

Effect of Crevice Former on Corrosion Damage Propagation

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- **The views, opinions, findings, and conclusions or recommendations of authors expressed herein do not necessarily state or reflect those of the DOE/OCRWM/OST&I.**

Objective

- **To determine the effect of the crevice former on the localized corrosion damage propagation**
- **Focus on post initiation stage, crevice propagation and arrest processes**
- **Determine the evolution of damage – severity, shape, location/distribution, damage profile**
- **Model of crevice corrosion propagation, i.e. the evolution of the crevice corrosion damage profile**

Approach

- **A combination of experimental measurements of localized corrosion with various crevice formers and analytical computations of the crevice damage profile as a function of crevice geometry, environment and corrosion resistance of the metal**
- **Preliminary results**
 - **Experimental**
 - **Analytical /computation**
- **Effect of crevice former material**
 - **Ceramic support with Poly Tetra Fluoro Ethylene (PTFE) tape**
 - **Polymer: PTFE, Polychlorotrifluoroethylene (PCTFE)**
 - **Ceramic: Al_2O_3**
 - **Metal**

Test Cell and Specimen

Test Cell



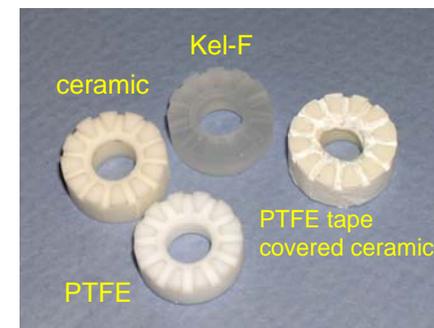
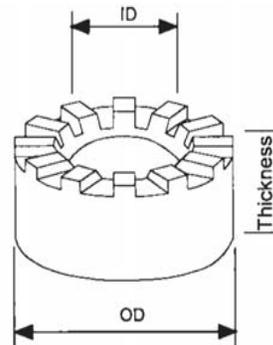
Specimen assembly

Multiple crevice assembly (MCA)



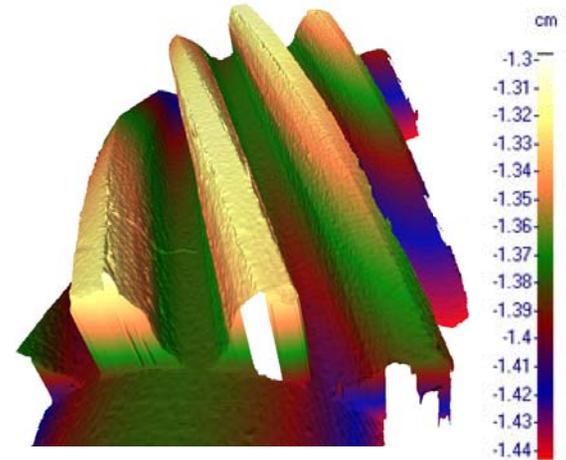
- Two segmented washers
- Applied Torque: 70 in-lb

Crevice formers: after ASTM G48-03



- 12 contact plateaus (feet)
- ID: 9.9 mm OD: 15.9 mm Thickness: 6.3 mm
- Contact area: about 6 mm²/foot

3D Measurement of Corrosion



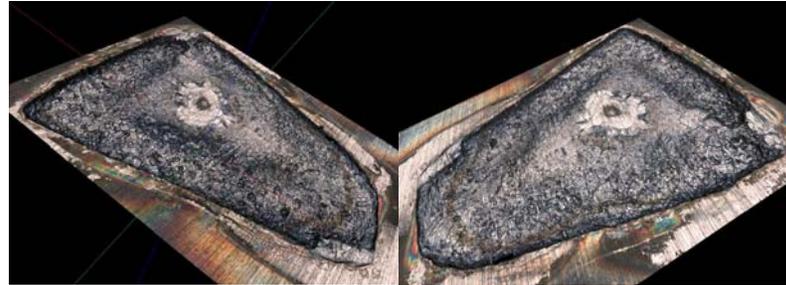
- **Optical device for 3D surface measurement**
Combines the small depth of focus of an optical system with vertical scanning to provide topographical and color information from the variation of focus
- **True color**
- **Depth profile**
- **Surface area, projected area, roughness**
- **Volume**
- **20x objective has a field of view $500 \mu\text{m} \times 400 \mu\text{m}$, and extended field of view $10 \text{ mm} \times 8.2 \text{ mm}$**

3D Measurement of Corrosion

Morphology of crevice sites

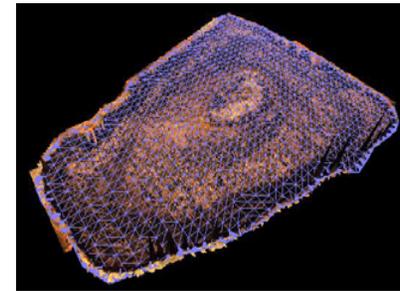
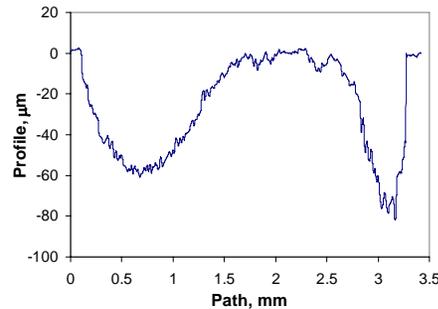
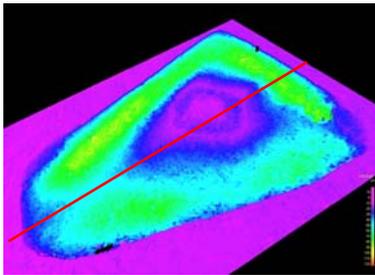


Overview of a contact foot



View from different tilt angles

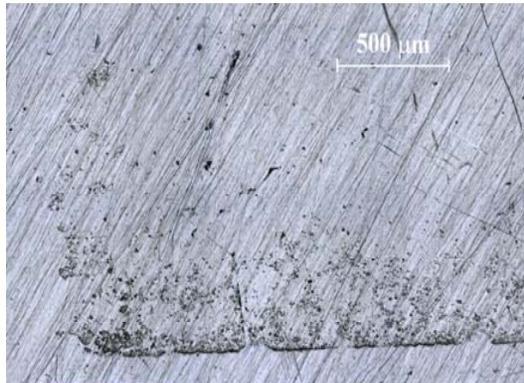
Depth profile and volume of corrosion site



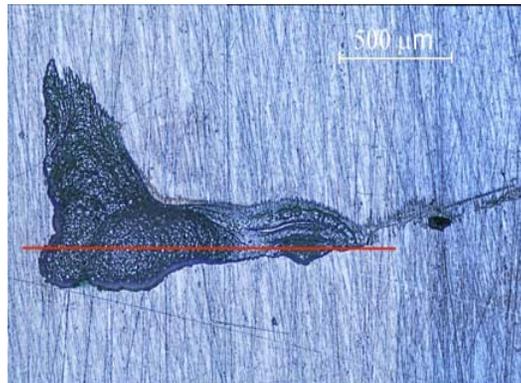
- Depth information represented by colors
- Depth profile determined along any line selected
- Maximum depth is 80 μm along line trace shown above
- Volume between the top plane and the metal surface is calculated
- Volume of the corroded metal is 0.21 mm^3

Effect of Crevice Former on 316 Stainless Steel (SS)

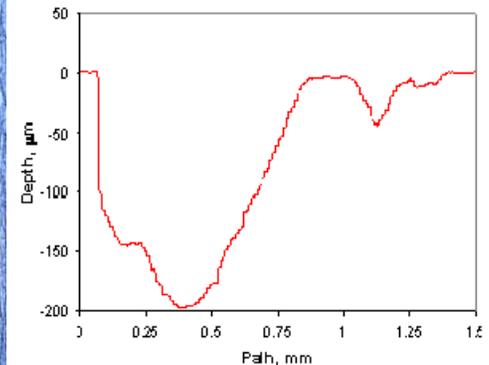
Ceramic vs. PTFE tape covered ceramic



Ceramic crevice former side



PTFE tape covered ceramic crevice former side



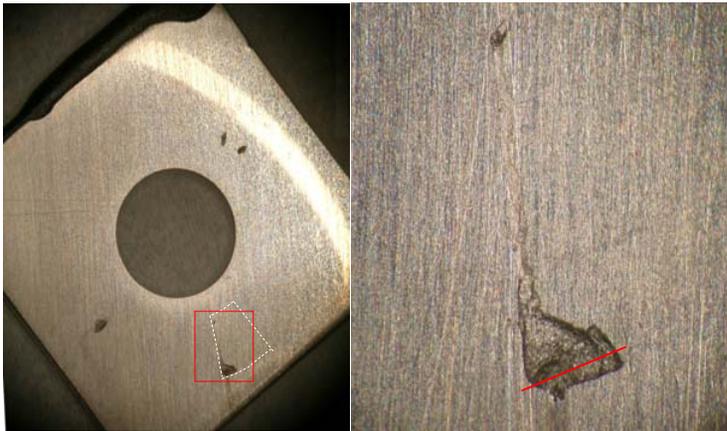
- 0.5M NaCl, room temperature, potentiostatic, $E = 0.1$ volt vs. SCE, specimens wetted by immersion for several minutes before assemblies were tightened
- On ceramic side, no distinguishable crevice corrosion found; damage was similar to that from mechanical assembly with no test exposure
- On PTFE tape covered ceramic side, corrosion is localized at the edge of the crevice former and deep; depth profile along line trace indicates up to 200 μm deep

Ceramic vs. Kel-F

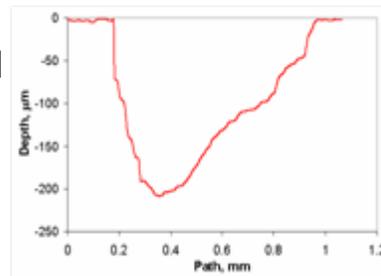
- On ceramic side, no distinguishable crevice corrosion found
- On Kel-F side, corrosion is localized and deep, similar to the result with PTFE tape covered ceramic crevice former

Effect of Crevice Former on SS316

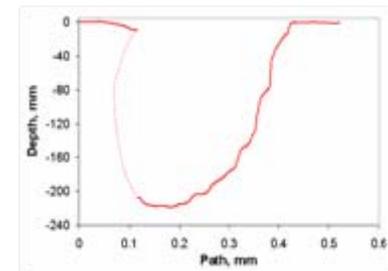
PTFE tape covered ceramic vs. Kel-F



PTFE tape covered
ceramic side



Kel-F side



- 0.5M NaCl, room temperature, potentiostatic, $E=0.1$ volt vs. SCE, wet specimens with test solution before assemblies tightened
- Corrosion on both PTFE tape covered ceramic side and Kel-F side, the corrosion is localized at the edge of crevice formers and deep, approximately $200 \mu\text{m}$

Effect of Crevice Former on SS316

Ceramic and PTFE tape covered ceramic crevice formers after cyclic potentiodynamic polarization tests



with ceramic crevice formers

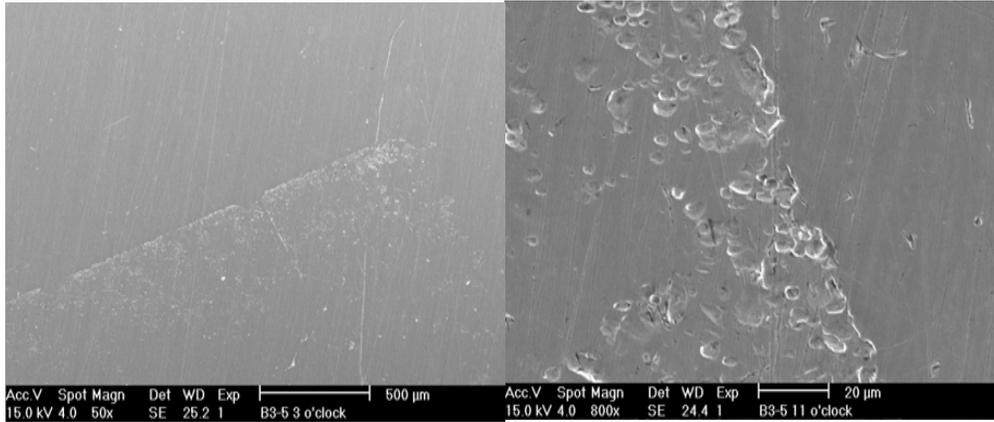


with PTFE tape covered ceramic crevice formers

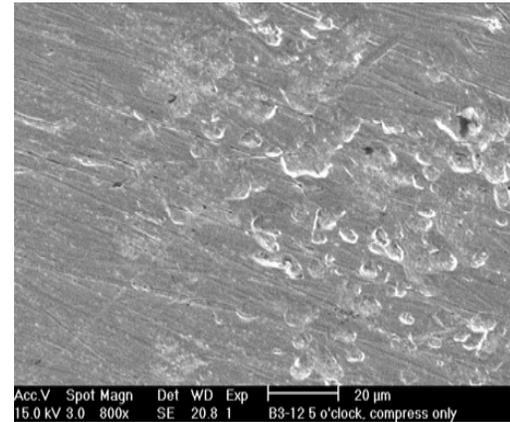
- 0.1M NaCl + 1M Na₂SO₄, 40°C, -0.18 to 1.24 volts vs. SCE, 0.167 mV/sec
- Crevice corrosion and pitting corrosion were found under the feet of crevice formers for both specimens
- For specimen with ceramic crevice formers, the pitting was over a larger area and not always along the edges
- For specimen with PTFE tape covered ceramic crevice formers, corrosion was localized along the edges

Effect of Crevice Former on SS316

Morphology of SS316 under ceramic crevice former



after crevice test (B3-5)



assembled only/no test (B3-12)

- B3-5: ceramic crevice former side, 0.5M NaCl, 22 to 26°C, potentiostatic, E= 0.1 volt vs. SCE for 2.5 hours
- B3-12: ceramic/ceramic crevice formers, under torque in air for two hours, no test exposure
- No distinguishable corrosion can be found on the metal under the ceramic crevice formers after test under the test condition
- Damage under the ceramic crevice former caused by compression of the ceramic crevice former against the metal

Effect of Crevice Former on SS316

Summary

- **PTFE tape covered ceramic/ceramic combination is the most aggressive crevice former combination**
- **Solid Kel-F crevice former also resulted in corrosion**
- **Ceramic crevice former without PTFE tape did not cause corrosion**

Effect of Crevice Former on C-22

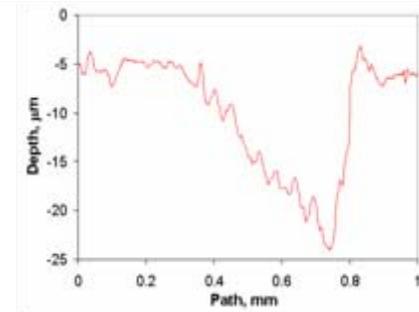
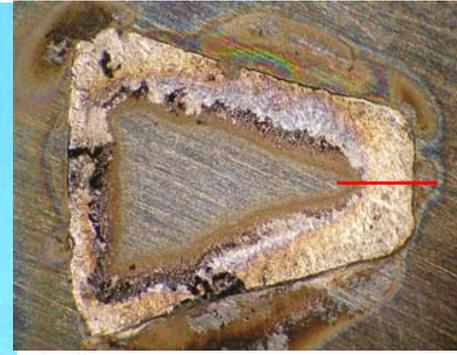
Ceramic vs. PTFE tape covered ceramic



Ceramic side



PTFE tape covered
ceramic side



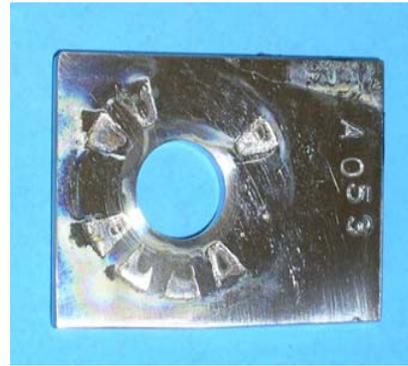
- 4M NaCl, 100°C, potentiostatic, anodic polarization to $E = -0.15$ volts vs. SCE, wet specimens with test solutions before tighten assemblies
- On ceramic side, no crevice corrosion was found
- On PTFE tape covered ceramic side, crevice corrosion to depth of about 25 μm after total flow of charge 10 coulombs to the whole specimen (67 hours)

Effect of Crevice Former on C-22

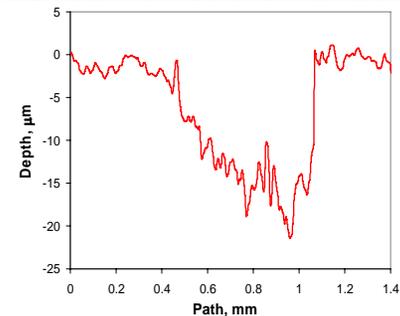
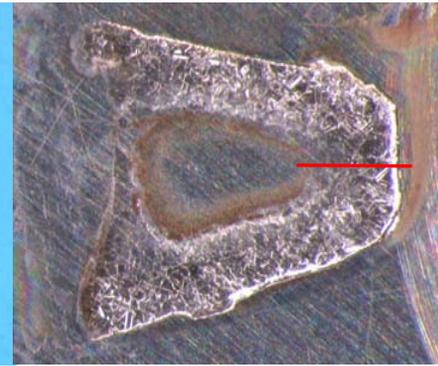
PTFE tape covered ceramic vs. Kel-F



Kel-F side



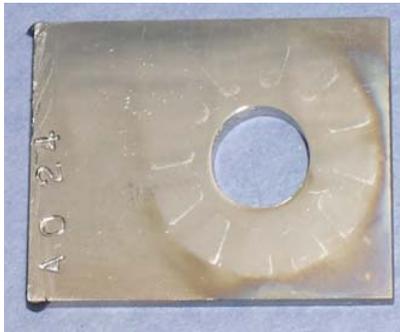
PTFE tape covered ceramic side



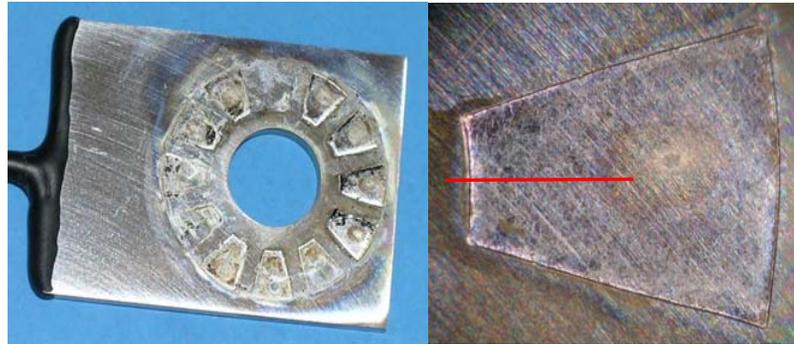
- 4M NaCl, 100°C, potentiostatic, anodic polarization to $E = -0.15$ volts vs. SCE, wet specimens with test solutions before assemblies tightened
- On Kel-F side, no corrosion was found
- On PTFE tape covered ceramic side, crevice corrosion to depth of about 20 μm after total flow of charge 10C to the whole specimen (34.5 hours)

Effect of Crevice Former on C-22

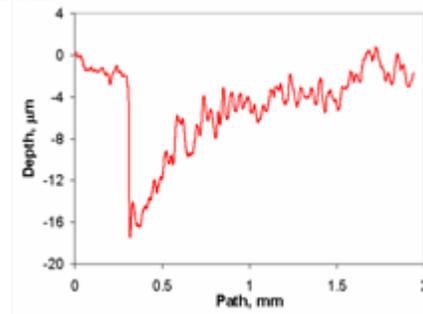
Ceramic vs. Kel-F



Ceramic side



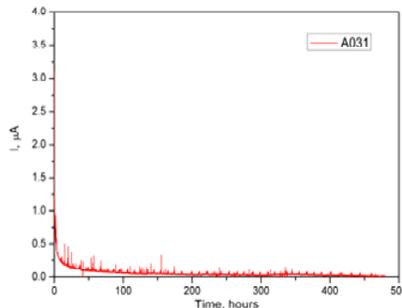
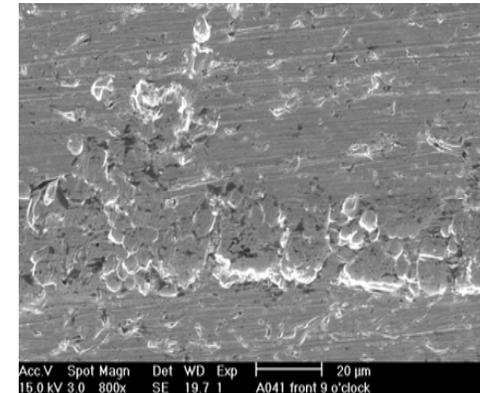
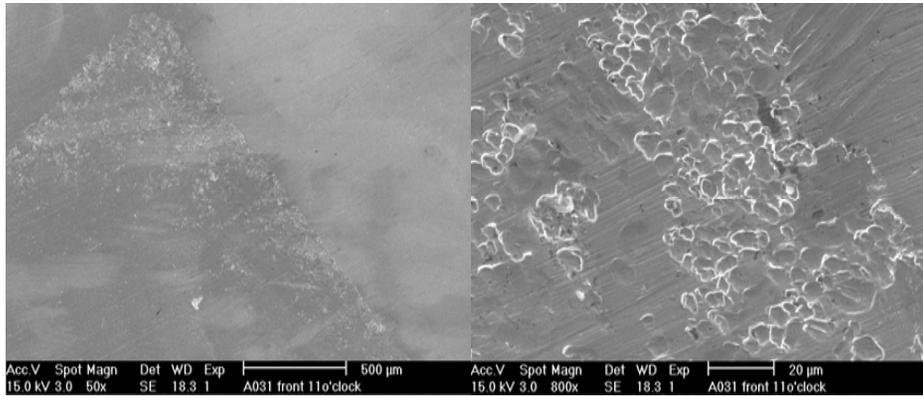
Kel-F side



- 4M NaCl, 100°C, potentiostatic, anodic polarization to $E = -0.15$ volts vs. SCE, wet specimens with test solutions before assemblies tightened
- On ceramic side, no crevice corrosion
- On Kel-F side, crevice corrosion on one of 5 specimens

Effect of Crevice Former on C-22

Morphology of C-22 under ceramic crevice former



After 20-day test, no indication of crevice corrosion; final current less than $0.04 \mu\text{A}$ (A031)

Assembled in air to full torque with no corrosion exposure

- 4M NaCl, 100°C , potentiostatic, anodic polarization to $E = -0.15$ volts vs. SCE, wet specimens with test solution before assemblies tightened
- Current decreased rapidly and remained low, i.e. no indication of crevice corrosion
- No distinguishable corrosion observed under ceramic crevice former; mechanical damage to surface

Effect of Crevice Former on C-22

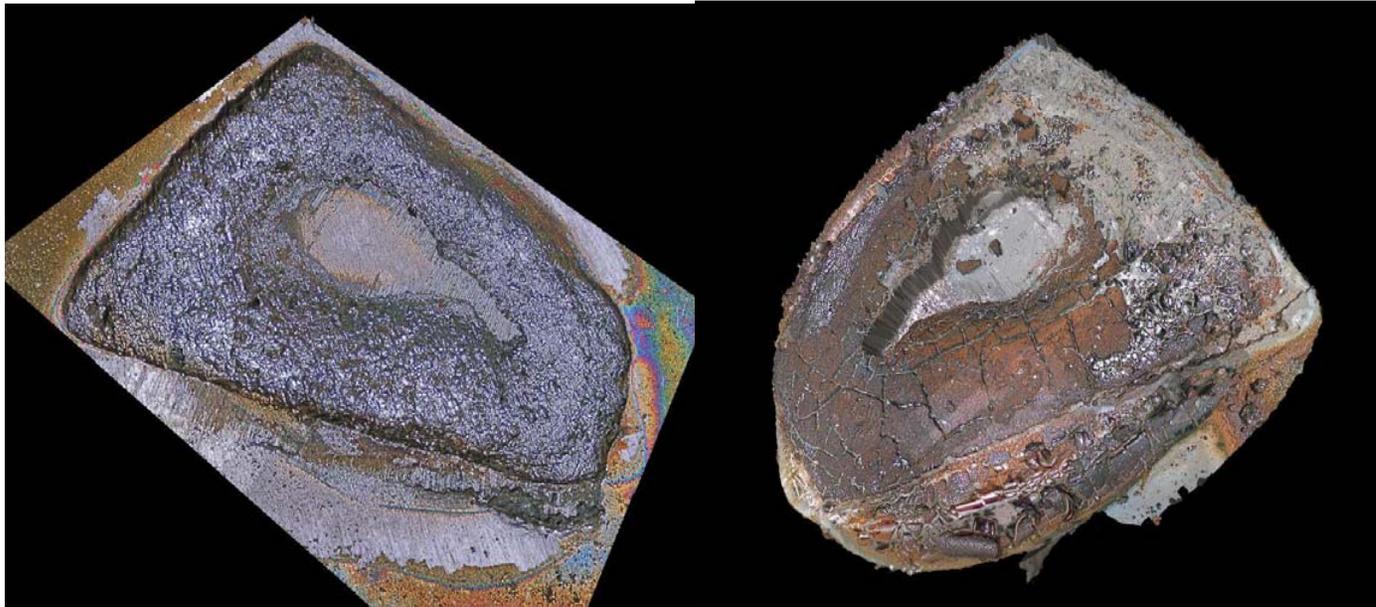
Summary

Crevice formers		Observation	Comments
C (0/5)	TC (5/5)	PTFE tape covered ceramic (TC) side always corroded. No crevice corrosion on ceramic side (C). The remaining torque after test is about 55 in-lb compared to initial 70 in-lb.	Most aggressive crevice formers combination.
C (0/5)	K(1/5)	One of five samples corroded on Kel-F side (K), the remaining samples no corrosion beneath the crevice formers, i.e K or C. The remaining torque after test is less than 5 in-lb.	
TC (3/3)	K (0/3)	TC side always corroded, no crevice corrosion on Kel-F side. The remaining torque after test is less than 5 in-lb.	
C (0/1)	T (0/1)	Tested for 35 days. Initial torque was 2.5 in-lb and tested for 15 days. Re-torqued to 14 in-lb at RT and tested for another 10 days. Followed by another re-torque of 25 in-lb at RT and tested for another 10 days. No corrosion was found on either sides. The remaining torque is less than 5 in-lb.	PTFE deforms/relaxes under high temperature (100 °C), not as aggressive crevices as with ceramic support
TC (0/1)	T (0/1)	Same re-torque schedule as the C/T crevice formers combination.	Same as the previous one
K (0/1)	T (0/1)	Same re-torque schedule as the C/T crevice formers combination.	Same as the previous one
C (0/1)	C (0/1)	Tested for 20 days, no corrosion.	For verification

TC = PTFE tape covered ceramic; C = ceramic; K= solid Kel-F; T = solid PTFE
Numbers in parentheses indicate number of specimens corroded vs number of specimens tested under each crevice former combination

Effect of Crevice Former

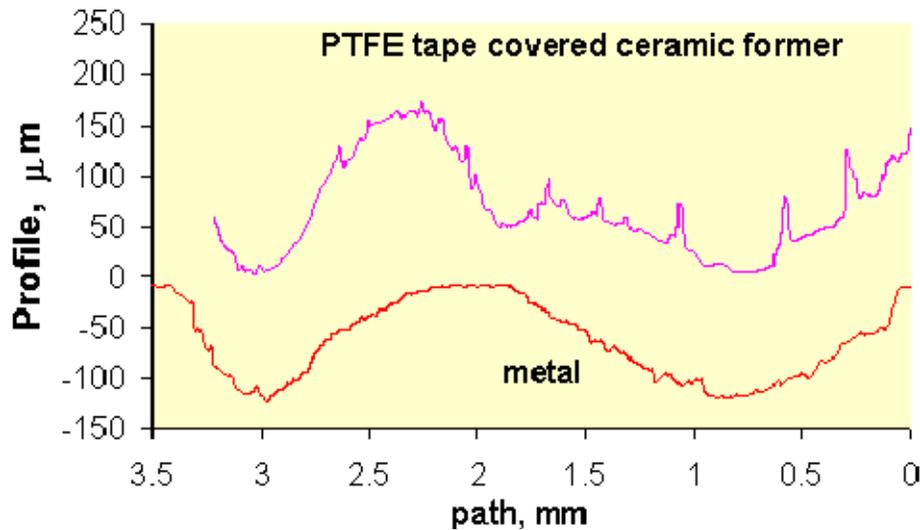
Comparison of morphology of crevice corrosion damage and crevice former surface after the test



- 4M NaCl, 100°C, potentiostatic, anodic polarization to $E = -0.15$ volts vs. SCE, wet specimens with test solution before assemblies tightened
- Crevice former foot size: 3.2 mm long by 2.6 mm wide (maximum width)
- Ceramic supported PTFE tape conforms to receding crevice corrosion surface during test
- A tight crevice is maintained and severe conditions persist

Effect of Crevice Former

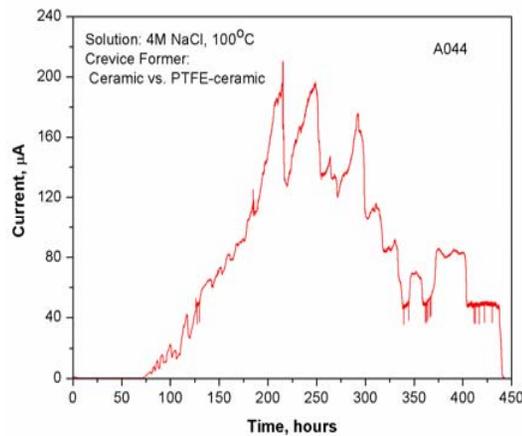
Comparison of morphology of crevice corrosion damage and crevice former surface after the test



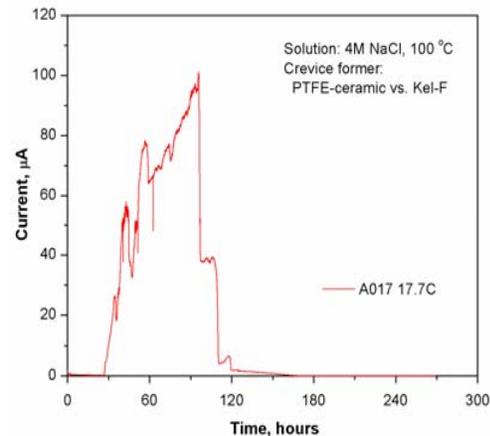
- 4M NaCl, 100°C, potentiostatic, anodic polarization to $E = -0.15$ volts vs. SCE, wet specimens with test solution before assemblies tightened
- Ceramic support maintains high load and PTFE conforms to crevice profile
- Maintaining a tight crevice even with a receding surface during the test

Effect of Crevice Former

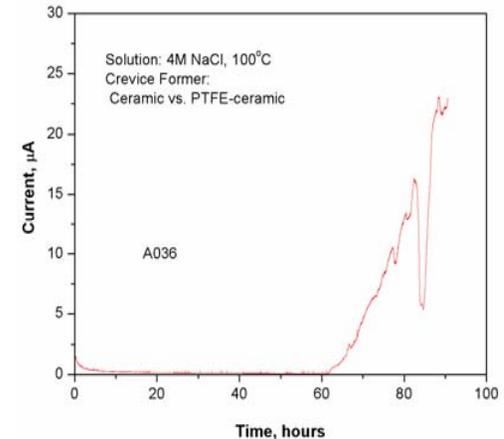
Initiation and arrest of crevice corrosion indicated by specimen current throughout the test



of feet corroded: 9



of feet corroded: 4

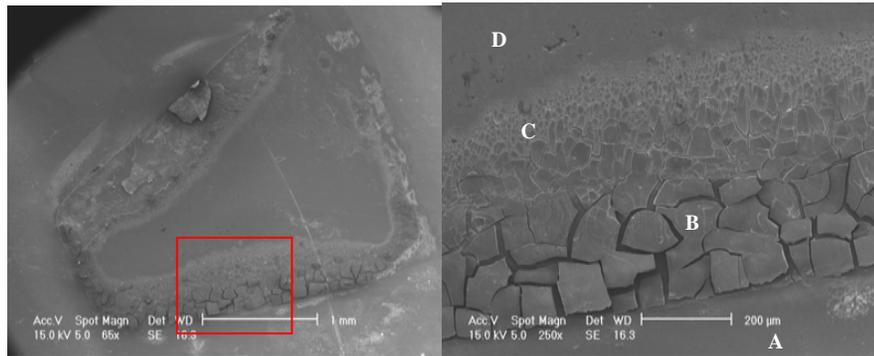


of feet corroded: 1

- Specimens become re-passivated after certain period of test
- Multiple initiation and arrest of crevice corrosion events indicated during the test
- Initiation and arrest events are loosely related to the number of feet corroded; future work will pursue more direct correlation
- Initiation and arrest of corrosion also observed on current response of a single crevice foot

C-22 Corrosion Deposits Within Crevice

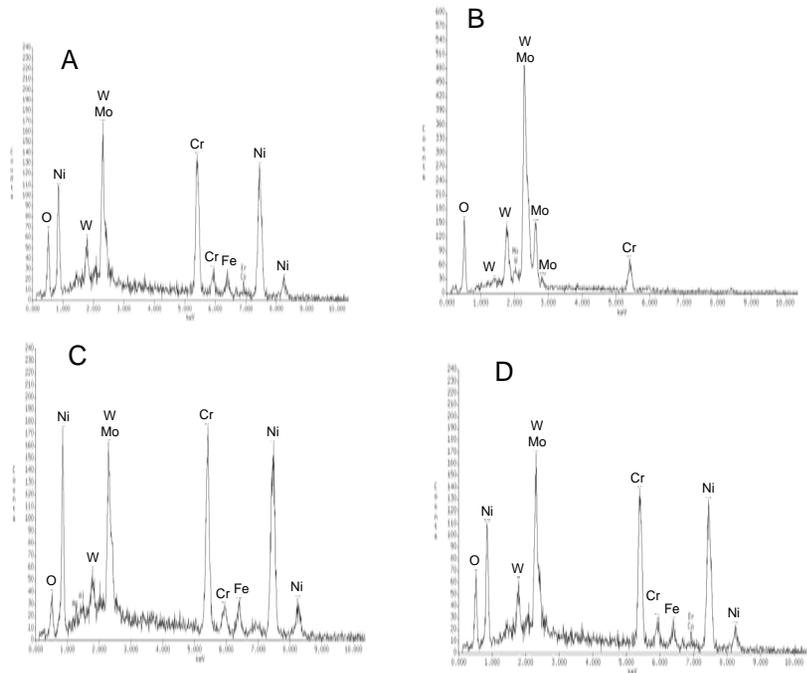
EDS analysis of C-22 corrosion product



	Cr (at%)	Mo (at%)	W (at%)	Fe (at%)	O (at%)	Ni (at%)
A	23.25	7.8	1.04	5.19	8.45	54.28
B	7.86	20.82	3.33	0	67.23	0.76
C	21.75	7.42	0.79	4.97	12.70	52.37
D	19.86	7.66	1.27	4.15	22.69	44.37

C-22 base composition (at%)

Cr	Mo	W	Fe	O	Ni	Co
26.05	8.34	1	3.3	n/a	58.7	2.6



- 4M NaCl, 100°C, potentiostatic, anodic polarization to $E = -0.15$ volts vs. SCE, wet specimens with test solution before assemblies tightened
- Loose, black corrosion products found under the crevice former after test
- Corrosion products are depleted in Ni, Cr and Fe, and enriched in W and Mo; it also contains high amount of oxygen

Summary

- **To determine the effect of the crevice former on the localized corrosion damage propagation**
 - **Determination of the effect of the crevice former on the localized corrosion damage propagation**
 - **Focus is on post initiation stage, crevice propagation and arrest processes**
 - **Determine the evolution of damage – severity, shape, location/distribution, damage profile**
- **Multiple techniques to examine crevice corrosion**
 - **Quantitative 3-D analysis of surface profiles and volume characterizes the corrosion damage**
 - **Analysis of corrosion products/deposits within the crevice provides insight into processes**
 - **Model of crevice corrosion damage evolution, i.e. the profile of crevice corrosion with time, is a work in progress**
- **Work is collaborative with other projects within the DOE/OST&I multi-university cooperative**

Conclusions

- **Ceramic/PTFE tape covered ceramic was most severe crevice former**
- **Solid polymer crevice formers (PTFE or Kel-F) are less severe**
- **No distinguishable crevice corrosion with ceramic crevice formed in direct contact with the metal**
 - **Controlled crevice corrosion test under highly aggressive, accelerated condition**
 - **Polarization current throughout the test indicates magnitude of corrosion and initiation/arrest/reinitiation events**
 - **Initiation and arrest events are loosely related to the number of feet corroded; future work will pursue more direct correlation**