Status of Ongoing NRC Research: Stress Corrosion Cracking (SCC) of Spent Nuclear Fuel Dry Storage Canisters

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RES/DE/CMB

RIRP Public Meeting
December 18, 2012

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United States Nuclear Regulatory Commission
Protecting People and the Environment

Participants

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- NMSS: T. Ahn, B. Einziger, S. DePaula, M. Gordon, J. Rubenstone
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Outline

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- Overview and schedule
- Status of current research activities
- Summary and key points

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Overview and Schedule

Background

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- NUREG/CR-7030 and various Japanese test results indicated that austenitic stainless steel is susceptible to SCC when exposed to chloride salts in certain conditions of salt quantity, temperature, humidity, and stress level.
- The significance of various parameters was difficult to quantify given differing test methodologies and interpretation of results.
- Additional work by NRC was needed to better understand conditions of susceptibility and determine whether further action is warranted.

Scope of Current Research Program

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- Deliquescence testing of sea salt and major constituents
- SCC testing at absolute humidity (AH) less than 30 g/m^3
- SCC testing at elevated temperatures
- SCC testing at high relative humidity (RH) conditions
- SCC testing at different specimen stress/strain levels
- SCC testing with non-chloride salt particulates

Schedule

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- Program began in October 2011
- Experimental work will end in January 2013
- NUREG/CR report expected to be published by July 2013
- Previous or planned presentations/papers:
  - NACE Corrosion 2012, Salt Lake City, UT, March 11-15, 2012
  - Waste Management Symposia, Phoenix, AZ, February 24-28, 2013
  - NACE Corrosion 2013, Orlando, FL, March 17-21, 2013
  - Environmental Degradation of Materials in Nuclear Power Systems, Asheville, NC, August 11-15, 2013

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Status of Current Research Activities

Deliquescence Testing

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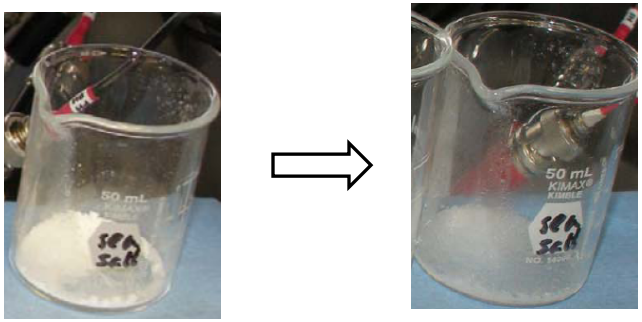
- Background: Deliquescence of dry salt on canisters may introduce moisture to support SCC
- Test objective: Identify the deliquescence RH (DRH) for sea salt at various temperatures
- Test methodologies:

Salts in beakers

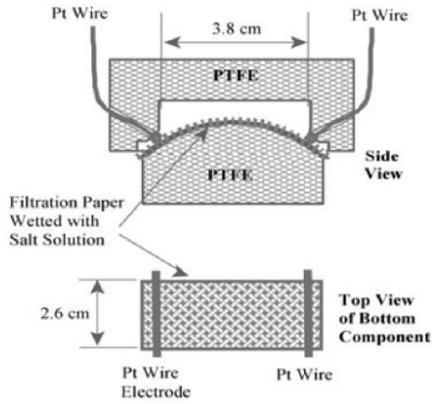


Low RH: Salt is dry

High RH: Salt has deliqueced

Observe beakers for moisture absorption

Conductivity cell impedance measurement



Record impedance drop as function of RH

Deliquescence Testing

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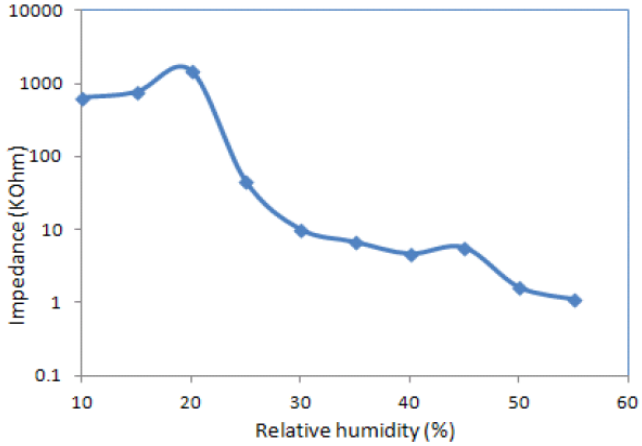
- Beaker test results: DRH based on observation of moisture absorption

		Temperature (°C)		
		45	60	80
Species	Sea salt (ASTM D1141-98)	43%	35%	25%
	$CaCl_2$	28%	20%	20%
	$MgCl_2$	37%	35%	25%
	NaCl	None up to 59%	None up to 60%	None up to 59%
	Na_2SO_4	None up to 59%	None up to 60%	None up to 59%

- Conductivity cell results: DRH based on impedance drop (sea salt only)
  - 43% at 45°C; 35% at 60°C; 25% at 80°C



Plot at 60°C

Notable observations:
- DRH for sea salt is close to that of $MgCl_2$
- DRH decreases somewhat with increasing temperature

SCC Testing at AH Less than 30 g/m^3

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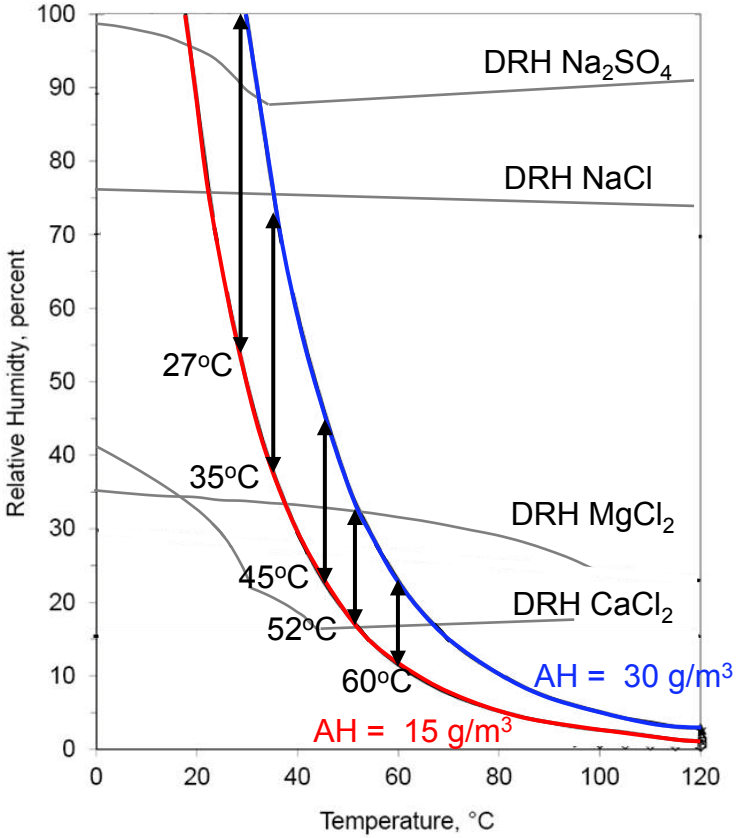
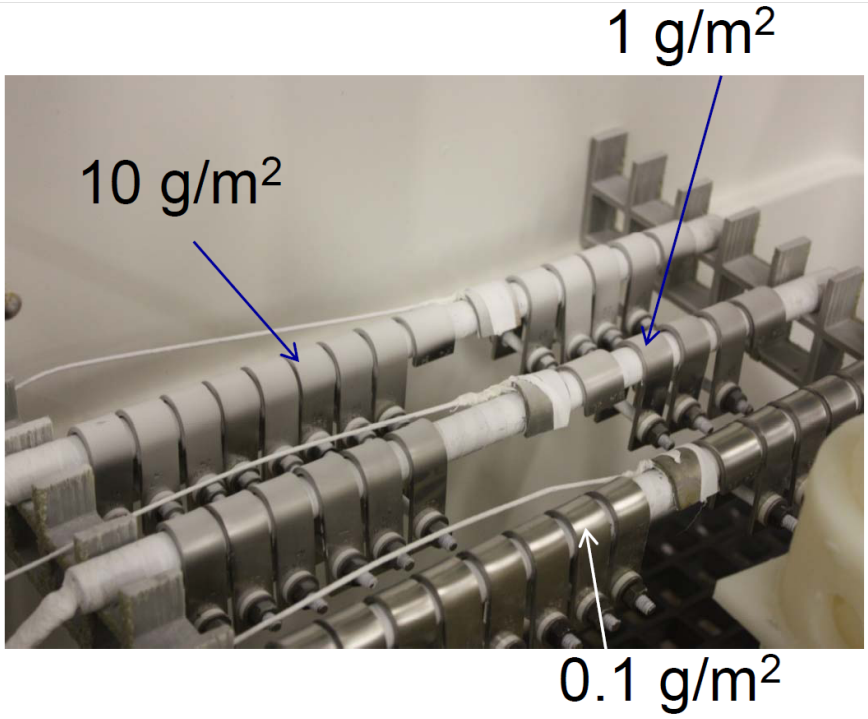
- Background: Test conditions where SCC was observed in NUREG/CR-7030 testing were at AH near 60 g/m^3. Limit of AH in natural conditions may be closer to 30 g/m^3. Also, quantity of salt deposition on specimens was not well-controlled.
- Test objectives:
  - Identify whether SCC can initiate at AH less than 30 g/m^3
  - Investigate effects of surface salt concentration and material condition
- Test methodology:
  - Deposited 0.1, 1, or 10 g/m^2 of sea salt on ASTM G30 U-bend specimens
  - Specimens were Type 304 in as-received, sensitized, or as-welded with Type 308
  - Specimens were exposed to cyclic AH between about 15 and 30 g/m^3 at various temperatures.

Test Setup



Test temperatures (arrows indicating span of humidity cycle) and lines showing calculated DRH for sea salt constituents



Specimens in test chamber

Test Results

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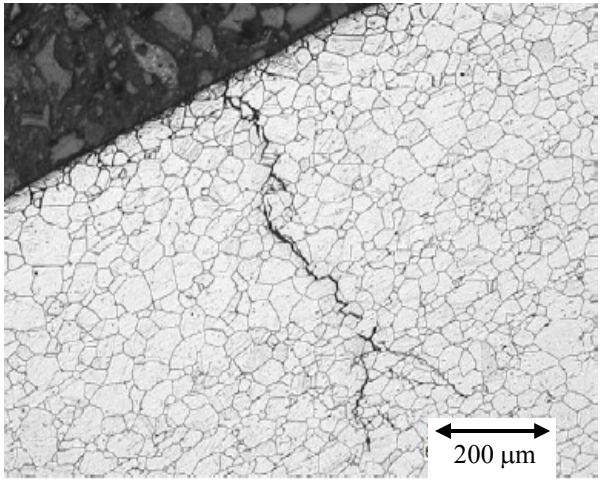
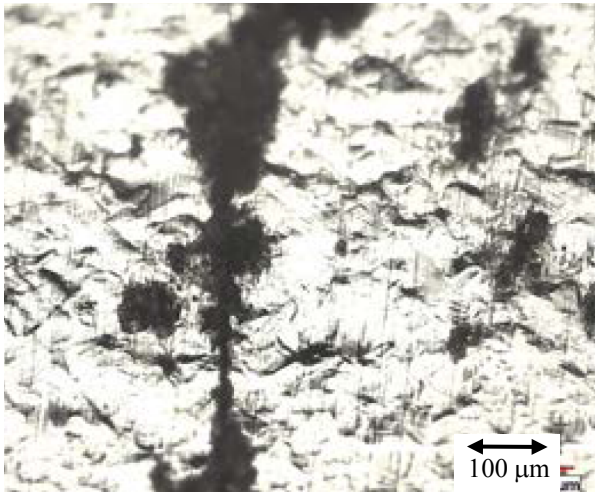
- SCC initiation observed at 35 and 45°C at salt concentration as low as 0.1 g/m^2.
- SCC initiation observed at 52°C at 10 g/m^2. Tests ongoing at 1 g/m^2.
- Tests at 60°C at 10 g/m^2 are still ongoing.
- Sensitized condition seems most susceptible to SCC.

Specimens at 10 g/m^2 (top), 1 g/m^2 (middle), and 0.1 g/m^2 (bottom)



Cross section of sensitized, 0.1 g/m^2 specimen at 45°C after 4 months



Top view of sensitized, 10 g/m^2 specimen at 52°C after 1 month

SCC Testing at Elevated Temperatures

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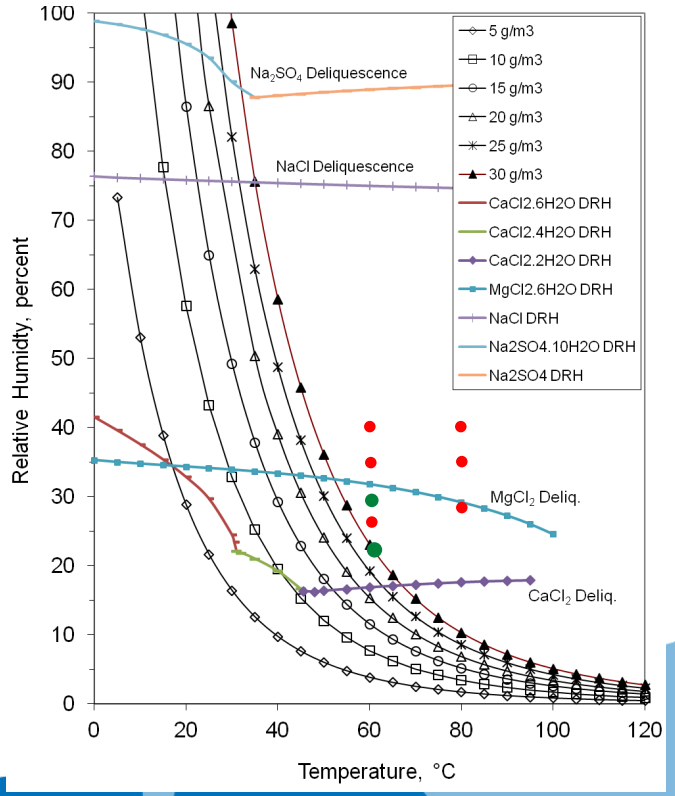
- Background: Japanese have reported SCC initiation at temperature up to 80°C at 15% RH. NUREG/CR-7030 testing showed cracking only at 43°C, not at 85 or 120°C.
- Test objective: Identify whether SCC can initiate at temperatures in the range of 60 to 80°C
- Test methodology:
  - Deposited 10 g/m^2 of sea salt on U-bend specimens
  - Exposed specimens to static RH at 60 and 80°C
  - Started tests at high RH and progressed lower as SCC initiation was observed



- SCC observed
- No SCC observed

Test Results

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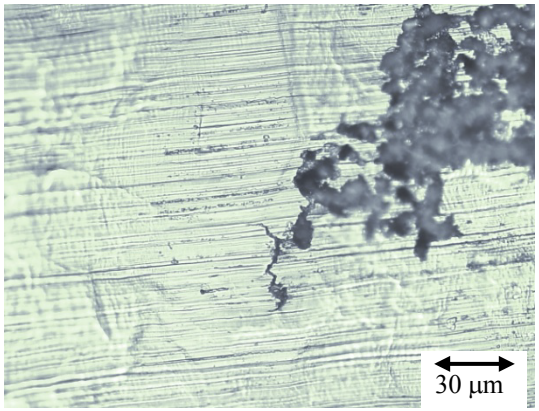
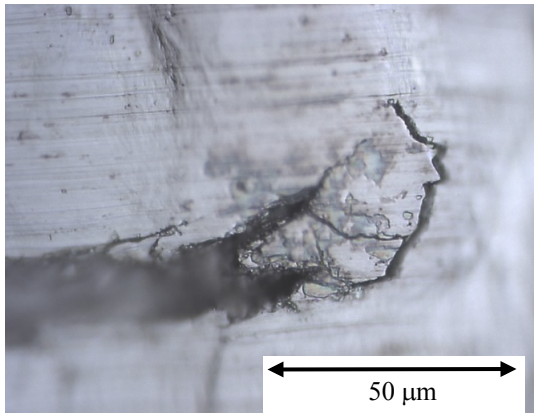
- SCC initiation was observed at 60°C as low as 25% RH and at 80°C as low as 28% RH.
- AH for tests was above 30 g/m^3 but results indicate that SCC can initiate by salt deliquescence if RH is high enough.

Specimens after 8 weeks at 80°C and 28% RH



Micrograph of specimen at 80°C and 28% RH



Micrograph of specimen at 60°C and 25% RH

SCC Testing at High RH

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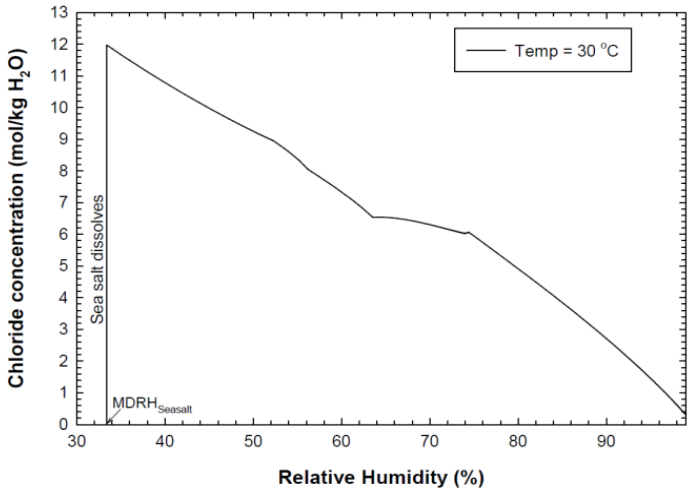
- Background: Equilibrium chloride concentration in solution decreases with increasing RH. Dilution of chlorides at high RH could inhibit SCC.
- Test objective: Determine whether SCC can initiate in conditions of high RH
- Test methodologies:
  - Immersed U-bend specimens in prepared saturated solutions for 30°C and 90% RH
  - Deposited 10 g/m^2 of sea salt on U-bend specimens for exposure at 30°C and 90% RH



Calculated chloride concentration in saturated sea salt solution as function of RH at 30°C

U-bend specimens immersed in solution

Test Results

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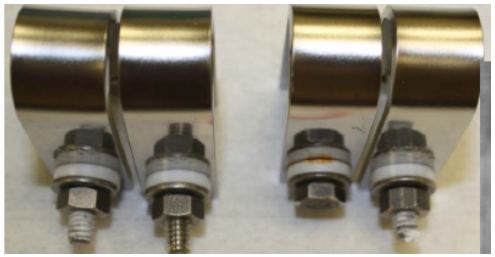
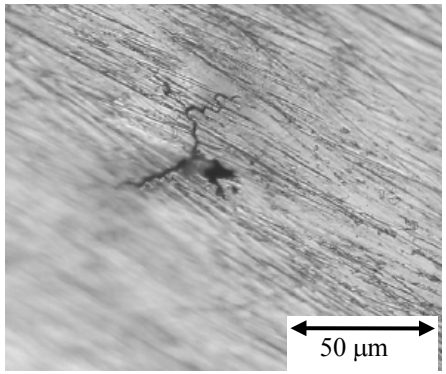
- For specimens with deposited salt, salt quickly deliquesced and ran off sides of specimens.
- For immersed specimens, pitting and SCC were observed within 5 weeks.

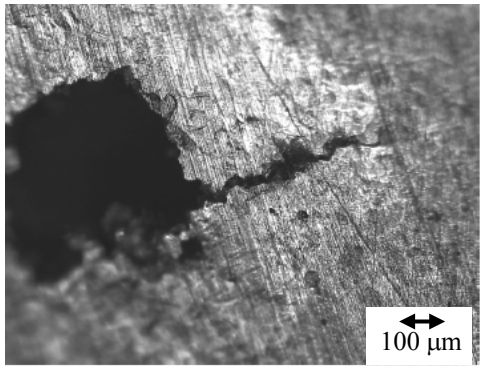
Chloride and Salt Concentrations in Saturated Solutions at 30°C and 90% RH			
		Chloride Concentration (mol/kg H_2O)	Salt Concentration (g/kg H_2O)
Solution	Sea salt	2.71	203
	NaCl	2.79	163
	MgCl2	3.01	306
	CaCl2	3.16	232



Specimens immersed in sea salt after 5 weeks, as received (L); sensitized (R)



Cracking on surface of specimen immersed in sea salt



Cracking on surface of specimen immersed in $MgCl_2$

C-Ring SCC Testing

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- Background: U-bend specimens represent a highly strained state, 13-14% at the apex. These may not be representative of canister conditions.
- Test objective: Use C-ring testing to control applied stress/strain and investigate effect on SCC initiation.
- Test methodologies:
  - Specimens fabricated following ASTM G38-01 and deposited with 1 or 10 g/m^2 of salt
  - Specimens were strained to yield stress (~0.3% plastic) or 1.5% plastic strain, as measured by strain gage.
  - Specimens were exposed at conditions of 35^{o}C and 72% RH, 45^{o}C and 44% RH, and 52^{o}C and 32% RH.

Test Results

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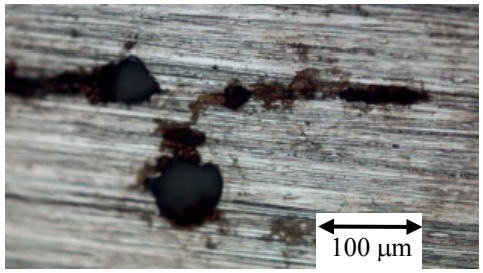
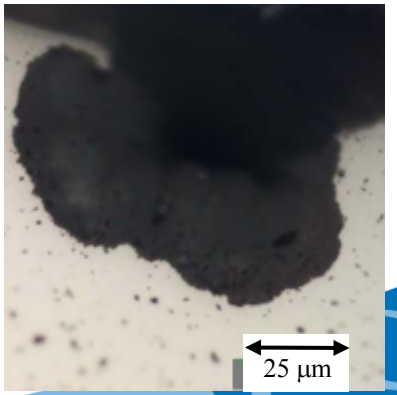
- Pitting and SCC initiation was observed for the specimen with 10 g/m^2 of salt at yield stress at 35^{o}C
- Pitting is visible on other specimens but SCC initiation has not been confirmed. Tests are ongoing.

35^{o}C, yield stress specimens

45^{o}C, yield stress specimens





SCC Testing with Non-Chloride Particulates

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- Background: Canisters may be exposed to non-chloride airborne particulates in regions with industrial, commercial, or agricultural activities. Effects of these particulates on SCC susceptibility is not well understood.
- Test objective: Assess SCC susceptibility for stainless exposed to common non-chloride airborne particulates.
- Test approach:
  - Identify common airborne particulates by reviewing atmospheric monitoring data
  - Perform deliquescence testing on particulates
  - Perform SCC testing using U-bend specimens deposited with the particulates. Tests at 45°C and 44%RH for 6 weeks, then 35°C and 72% RH for one month.
  - Perform SCC testing with the particulates and the addition of NaCl
- Species tested:
  - Fly ash, Class F
  - $(NH_4)_2SO_4$
  - NH_4NO_3
  - NH_4HSO_4
  - $(NH_4)_2SO_4 + NH_4NO_3$ mixtures (0.5, 1, 3 SO_4^{2-} to NO_3^- mole ratio)
  - NH_4NO_3 + NaCl mixture (3, 6 NO_3^- to Cl^- mole ratio)

Test Results

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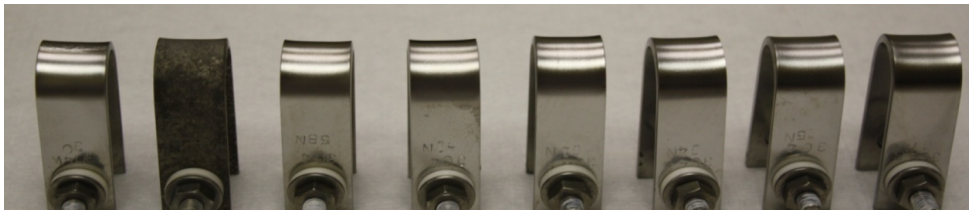
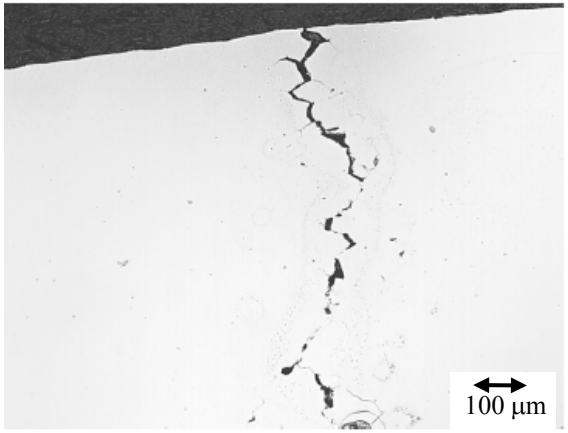
- For specimens with no chloride salt present, no SCC initiation was observed. Only specimen exposed to NH_4HSO_4 (second from left, below) showed general corrosion.



- Severe cracking observed on specimens exposed to NH_4NO_3 + NaCl



Summary and Key Points

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- DRH for sea salt appears to be close to that of $MgCl_2$ and less than NaCl.
- SCC can initiate at AH less than 30 g/m^3 at surface salt concentration as low as 0.1 g/m^2.
- SCC can initiate at temperatures in the range of 60 to 80°C if the RH is high enough.
- Equilibrium chloride concentration in solution decreases at high RH but SCC may initiate if the chloride is present.
- SCC initiation was observed on specimens tested at yield stress.
- Non-chloride particulates did not cause SCC initiation. Initiation was observed for NH_4NO_3 + NaCl.