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

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&
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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
RCS Thruster

- Electron beam weld joining the nozzle to the injector
- Flange containing the 16 counterbored holes
Thruster Cross-Section
Sectioned S/N 120

Injector Cross Section
RCS Thruster, S/N 120

Thrust was sectioned through the EB weld

Acoustic Cavities

Hole H

Counterbored Holes

Hole I

Hole J

Hole K

Hole L

Hole M

Hole N
Counterbore Cracks

Counterbore locations
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

<table>
<thead>
<tr>
<th>Thruster SN</th>
<th>Prebake ultrasonic</th>
<th>Leak Test</th>
<th>Torque Bolts</th>
<th>Insulation Bake</th>
<th>Post bake Ultrasonic (One time fleet insp. after 128 found)</th>
<th>Post bake Ultrasonic (Mfg. insp.Initiated after 322 found)</th>
<th>Post bake Leak test</th>
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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…
HF Etchant Tests/Specimen Loading

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

Cantilever Test Configuration

- Load = Constant (Equivalent to 40 KSI)
- Ti 6-4
- HF Etchant
- S.S. Clamp
- Hardness Indents
- C103
- 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
Specimen #3 – IG Fracture
Specimen #5 – HF + 600F

• 3 IG Crack Initiation Sites
  (Correspond to Hardness Indents)
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/Hard + Ti cover + 40KSI + Nb etch + dry + 600°F + 48hrs | X  
6 | Vise/Hard + Ti cover + 40KSI + Nb etch + dry + 400°F + 48hrs |  
8 | Vise/Hard + Ti cover + 40KSI + Nb etch + dry + 500°F + 48hrs | X (minor)  
9 | Vise/Hard + Ti cover + 40KSI + Nb etch + dry + 600°F + 3hrs | X  
10 | Vise/Hard + Ti cover + 40KSI + Nb etch + dry + 70°F + H2O (3X/day) + 260hrs + 600°F + 48hrs | X  
11 | Vise/Hard + Ti cover + 40KSI + Nb etch + dry + 600°F + 168hrs | X  
12 | Vise/Hard + Nb cover + 40KSI + Nb etch + dry + 600°F + 48hrs | X  
13 | See Table 2 |  
14 | Hard + Ti cover + 40KSI + air + + 600°F + 48hrs |  

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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

3-Point Bend (Constant Deflection)

Deflection

Hardness Indents
(Top & Bottom Surfaces)

Stainless Steel

C103
≈ 1.0 x 0.15 x 0.043

10-38 Screw

PFPE Oil
(Tension Surface)

Deflection

2.0”
Specimen #13 – Krytox + 600F
Specimen #13 – Krytox + 600F

- Missing Grains at Crack Origin
- “Popcorn” on Free Surface + IG Crack
## Krytox Exposure Test Matrix

### C-103 Cantilever Beam Tests (Constant Load)

<table>
<thead>
<tr>
<th>Specimen #</th>
<th>C-103 Cantilever Beam Tests (Constant Load)</th>
<th>X = Cracks</th>
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<td>Hardness + Ti cover + 40KSI + Krytox + 600°F + 48hrs</td>
<td>X</td>
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<td>14</td>
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<td>15</td>
<td>Hardness + Ti cover + 40KSI + Krytox + 400°F + 48hrs</td>
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<tr>
<td>16</td>
<td>Hardness + Ti cover + 40KSI + Krytox + 500°F + 48hrs</td>
<td>X (minor)</td>
</tr>
<tr>
<td>17</td>
<td>Hardness + Ti cover + 40KSI + Krytox + 600°F + 96hrs</td>
<td>X</td>
</tr>
<tr>
<td>18</td>
<td>Hardness + Ti cover + 40KSI + Krytox + 600°F + 3hrs</td>
<td>X</td>
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### C-103 Three-Point-Bend Tests (Constant Deflection)

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<th>C-103 Three-Point-Bend Tests (Constant Deflection)</th>
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<tbody>
<tr>
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<td>Hardness + no Ti + &gt;45KSI + Krytox + 600°F + 1hrs</td>
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<tr>
<td>31</td>
<td>Hardness + no Ti + &gt;45KSI + Brayco + 600°F + 1hrs</td>
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### Cb752 Three-Point-Bend Tests (Constant Deflection)

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<tbody>
<tr>
<td>32</td>
<td>Hardness + no Ti + &gt;45KSI + Brayco + 600°F + 23 hrs</td>
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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

SSRCS-PRIMARY INJECTOR

- Mounting Flange
- Injector Flange
- Spacer
- Injector Bolt
- Boundary Layer Cooling Hole Passage
- Chamber
- Acoustic Cavity
- FUEL TUBE (P/N 234121)
- OXIDIZER TUBE (P/N 234122)
- FUEL CLOSURE (P/N 234124)
- STANDOFF (P/N 234118)
- FLOW SPLITTER (P/N 234126)

- Injector-chamber EB Weld
- Relief Radius Cracks
- Counter bore Cracks
- Titanium
Fracture Face of Relief Radius Crack

Overview of fracture face

Machined Radius

I/G RUPTURE

CLEAVAGE

DIMPLED

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

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<th>1982</th>
<th>2004</th>
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<td>130</td>
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<td>Crack Initiated in Mfg.</td>
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<td>YES</td>
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<td>YES</td>
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<td>Cracked 100% Intergranular</td>
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<td>YES</td>
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<td>Crack Extension Beyond Discoloration</td>
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<td>No service history</td>
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- **Crack Initiated in Mfg.**: YES indicates crack initiated during manufacture.
- **Cracked 100% Intergranular**: YES indicates 100% of the surface is cracked.
- **Fracture Discolored**: YES indicates discolored surface.
- **Surface Chemistry**: F,O,C indicates presence of fluorine, oxygen, and carbon.
- **Crack Extension Beyond Discoloration**: NO indicates no extension beyond discoloration.
- **Crack Extension In service/Qual Test**: "No service history" indicates no history of crack extension in service or qualification tests.
- **17 flights, No**: Indicates 17 flights with no service history.
Chemical Breakdown of Krytox PFPE Polymer

Krytox (Perfluoropolyether)

\[
\text{CF}_3\text{--CF}_2\text{--CF}_2\text{--O} \begin{array}{c} \text{CF}_3 \\ \text{CF}_2\text{--CF}_2\text{--O} \end{array} \text{CF}_2\text{--CF}_3
\]

**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.

![Chemical breakdown diagram](image-url)
Injector Crack Flight Rationale Road-Map

- Conditions for cracking no longer exist
- M&P "popcorn" oxidization theory
- All thrusters stability screened
- WSTF OMDP helium mass spec external leak test requirement

- All thrusters passed a 525 psig external leak test during original ATP.
- S/N 130 & 132 qual test history
- Risk of on-orbit instability is low

- Injectors are leak free
- Existing cracks do not propagate due to service conditions
- Instabilities could possibly induce external leakage (S/N 132 history)
- Thrusters routinely screened for leakage

- WSTF OMDP helium mass spec external leak test requirement (50 psig)
- S/N 120 flight history
- 20+ years of service for etchant exposed thrusters with no external leakage
- Effect of external leakage is not catastrophic

- Consistency across all F/A's despite usage
- Pcb tube burn through test history indicates small flow paths would likely plug with combustion residue
- Impingement areas understood and risks are minimal

- Minimal risk to flight safety

Possible to add KSC turnaround external leak test requirement if absolutely necessary