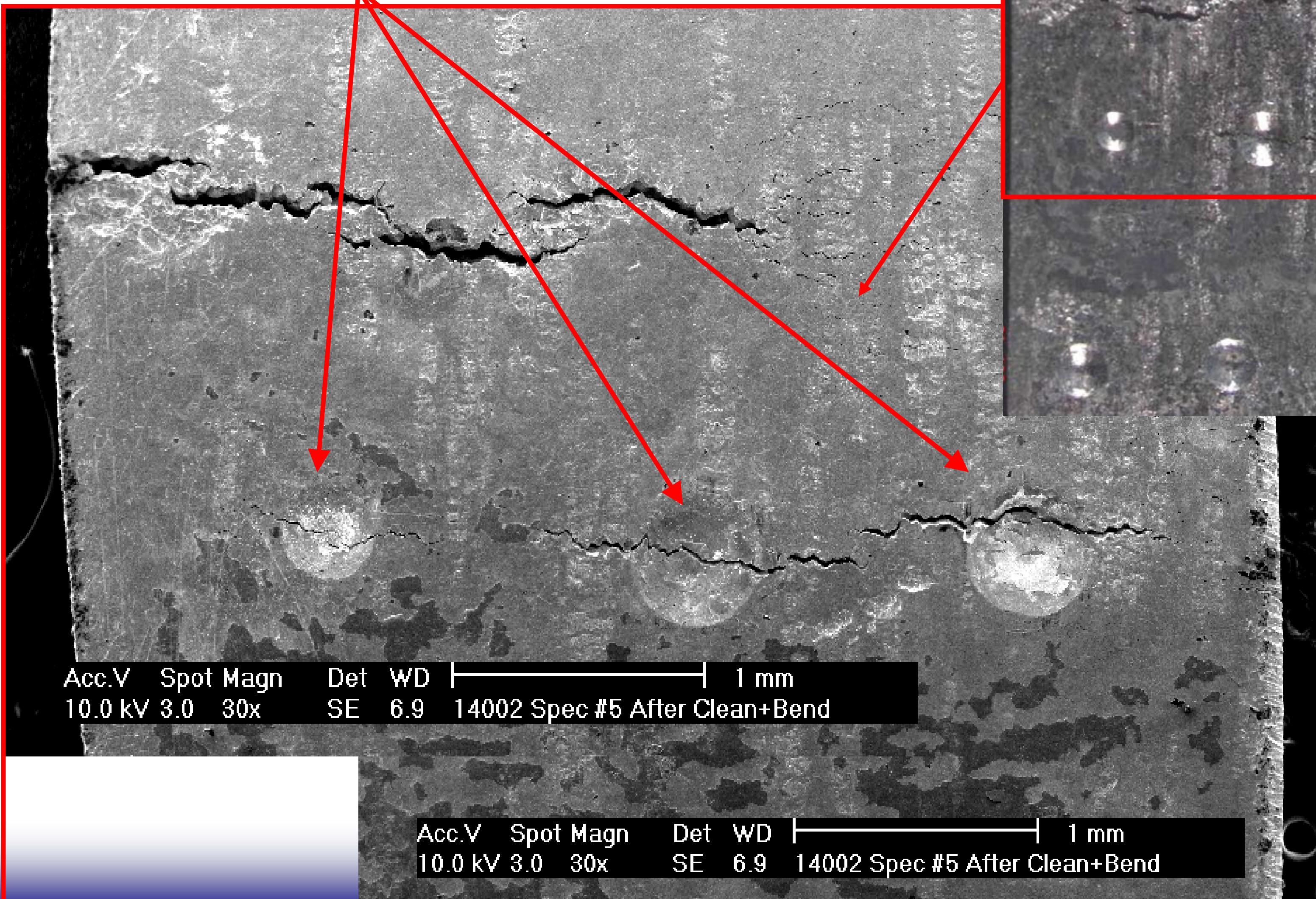


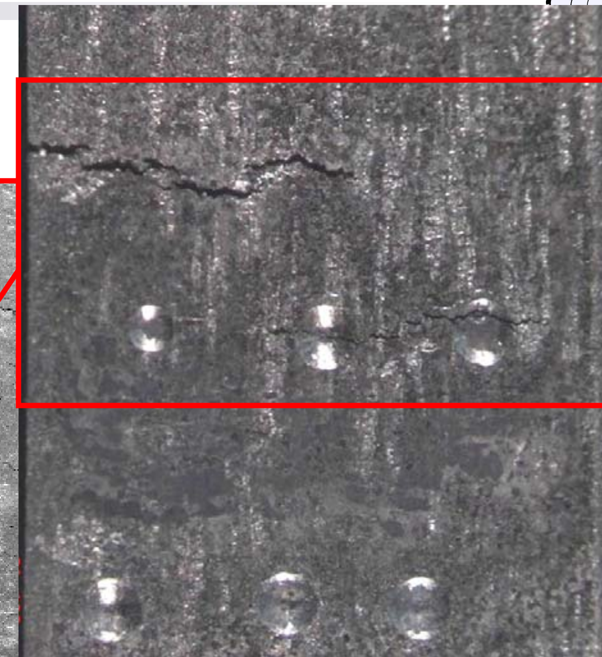




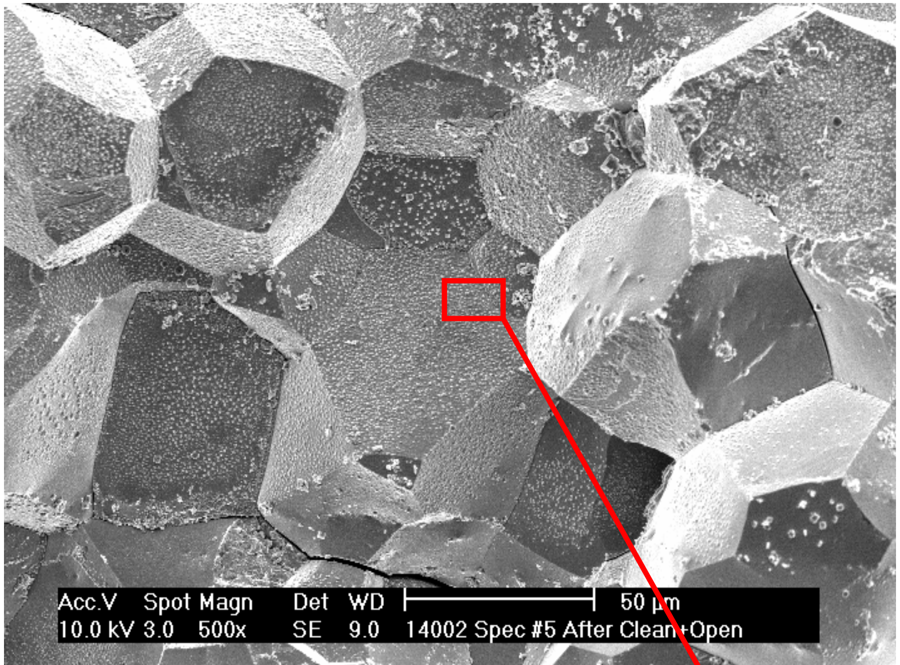
Specimen #5 – HF + 600F

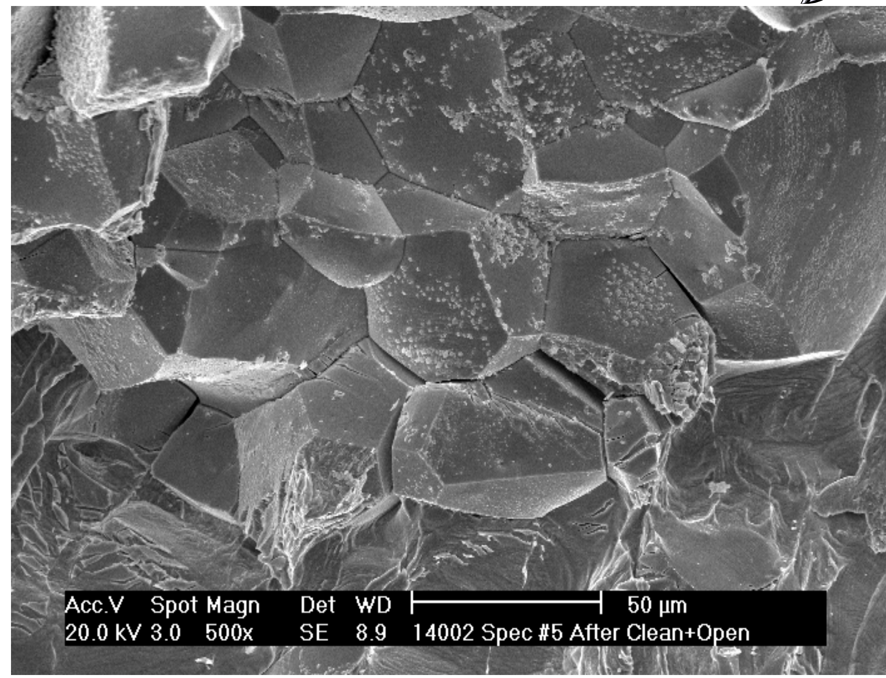
- 3 IG Crack Initiation Sites
  (Correspond to Hardness Indents)



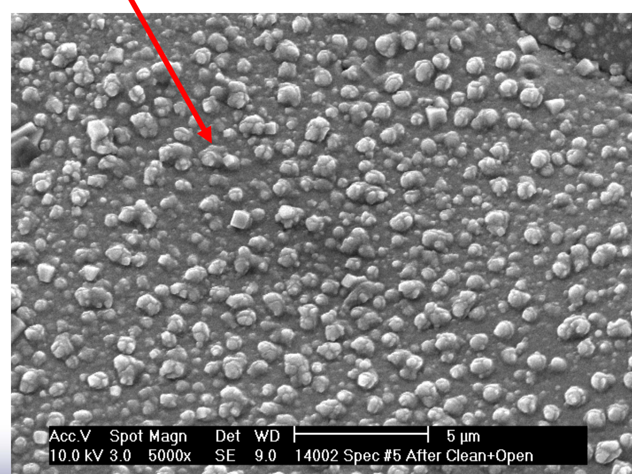


Specimen #5 – “Popcorn”



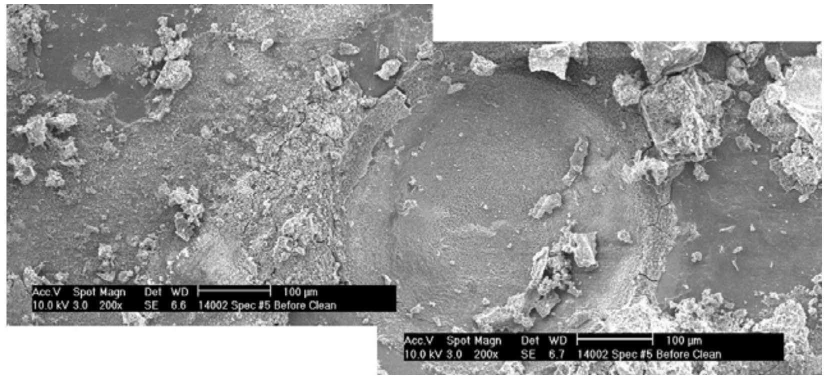


Near Origin

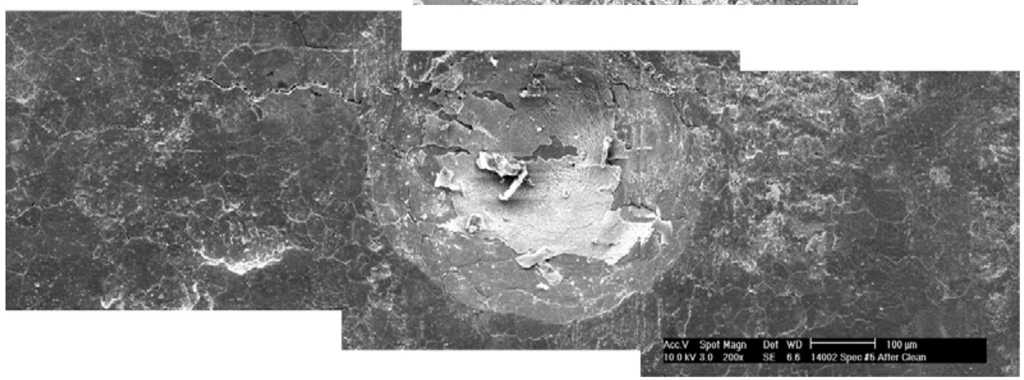


Near Termination

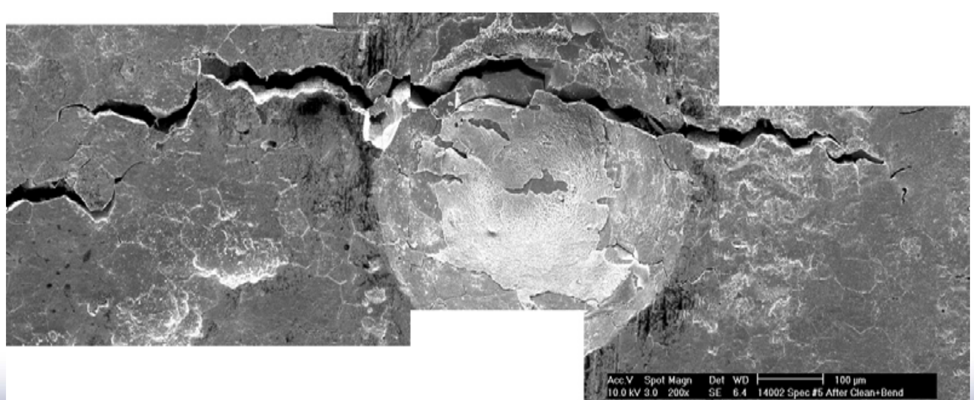
Specimen #5 – Cleaned and Bent



As-Tested



As-Cleaned



As-Bent

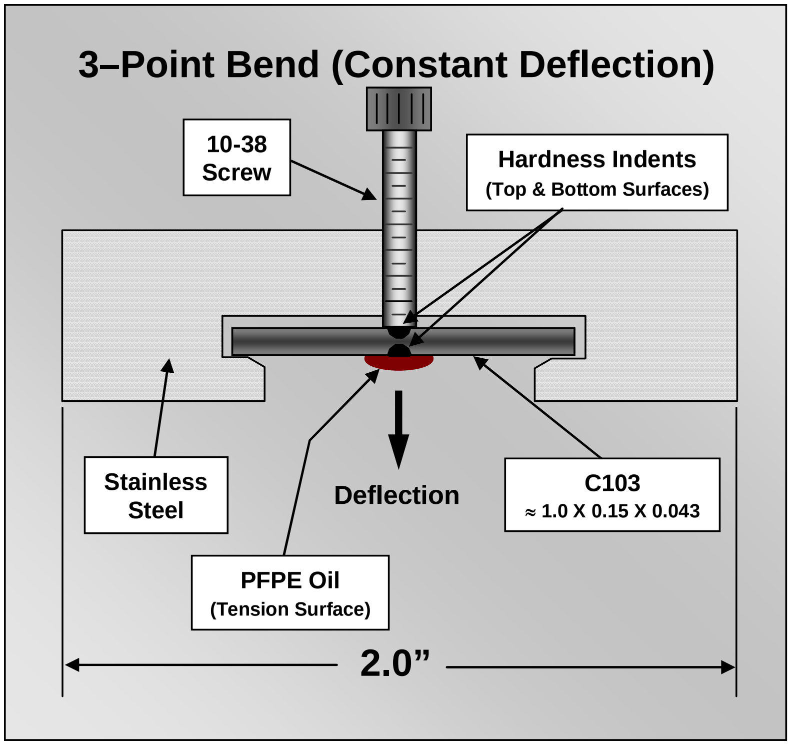
HF Exposure Test Matrix

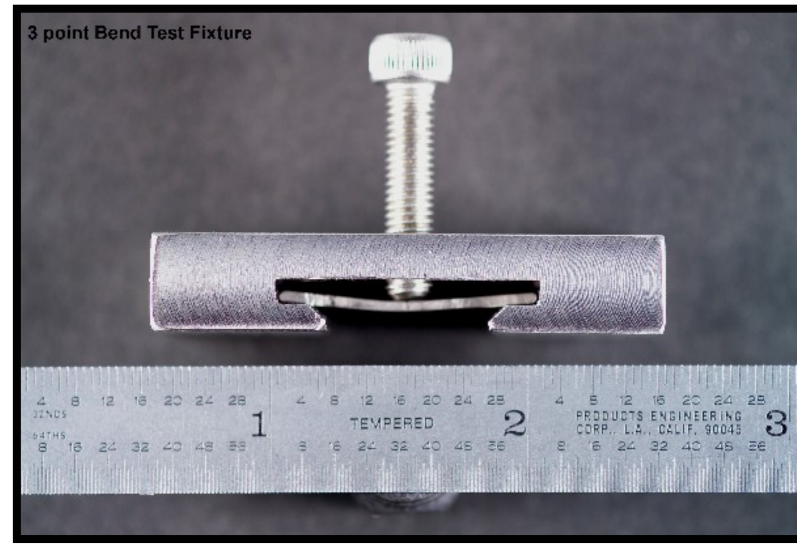
Specimen #	C103 Cantilever Beam Tests (Constant Load)	X = Cracks
1	Vise + Nb etch + Ti cover + dry + 40KSI + 600°F + 48hrs	
2	Vise + Nb etch + dry + Ti cover + 40KSI + 600°F + 48hrs	
3	Vise + Ti cover + 40KSI + Nb etch + dry + 600°F + 48hrs	**X**
4	Vise + Nb cover + 40KSI + Ti etch + dry + 600°F + 48hrs	
5	Vise/<u>Hard</u> + Ti cover + 40KSI + Nb etch + dry + 600°F + 48hrs	**X**
6	Vise/<u>Hard</u> + Ti cover + 40KSI + Nb etch + dry + <u>400°F</u> + 48hrs	
8	Vise/<u>Hard</u> + Ti cover + 40KSI + Nb etch + dry + <u>500°F</u> + 48hrs	**X (minor)**
9	Vise/<u>Hard</u> + Ti cover + 40KSI + Nb etch + dry + 600°F + 3hrs	**X**
10	Vise/<u>Hard</u> + Ti cover + 40KSI + Nb etch + dry + 600°F + 48hrs + 70°F + $H2O$ (3X/day) + 260hrs	**X**
11	Vise/<u>Hard</u> + Ti cover + 40KSI + Nb etch + dry + 600°F + 168hrs	**X**
12	Vise/<u>Hard</u> + Nb cover + 40KSI + Nb etch + dry + 600°F + 48hrs	**X**
13	See Table 2	
14	Hard + Ti cover + 40KSI + <u>air</u> + + 600°F + 48hrs	

Krytox 143AC Tests

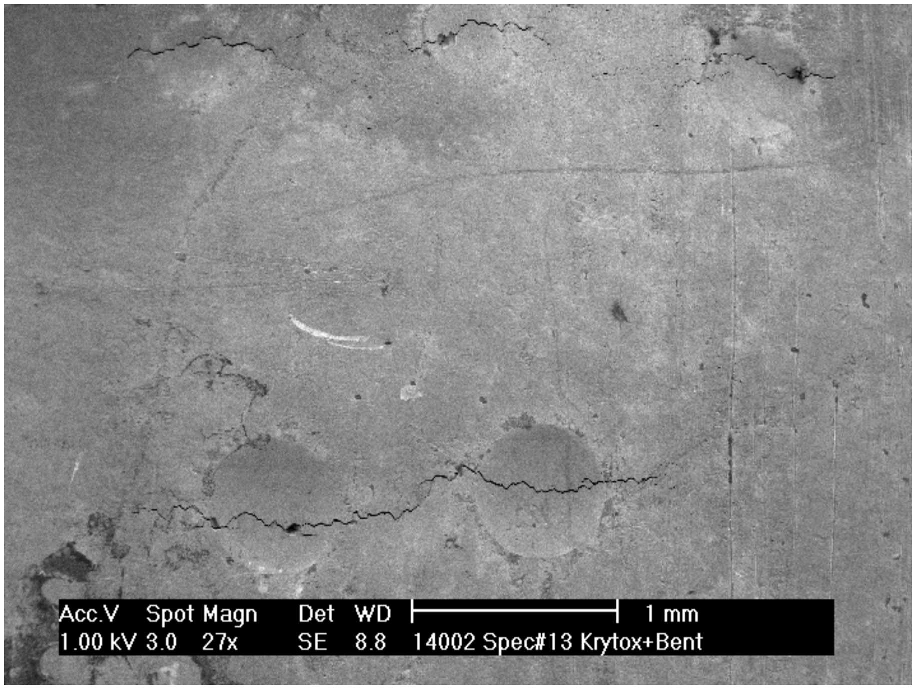
- What is Krytox?
  - Perfluoropolyether (PFPE) Polymer
  - Also Referred to as a Synthetic Oil
  - Composed of Carbon, Oxygen and Fluorine
- Why Test Krytox?
  - Used in Thruster Manufacturing
  - Contained Fluorine
  - Original Compatibility Tests at 1100F not 600F
  - XPS Analysis Showed PFPE on Fracture Surface

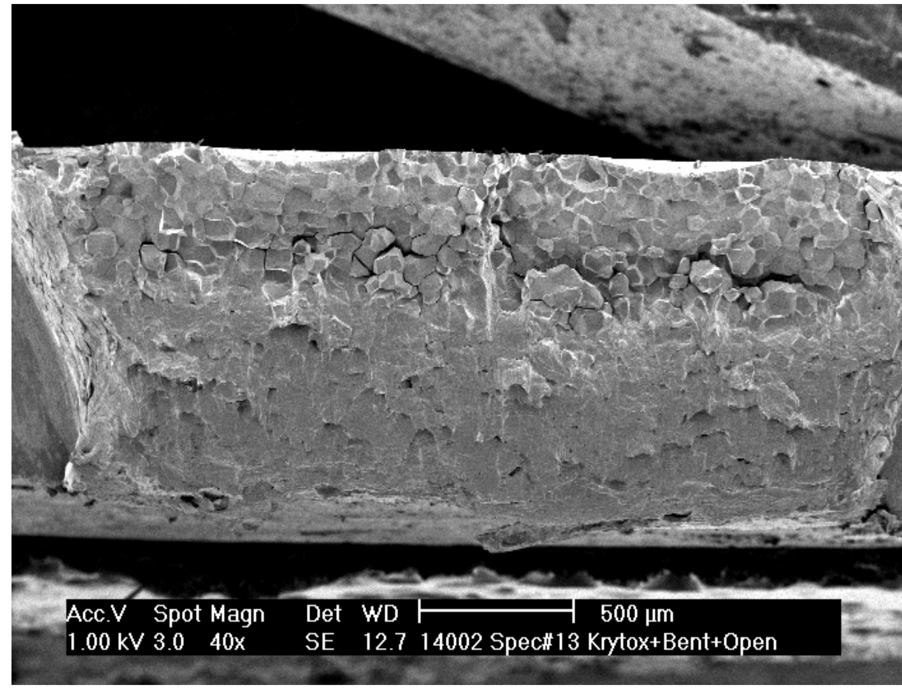
Krytox Tests/Specimen Loading



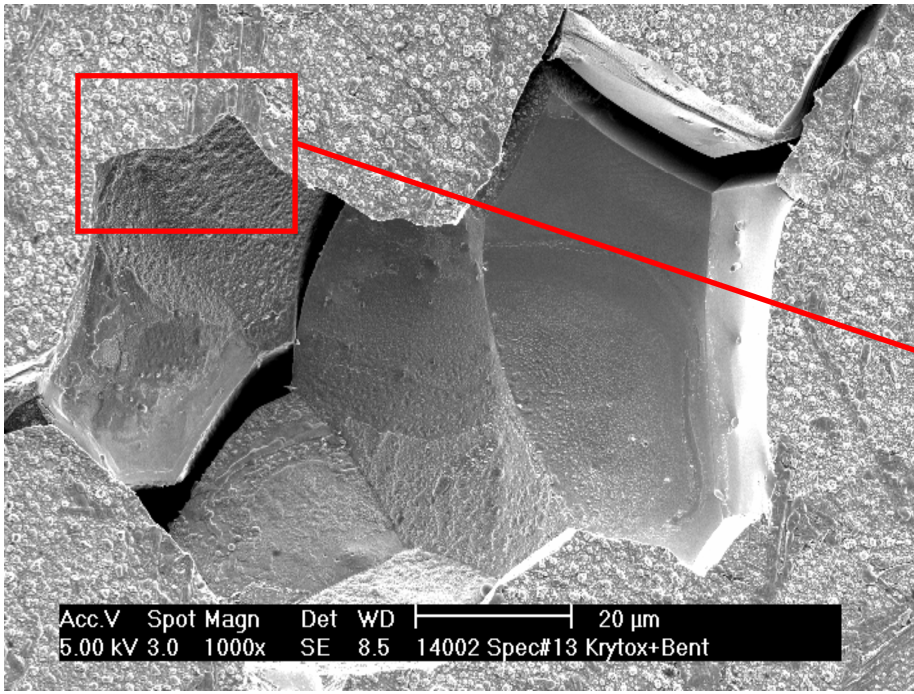


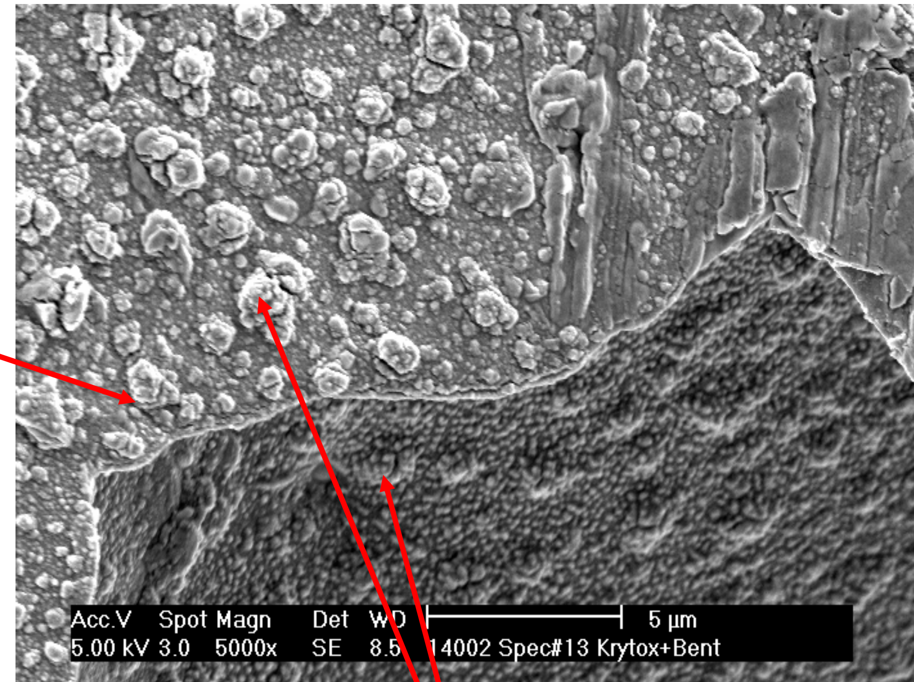
Specimen #13 – Krytox + 600F





Specimen #13 – Krytox + 600F





Missing Grains at Crack Origin

"Popcorn" on Free Surface + IG Crack

Krytox Exposure Test Matrix

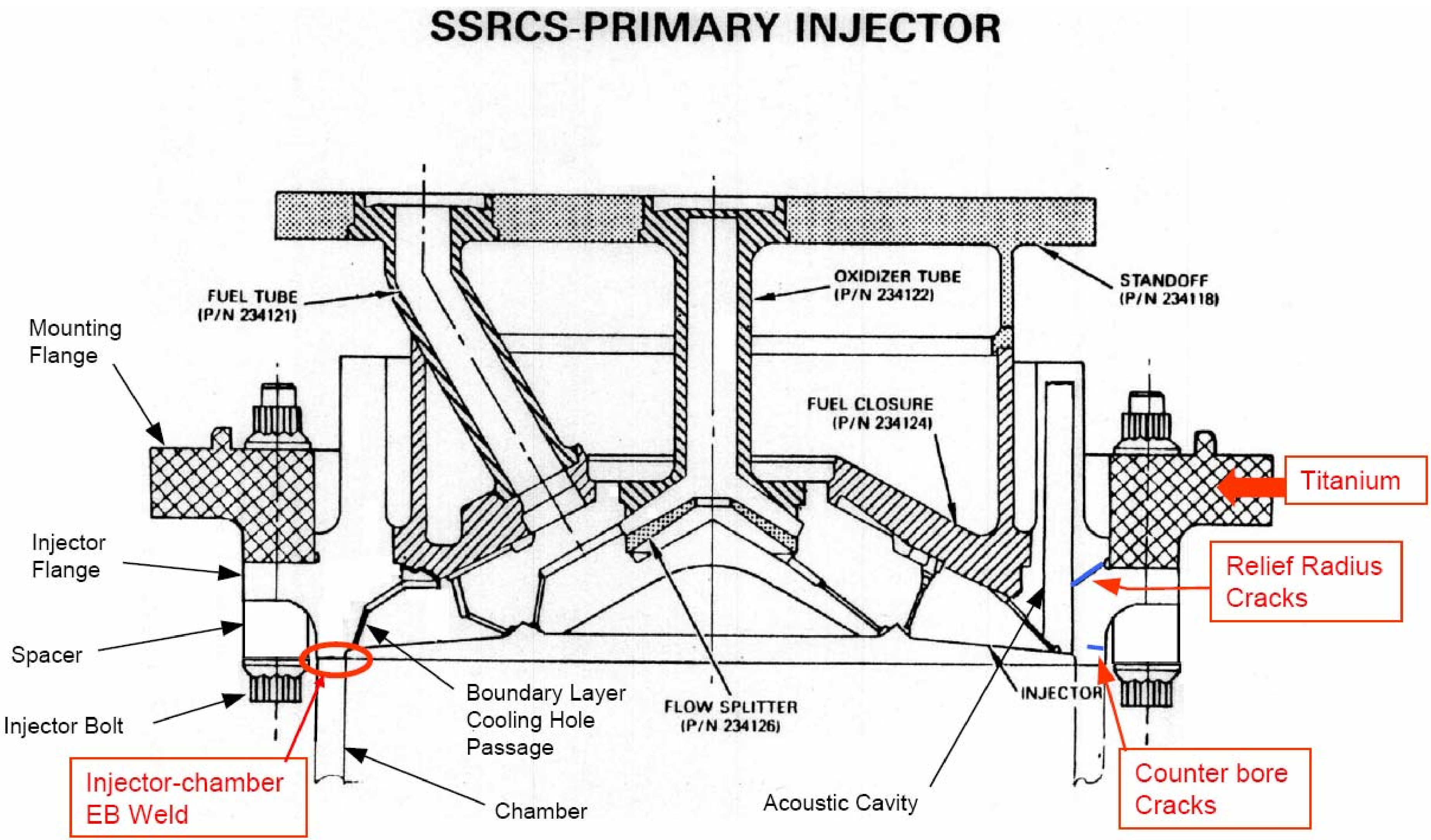
Specimen #	C-103 Cantilever Beam Tests (Constant Load)	X = Cracks
13	Hardness + Ti cover + 40KSI + Krytox + 600°F + 48hrs	X
14	*See Table I*	
15	Hardness + Ti cover + 40KSI + Krytox + 400°F + 48hrs	
16	Hardness + Ti cover + 40KSI + Krytox + 500°F + 48hrs	X (minor)
17	Hardness + Ti cover + 40KSI + Krytox + 600°F + 96hrs	X
18	Hardness + Ti cover + 40KSI + Krytox + 600°F + 3hrs	X
Specimen #	**C-103 Three-Point-Bend Tests (Constant Deflection)**	
29	Hardness + no Ti + >45KSI + Krytox + 600°F + 1hrs	X
30	No indent + no Ti + >45KSI + Krytox + 600°F + 1hrs	
31	Hardness + no Ti + >45KSI + Brayco + 600°F + 1hrs	
Specimen #	**Cb752 Three-Point-Bend Tests (Constant Deflection)**	
32	Hardness + no Ti + >45KSI + Brayco + 600°F + 23 hrs	X

Conclusions / Recommendations

- Cracks Successfully Reproduced in Laboratory
- Simultaneous Conditions Necessary for Cracking
  - Mechanically Disturbed/Cold Worked Surface
  - Externally Applied Sustained Stress near Yield
  - Fluorine Containing Fluid
  - Sustained Temperature above 400F
- Cracking Occurred During Manufacturing
  - Only Time all Four Conditions act Simultaneously
  - Eliminate use of Fluorine with Niobium
- Same Cracking Mechanism Operating in HF and PFPE Fluids
- Specific Cracking Mechanism not Identified

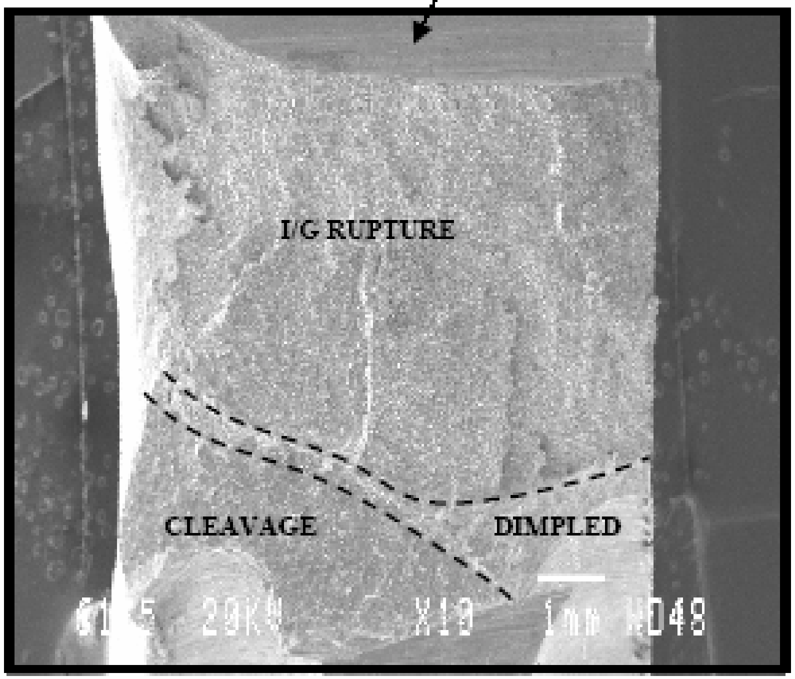
Back-Up

Location Of Cracks

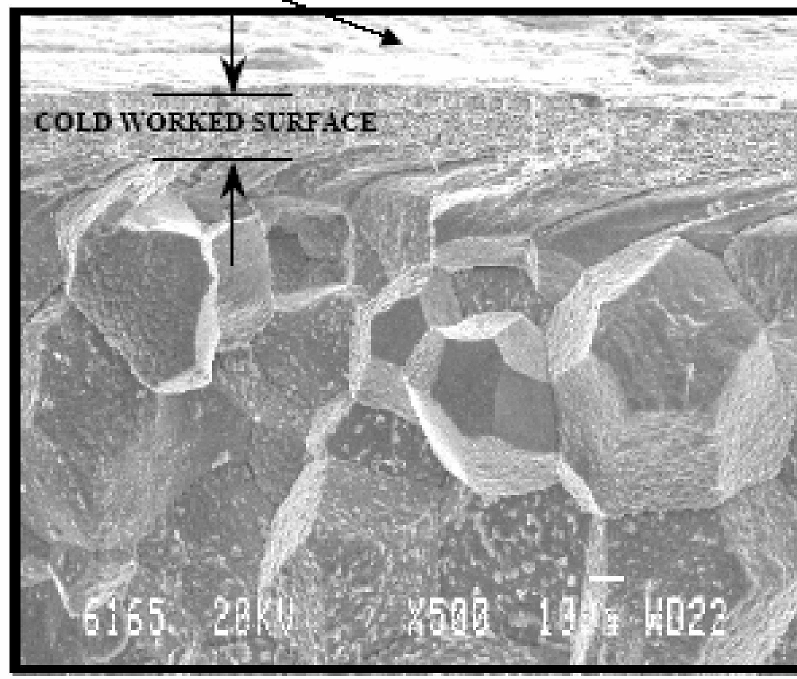


Fracture Face of Relief Radius Crack

Machined Radius



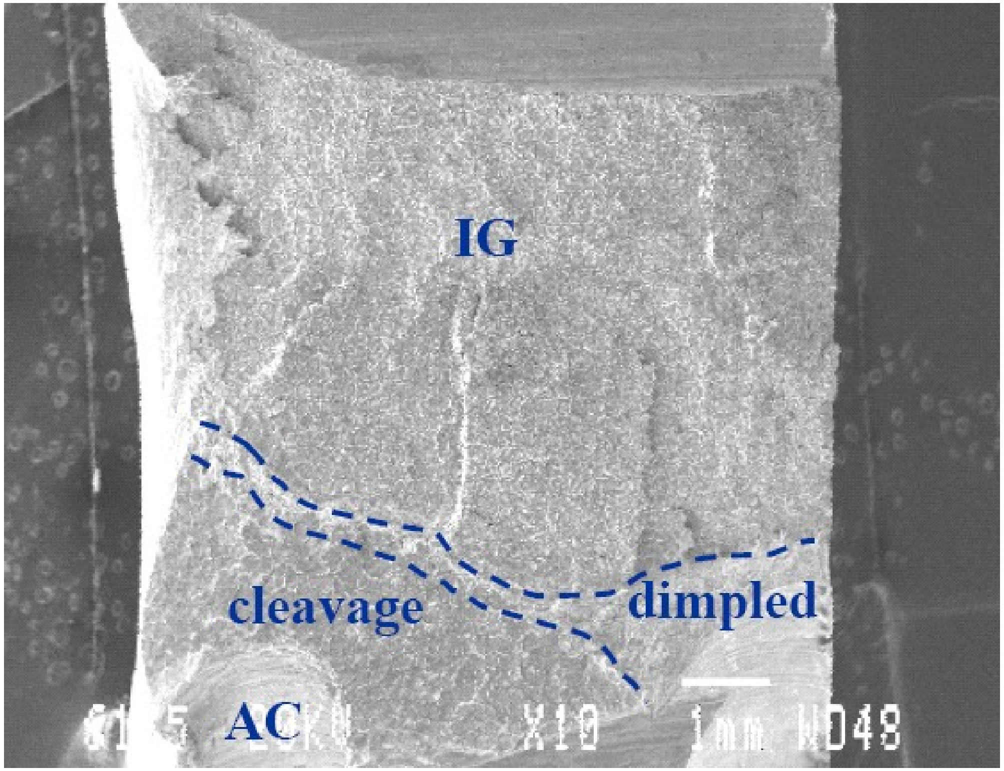
Overview of fracture face

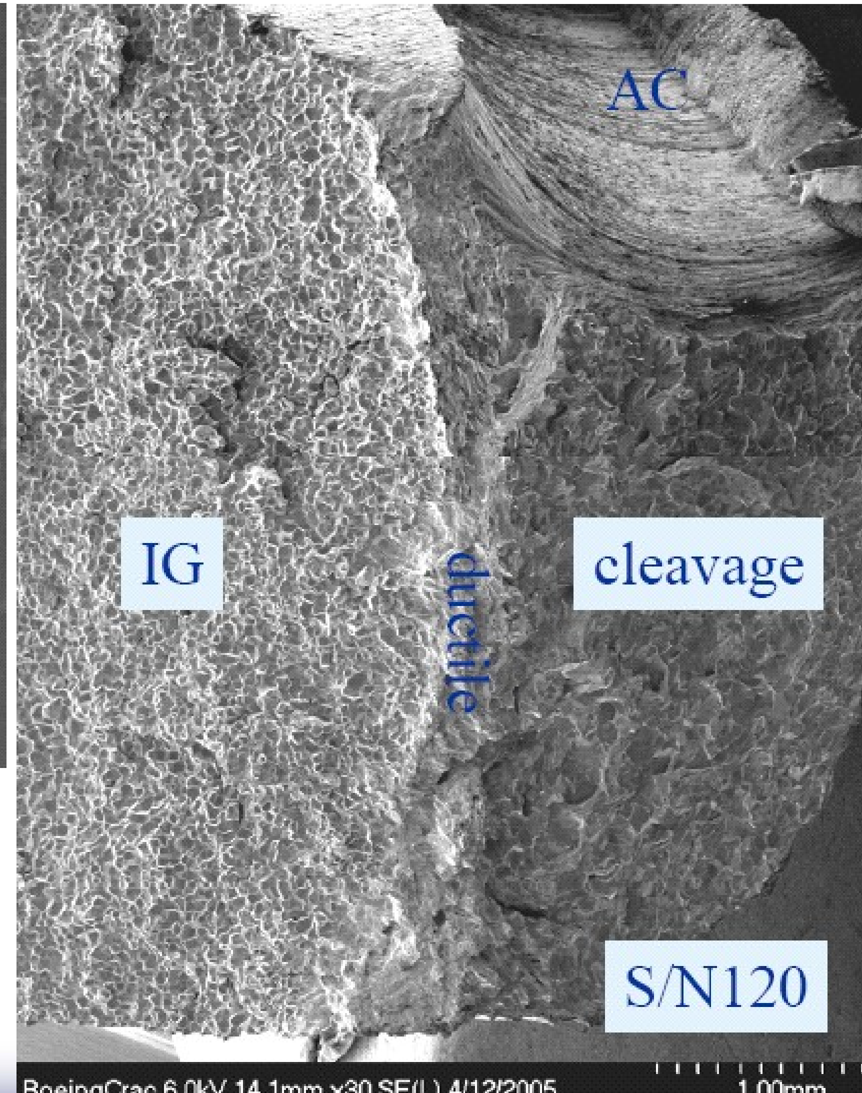


Cold worked surface layer and intergranular facets in failure origin area

Cleavage Fracture on S/N 120

Opened Relief Radius (Between Holes J & K)



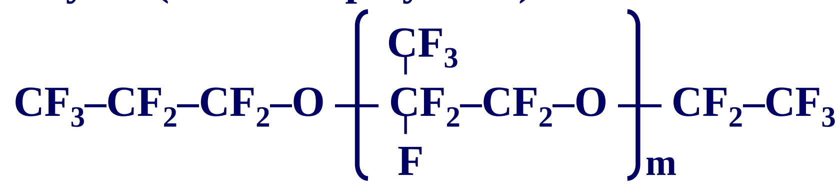


Comparison of Thruster Failure Analysis Findings

	1979			1982				2004
Thruster SN	128	130	132	322	415	416	433	120
Crack Initiated in Mfg.	YES	YES	YES	YES	YES	YES	YES	YES
Cracked 100% Intergranular	YES	YES	YES	YES	YES	YES	YES	YES
Fracture Discolored	YES	YES	YES	YES	YES	YES	YES	YES
Surface Chemistry	F,O,C	F	F,O,C	F	F	F	F	F,O,C
Crack Extension Beyond Discoloration	No	NO	NO	NO	NO	NO	NO	NO
Crack Extension In service/Qual Test	No service history	Qual, No	Qual, No	No service history	No service history	No service history	No service history	17 flights, No

Chemical Breakdown of Krytox PFPE Polymer

Krytox (Perfluoropolyether)



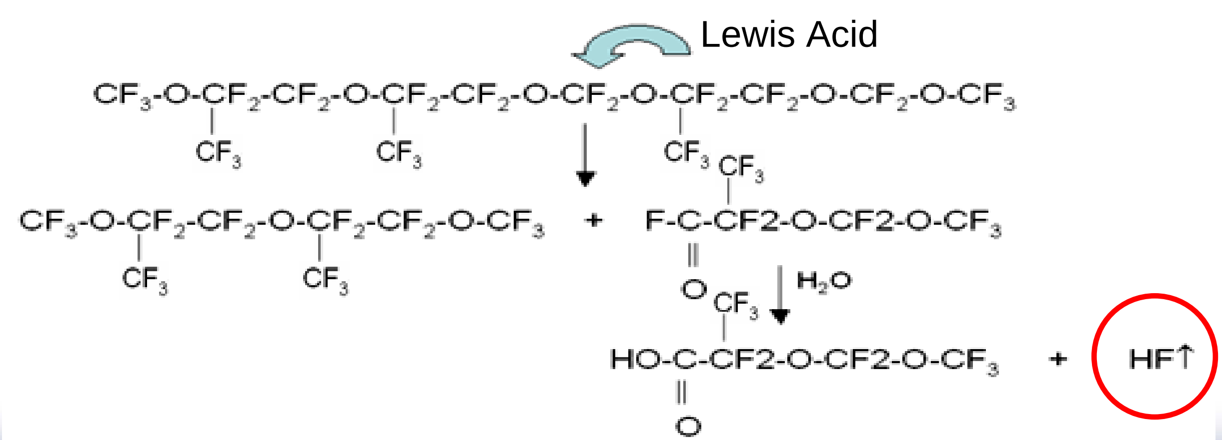
Discussion

- Chemical breakdown of the PFPE polymer chain is a well known phenomena to the lubrication community.
- The mechanism tends to follow three steps:
  1. The metal/oxide surface reacts with fluorine from the PFPE to form a Lewis acid (i.e. NbF3 / NbF5).
  2. The Lewis acid catalyzes the cleavage of the polymer chain.
  3. The broken polymer chain can react with water to form a carboxylic acid terminated group releasing hydrofluoric acid.

STEP 1

STEP 2

STEP 3



Injector Crack Flight Rationale Road-Map