

10/23/12

**CNWRA**

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# **SCC Project Update October 25, 2012**

E121

# Project Objectives and Tasks

1. Examine the effects of surface salt concentration and realistic environmental conditions on SCC behavior of austenitic stainless steel alloys used for dry storage canisters
2. Scoping study of non-coastal environment on SCC susceptibility

## Coastal Environment

- Task 1: Determine the Minimum Chloride Concentration for Onset of Stress Corrosion Cracking
- Task 3: Elevated Temperature Effects
- Task 4: Evaluate the Effects of Chloride Dilution on the Initiation Threshold of Stress Corrosion Cracking
- Task 5: Evaluate Effects of Stress on Crack Initiation

## Non-coastal Environment

- Task 2: Evaluate Effects of Dust and Other Atmospheric Deposits on Stress Corrosion Cracking



# Task 1—Determine the Minimum Chloride Concentration for Onset of Stress Corrosion Cracking (SCC)

Environmental Cycling Test Matrix

Temperature, °C	Salt Concentration, g/m <sup>2</sup>	Metallurgical Type	Test Duration, month
27 (4/13/12 started)	10	As-Received, Sensitized	1, TBD*
35 (12/14/11 started)	10	As-Received, Sensitized, Welded	1, 4
	1.0	As-Received, Sensitized	1, 4
	0.5 (4/13/12 started)	As-Received, Sensitized	1, TBD
	0.1	As-Received, Sensitized, Welded	1, 4, TBD
45 (12/14/11 started)	10	As-Received, Sensitized, Welded	1, 4
	1.0	As-Received, Sensitized	1, 4
	0.1	As-Received, Sensitized, Welded	1, 4, TBD
52 (4/13/12 started)	10	As-Received, Sensitized	1, 2.7
	1	As-Received, Sensitized	TBD
60 (5/30/12 started)	10	As-Received, Sensitized	TBD

\*TBD=To Be Determined

# Summary of Completed Task 1 Tests

- SCC occurred at 35, 45, 52 °C
- The minimum simulated sea salt concentration for onset of SCC was 0.1 g/m<sup>2</sup>
- Lower temperature specimens showed greater SCC susceptibility
- SCC susceptibility increased with surface chloride concentration
- Sensitized specimens were more susceptible than as-received



# Path Forward for Task 1

- All the remaining tests are moving forward smoothly
  - 0.1 g/m<sup>2</sup> salt specimens at 35 and 45 °C
  - 0.5 g/m<sup>2</sup> salt specimens at 35°C
  - 1 g/m<sup>2</sup> salt specimens at 52 °C
  - 10 g/m<sup>2</sup> salt specimens at 27 and 60 °C
- On 12/14/12, the longest test will be 1 year, should all the specimens be pulled out on that day?

## Task 2: SCC Tests for Non-Coastal Salts

Completed SCC Tests at 45 °C-44% RH and 35 °C-72% RH without addition of chloride

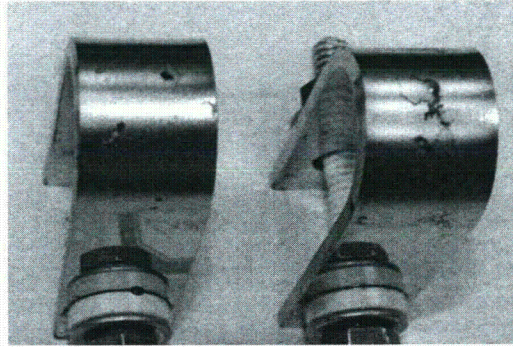
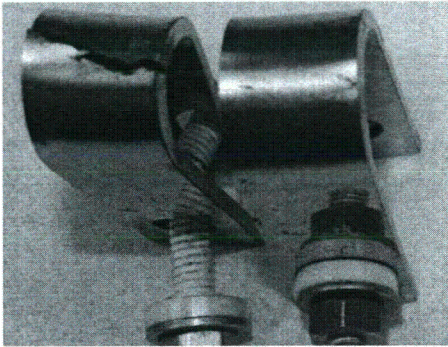
Chemicals	Specimens	Observation
NH <sub>4</sub> HSO <sub>4</sub> , NH <sub>4</sub> NO <sub>3</sub> , (NH <sub>4</sub> ) <sub>2</sub> SO <sub>4</sub> + NH <sub>4</sub> NO <sub>3</sub> (SO <sub>4</sub> <sup>2-</sup> /NO <sub>3</sub> <sup>-</sup> = 0.5, 1, and 3), Fly ash	as-received, sensitized	No cracking observed
NH <sub>4</sub> HSO <sub>4</sub> , NH <sub>4</sub> NO <sub>3</sub> , Fly ash	welded	

SCC Tests at 45 °C and 44% RH with addition of chloride

Mole ratio of NH <sub>4</sub> NO <sub>3</sub> to NaCl	Specimens	Test duration, month	Observation
3	3 as-received, 3 sensitized	1, TBD	Severe cracking observed on all sensitized specimens. As-received specimens are subject to longer exposure for further examination <sub>6</sub>
6	3 as-received 3 sensitized		



Sensitized specimens deposited with  $3\text{NH}_4\text{NO}_3\cdot\text{NaCl}$  and  $6\text{NH}_4\text{NO}_3\cdot\text{NaCl}$  exposed at 45 °C-44% RH for 8 weeks



## Path Forward for Task 2

- Pull the remaining 4 as-received specimens in December to examine.

# Task 3: Elevated Temperature Testing for Coastal Salts

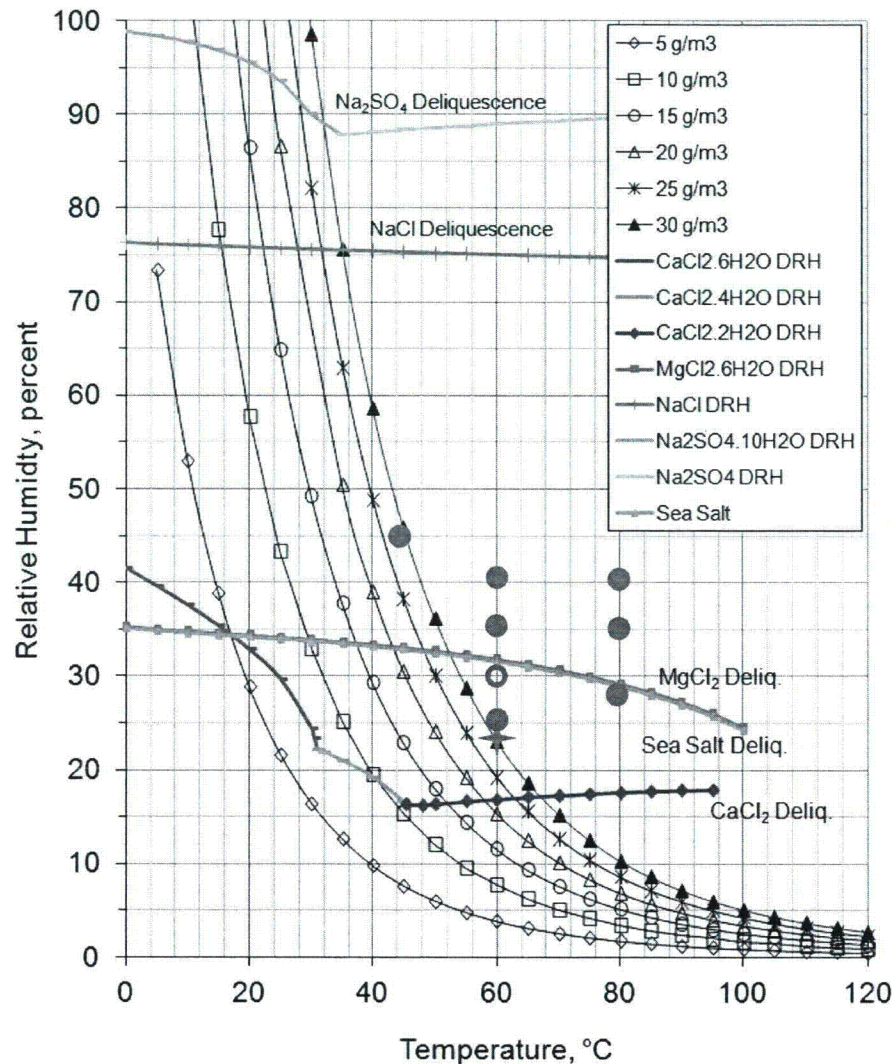
- All tests are terminated

Static Environmental Test Matrix

Temperature, °C	Relative Humidity, %	Number and Metallurgical Type
45	44	6 as-received, 6 sensitized
60	22	3 as-received, 3 sensitized
	25	3 as-received, 3 sensitized
	30	3 as-received, 3 sensitized
	35	3 as-received, 3 sensitized
	40	3 as-received, 3 sensitized
80	28	3 as-received, 3 sensitized
	35	3 as-received, 3 sensitized
	40	3 as-received, 3 sensitized



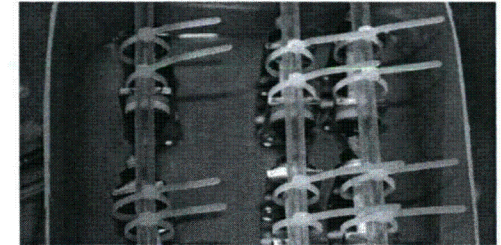
# Summary and Path Forward for Task 3



- Terminated and cracking observed
- ★ Terminated, but cracking not observed
- Terminated. Specimens are under examination
- Examine the cross section of the last two specimens exposed to 60 °C-30% RH as cracking was not observed from surface

# Task 4: Evaluate the Effects of Chloride Dilution on the Initiation Threshold of SCC

- Tests started at 30 °C-90% RH on 8/27/12 with U-bend specimens hung up in solutions with crown facing downwards and immersed in solution.

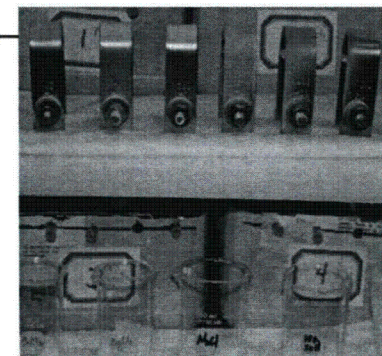


**Calculated chloride concentration and Tests at 30 °C and 90 %RH**

Salt	Cl <sup>-</sup> Concentration (mol/kg H <sub>2</sub> O)	Measured pH (~20 °C)	Number and Metallurgical Type	Test Duration, month
NaCl	2.79	6.38	3 as-received, 3 sensitized	1, TBD*
CaCl <sub>2</sub>	3.16	6.21	3 as-received, 3 sensitized	1, TBD
MgCl <sub>2</sub>	3.01	5.09	3 as-received, 3 sensitized	1, TBD
Sea Salt	2.71	7.93	3 as-received, 3 sensitized	1, TBD

\*TBD=To Be Determined

- Three as-received and three sensitized specimens deposited with 10 g/m<sup>2</sup> simulated sea salt and small beakers with small amount of each salt were also included in the chamber





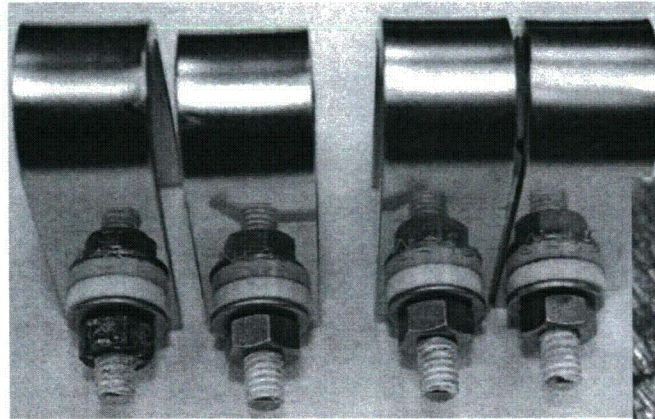
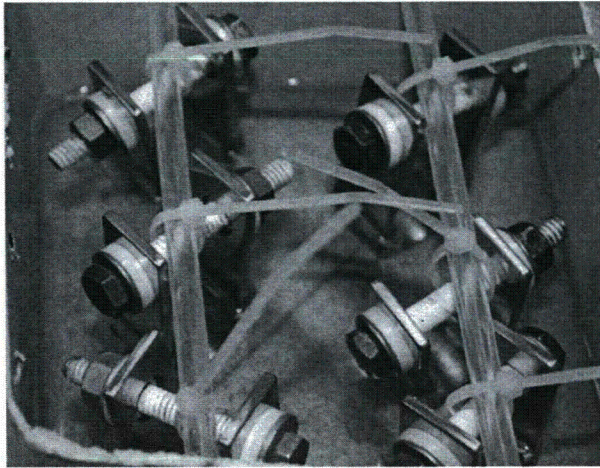
## Task 4: 1-month SCC Test Summary

- Two out of three as-received and sensitized specimens were pulled after exposure for 5 weeks
- NaCl, MgCl<sub>2</sub>, and CaCl<sub>2</sub> solutions were yellowish from the formation of the stainless steel corrosion products. Sea salt solution appeared clear (shown in next 4 slides)
- Pitting corrosion was observed from the surface of all as-received and sensitized specimens exposed to NaCl, MgCl<sub>2</sub>, and CaCl<sub>2</sub> solutions, mostly at the edges and sides. Remaining surface remained pristine. Cracks were found to grow outward from the pits. The extent of cracking of the sensitized specimen is more severe than that of the as-received specimen. (shown in next 4 slides)
- Minor pitting and cracks observed from sea salt solution (shown in next 4 slides)

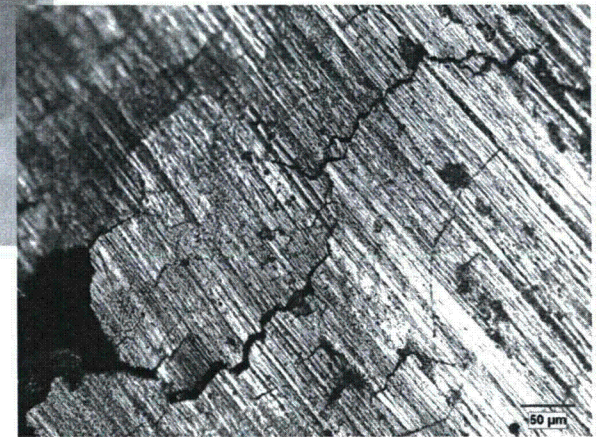


## Task 4: SCC Tests — NaCl

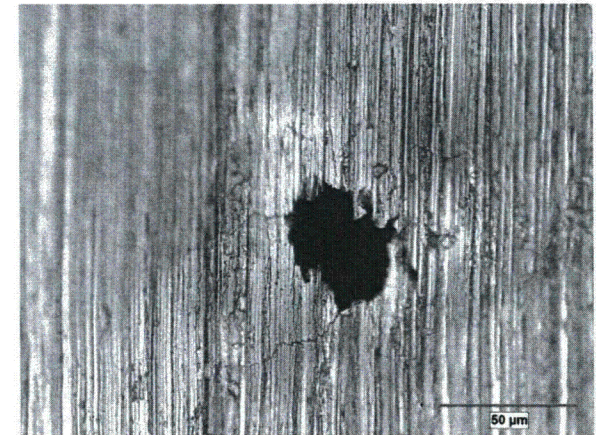
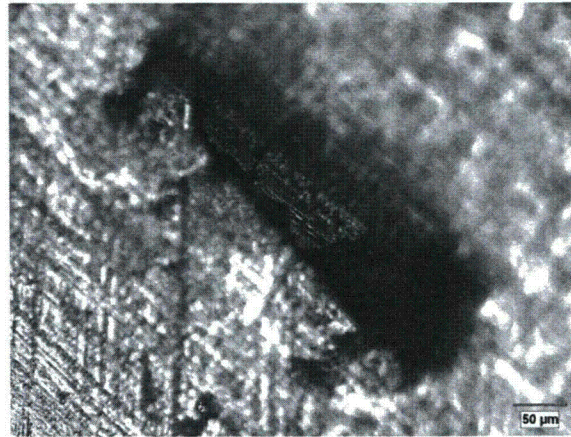
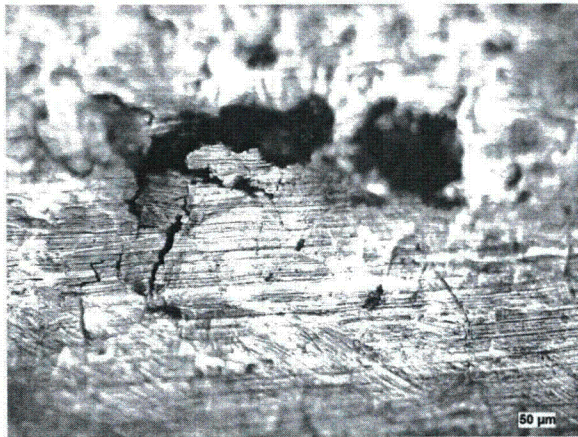
As-received    Sensitized



Sensitized



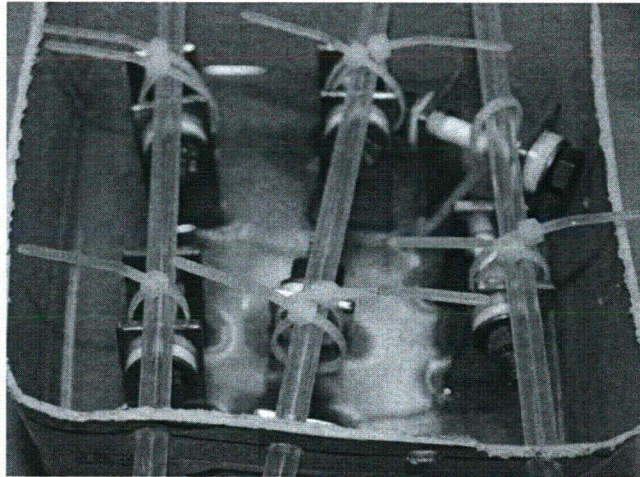
As-received



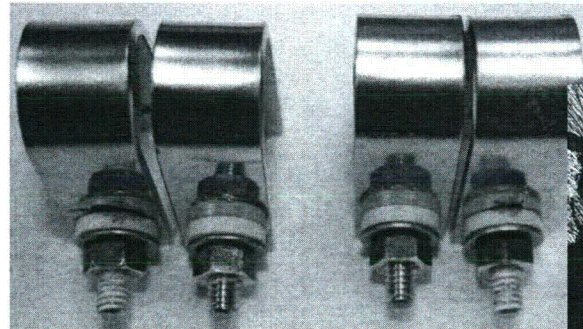
- Cracks grew outward from the pits



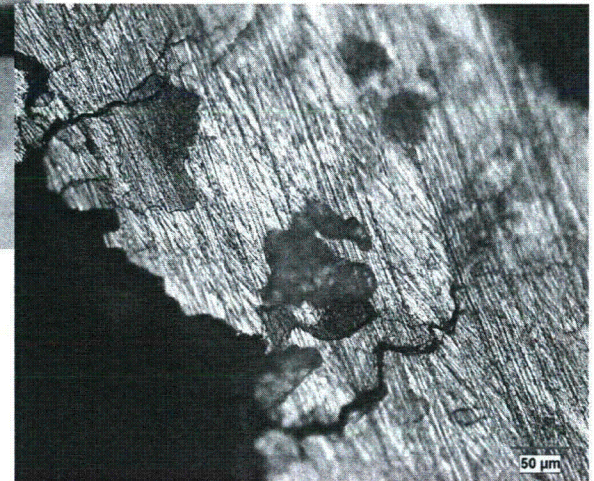
## Task 4: SCC Tests — $\text{CaCl}_2$



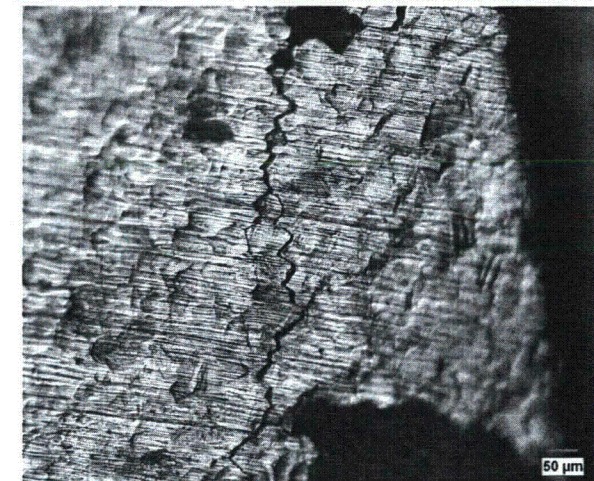
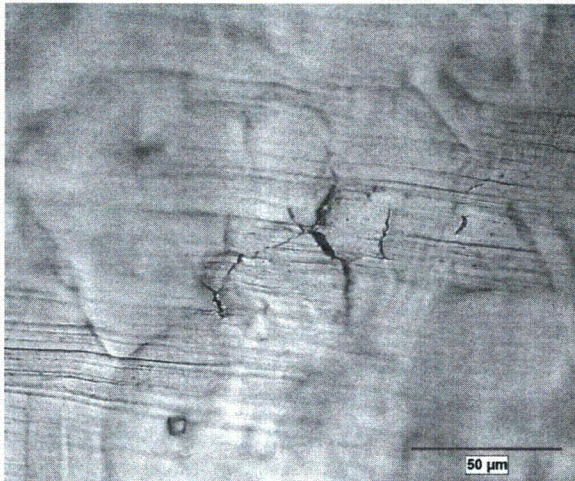
As-received    Sensitized



Sensitized



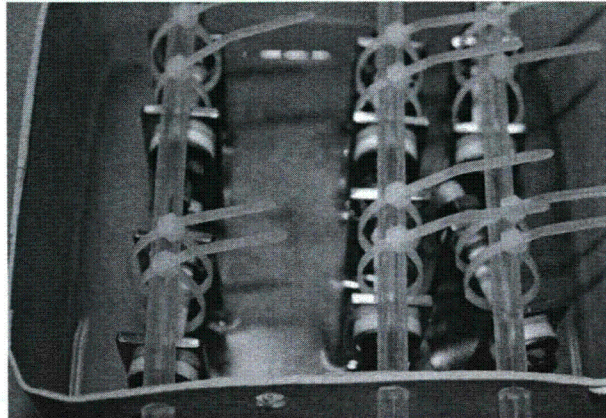
As-received



- Cracks grew outward from the pits. More cracks from sensitized



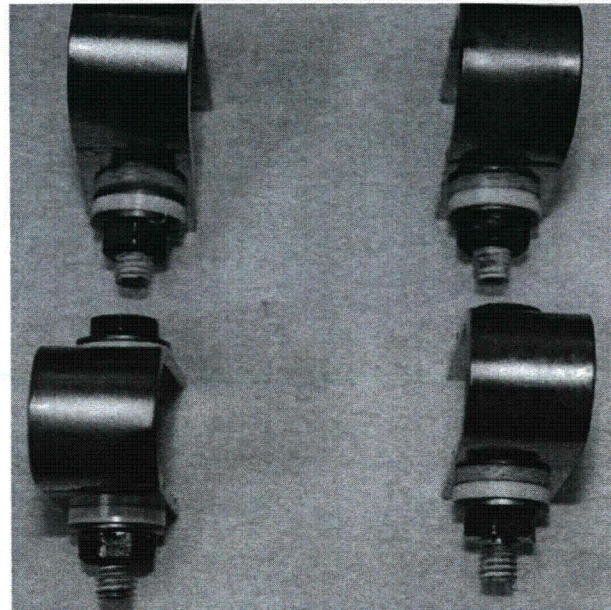
## Task 4: SCC Tests — $\text{MgCl}_2$



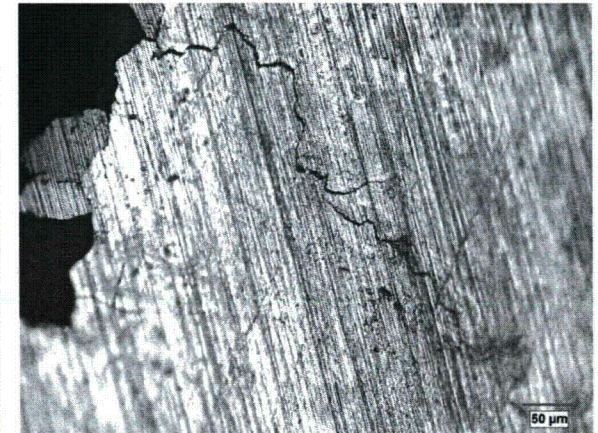
As-received



As-received      Sensitized



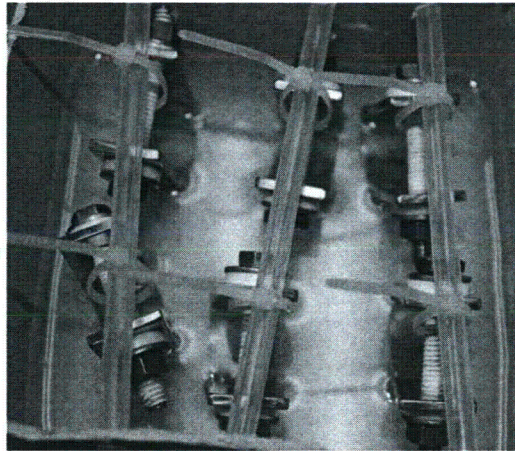
Sensitized



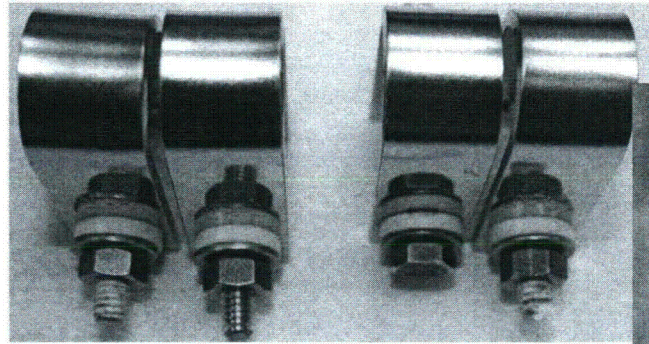
- Cracks grew outward from the pits. More cracks from sensitized



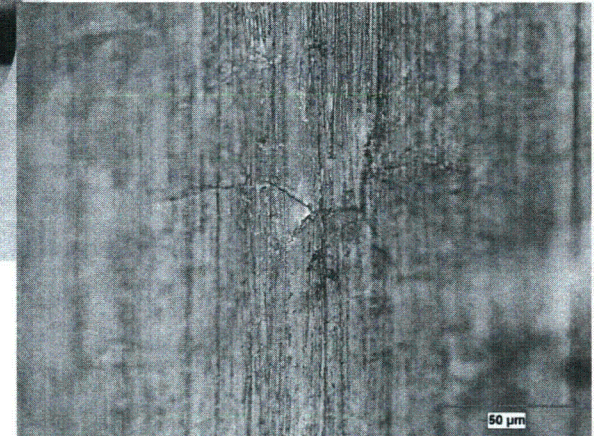
## Task 4: SCC Tests — Sea Salt



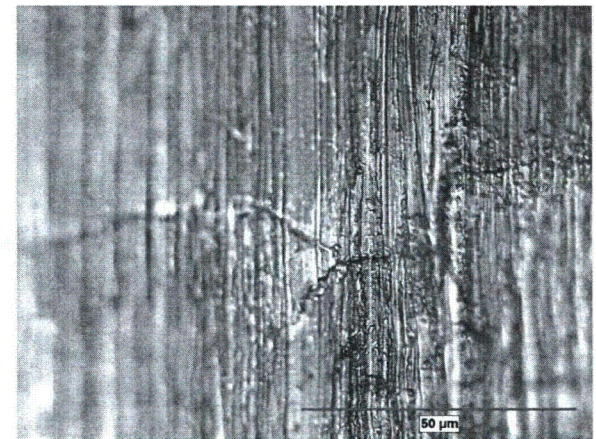
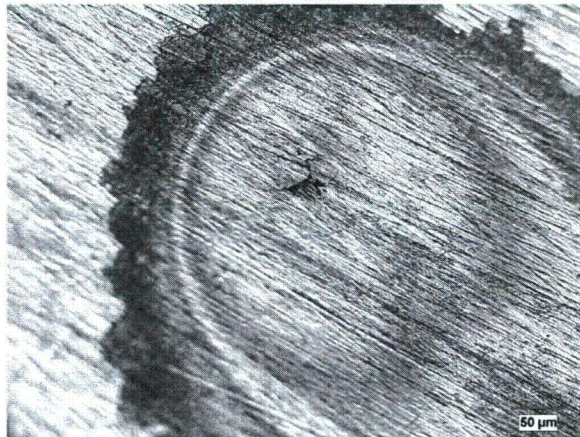
As-received      Sensitized



Sensitized



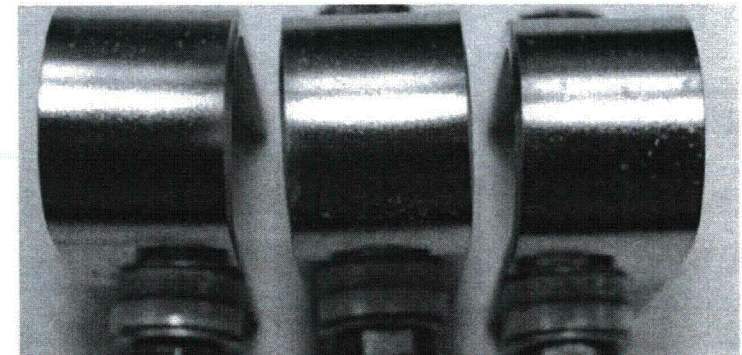
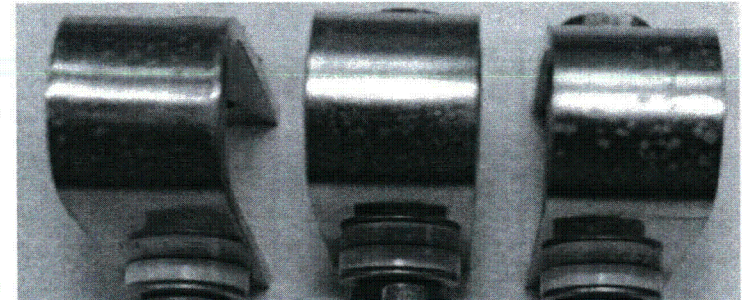
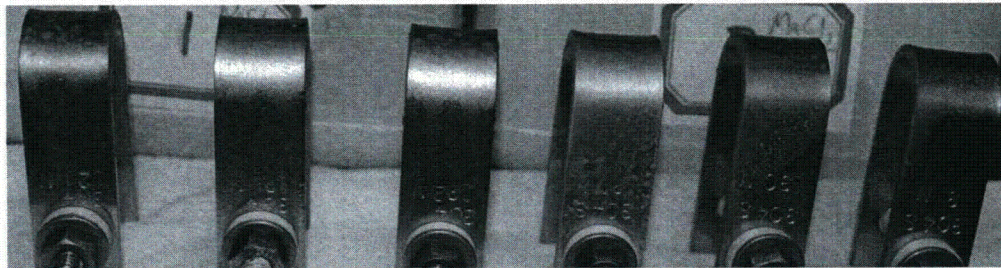
As-received



- Minor pitting and cracks observed



## Task 4: SCC Tests — Posttest U-bends Deposited with 10 g/m<sup>2</sup> Sea Salt and Beakers



- Most of the salts deliquesced and drained down. No corrosion observed from the surface, but the surface was covered with some white scales, which could be the undeliquesced constituents in the



- At equilibrium, the chloride concentration will be measured to compare with the calculated values

- MgCl<sub>2</sub> and CaCl<sub>2</sub> salts have deliquesced. The NaCl and sea salt were partially deliquesced



## Task 4 Path Forward

- Some 1-month posttest specimens were cross sectioned and are under examination
- One as-received and one sensitized specimens will be left in the environmental chamber for a longer period to allow further initiation and growth of cracks. As cracking initiation is observed, further tests will be conducted at higher RH, while maintaining the same temperature of 30 °C to continue to evaluate the dilution effect.

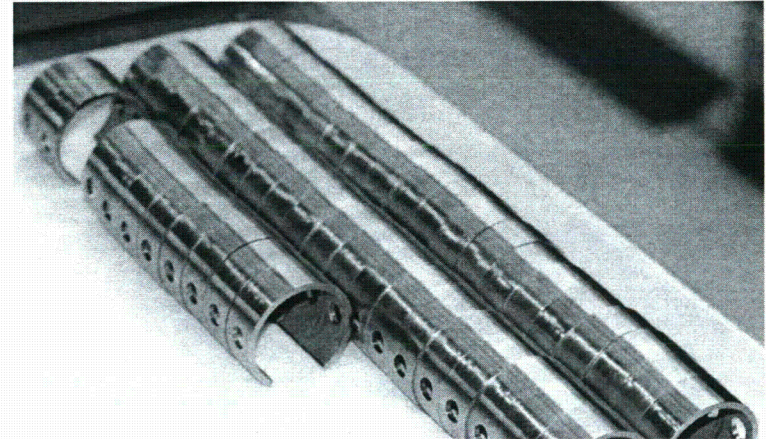
## **Task 5: Evaluate Effects of Stress on Crack Initiation**

- Subtask 5-1: Test plan development to evaluate the effects of stress level on crack initiation
- Subtask 5-2: SCC Tests



# Subtask 5-1: C-ring Specimens

- C-ring specimens
  - Fabricated from seamless 304L stainless steel tube\* with 2" OD and 0.12" wall thickness
  - 0.75" as the width of the specimens to keep the transverse stress low
  - Specimen types: as-received and sensitized
  - Sensitization was conducted at 600 °C for 48 hours



Chemical Composition of Type 304L Stainless Steel Tube											
	C	S	Mn	P	Si	Cr	Mo	Ni	Cu	Co	N
Confirmatory analysis	0.013	0.004	1.06	0.034	0.39	18.65	0.01	8.18	0.04	0.19	0.085

\*Type 304 stainless steel tube is not commonly available in the market



# C-ring SCC Test Matrix

Temperature, °C	Relative Humidity, %	Salt Concentration, g/m <sup>2</sup>	Stress level	Number and Metallurgical Type	Test Duration, month
35	72	10	Yield strength	3 as-Received, 3 sensitized	1 (10/3/12-)
45	44	1	Yield strength	3 as-Received, 3 sensitized	3 (9/28/12-
			16.6% strain¶	3 as-Received, 3 sensitized	1
		10	Yield strength	3 as-Received, 3 sensitized	1 (10/3/12-)
			16.6% strain	3 as-Received, 3 sensitized	1
52	32	10	Yield strength	3 as-Received, 3 sensitized	1 (10/12/12-)
			16.6% strain	3 as-Received, 3 sensitized	1

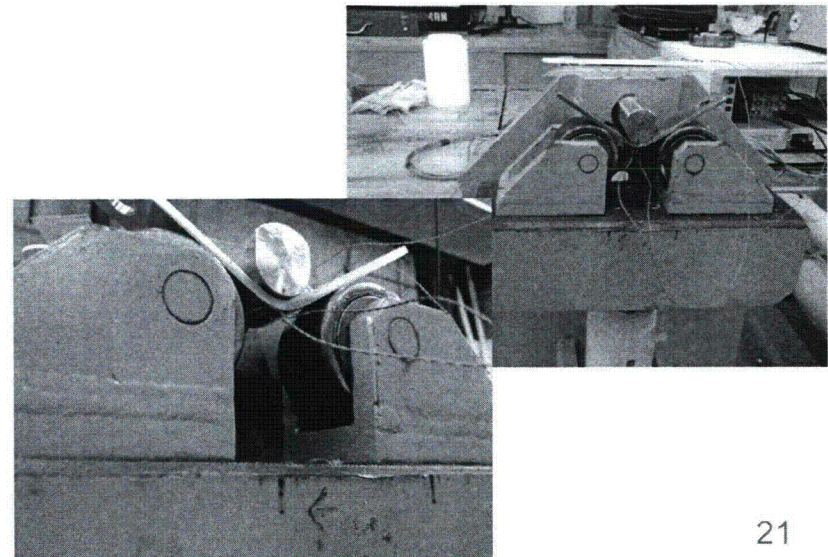
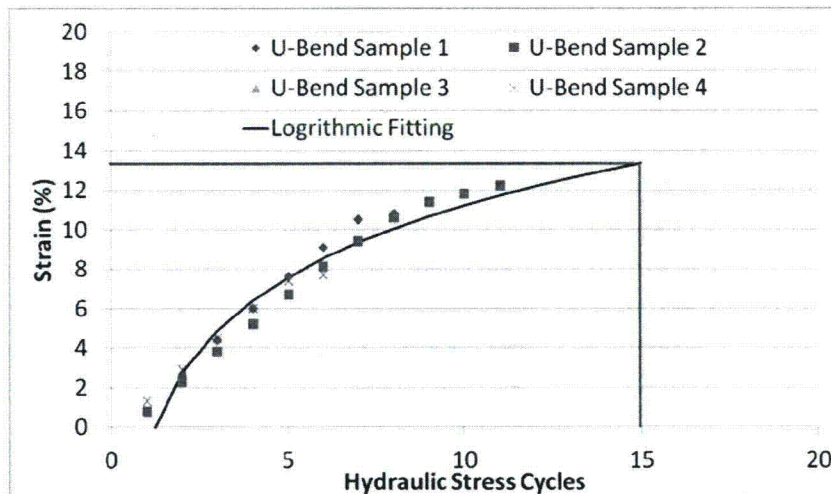
\*Shaded ones are on-going.

¶Strain was calculated from U-bends and confirmatory test of strain is shown in next slide. 20



# U-bend Strain Measurements

- ASTM G30 indicates that U-bend strain is  $T/2R$ , where T is specimen thickness and R is radius of bend
  - Current U-bends have a  $T = 1/8$  in and  $R = 3/8$  in
  - Strain calculated as 16.7 %
- Measurement of actual strain was conducted by gluing strain gauges on U-bends
  - Strain measurements were complicated because at high strain the gauges would detach
- Strain measurements indicate strain was in the range of 13 to 14%

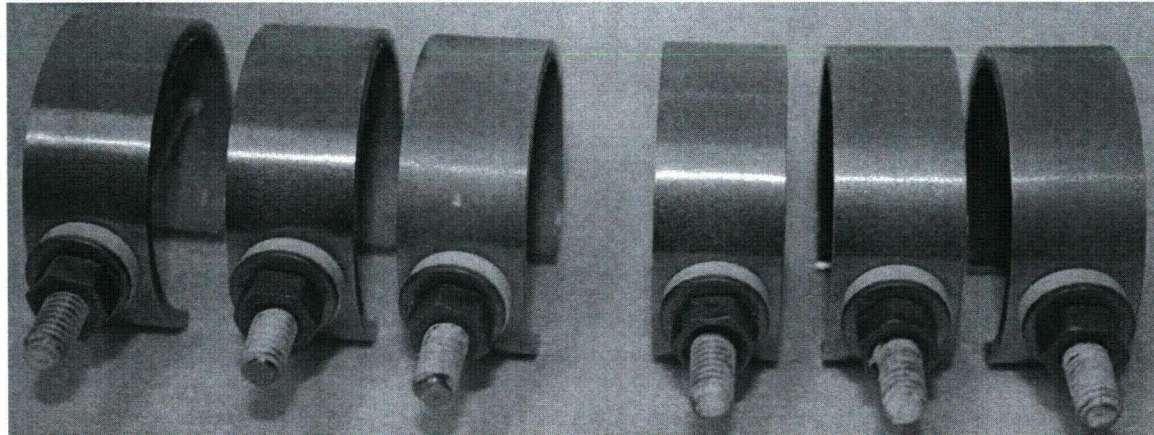




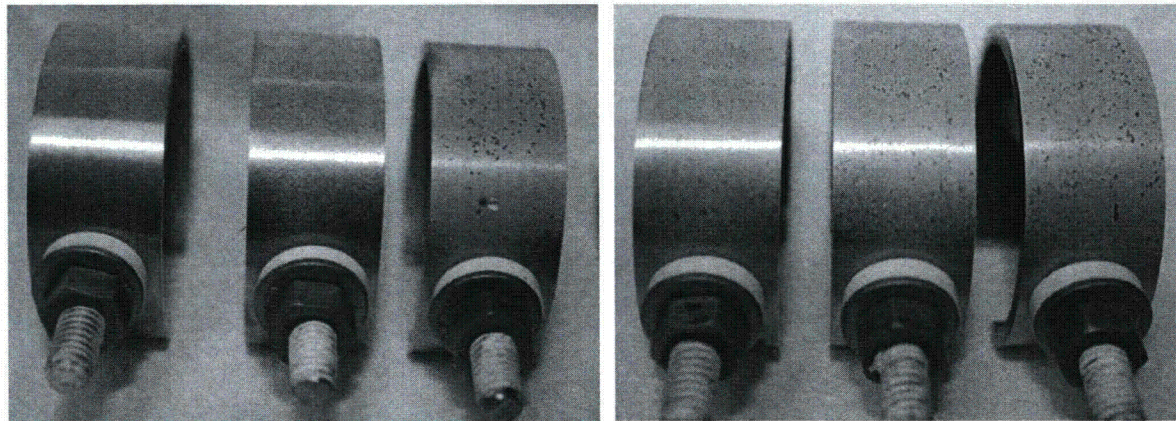
## Subtask 5-2: SCC Tests at 45 °C-44% RH

As-received

Sensitized



C-rings with 1 g/m<sup>2</sup> salts before the test



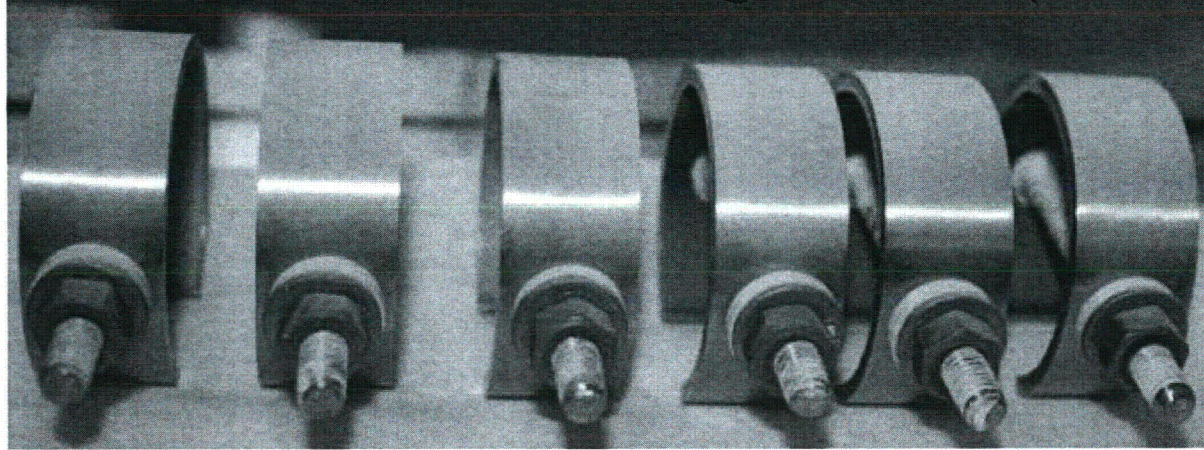
After exposure for 4 days

- Pitting corrosion observed in a couple of days. Same for 10 g/m<sup>2</sup> specimens at the same condition.



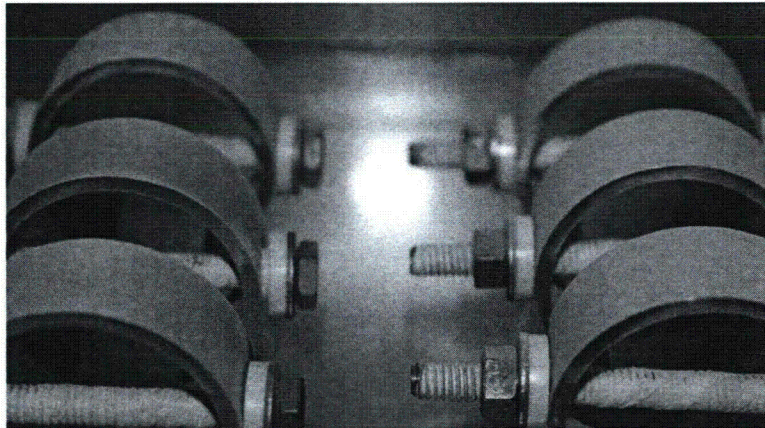
# C-ring SCC Tests

35 °C-72% RH test with 10 g/m<sup>2</sup> salts (before test)



- Salt partially deliquesced after several minutes in chamber
- Minor corrosion observed

52 °C-32% RH test with 10 g/m<sup>2</sup> salts (before test)



- Pitting observed

## Task 5 Path Forward

- 1<sup>st</sup> pull of 35 °C-72% RH and 45 °C-44% RH will be on 11/6/2012
- Specimens with high strain will be prepared and SCC tests will be started
  - Based on discussion with NRC staff



# NUREG/CR Report Outline

- Due by 12/31/12
- Draft is ready at SharePoint

ISFMP (Integrated Spent Fuel Management Program)\  
EXTENDED STORAGE AND TRANSPORTATION, WASTE  
CONFIDENCE and TRAINING\Extended Storage and  
Transportation (EST)\Stress Corrosion Cracking\Outline of  
NUREG-CR Report (Word Document)



4/23/12

# NRC Research at CNWRA – Overview



- Ongoing research program at CNWRA
  - Experimental work October, 2011 – December, 2012
  - NUREG/CR report expected in Spring/Summer, 2013
- Research focus
  - Minimum salt concentration on surface for crack initiation
  - Humidity and temperature conditions for deliquescence and SCC
  - Effects of non-coastal dusts or atmospheric species
- Presentations
  - 2012 NACE Meeting: T. Mintz, *et al*, "Atmospheric Salt Fog Testing to Evaluate Chloride-Induced Stress Corrosion Cracking of Type 304 Stainless Steel," ADAMS Accession Number ML120720549.
  - NRC/NEI RIRP Public Meeting, April 12, 2012. ADAMS Accession Number TBD.



# NRC Research at CNWRA – Preliminary Findings



## ■ Minimum salt concentration for SCC initiation

For cycling between 15 and 30 g/m<sup>3</sup> absolute humidity at 35 and 45°C, cracking is observed on 304 SS U-bend specimens with 1 and 10 g/m<sup>2</sup> of sea salt on surface. Minor pitting is observed on specimens with 0.1 g/m<sup>2</sup> of ASTM sea salt on surface.

Conditions are below DRH for NaCl but above DRH for MgCl<sub>2</sub> for at least part of the cycle. Future tests may consider different temperatures or surface salt concentrations.

## ■ Effects of temperature and humidity on deliquescence and SCC

DRH for ASTM sea salt and constituents was measured at temperatures between 45 and 80°C. DRH for sea salt was close to that of MgCl<sub>2</sub>, about 30 to 35% RH in the temperature range. DRH for NaCl was above 50% RH.

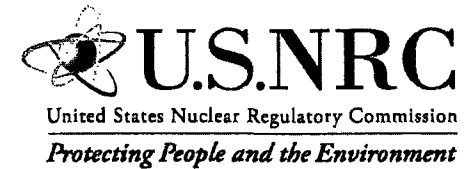
SCC tests are conducted using U-bend specimens exposed in constant temperature and humidity conditions. Specimens will first be tested at high RH conditions where SCC is expected (e.g., above DRH for MgCl<sub>2</sub>). If cracking is observed, subsequent tests progress to lower RH at same temperatures to see if there is a lower threshold RH.

Cracking appears to occur at 40% RH at 60 and 80°C. Subsequent tests are planned for lower RH.

Future tests may consider effects of chloride solubility and dilution at high RH or alternatives to U-bend testing such as notched tensile or C-ring.

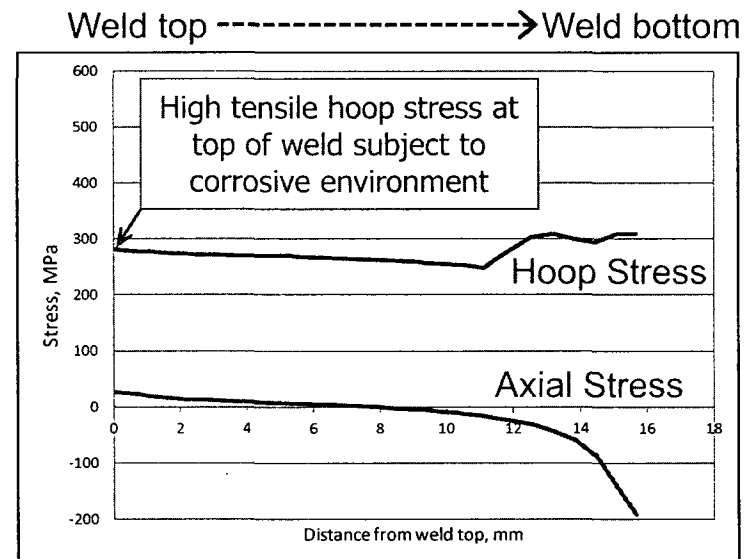


# Other NRC Research Activities



- NRC review of operational experience indicates SCC events attributed to atmospheric chloride exposure of stainless steel at coastal plants. These may provide some insight for identifying susceptible conditions.

- NRC scoping residual stress analyses for a generic lid weld shows potential for through-wall tensile hoop stress. Additional information concerning weld design and parameters is needed for refined analyses.



- Future NRC work may involve reviewing the efficacy of non-destructive examination methodologies for canister systems.

7

**From:** Einzig, Robert  
**To:** ForeignTravel  
**Cc:** Brach, Bill; Ordaz, Vonna; Pstrak, David; Mohseni, Aby; Rubenstone, James; Einziger, Robert; VanWert, Christopher; Brown, Christopher  
**Subject:** RE Einziger travel to ESCP meeting in Berlin Germany June 5, 2011  
**Date:** Tuesday, April 19, 2011 7:40:18 AM  
**Attachments:** Pre-Trip Notification june 2011.docx

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Participate in EPRI Extended Storage Cooperative Program (ESCP) meeting hosted by BAM that has the goal of defining; 1) the International gaps for extended storage and transportation, and 2) what research and demonstrations international participants will do to fill those gaps.



# Foreign Travel Pre-Trip Notification

This is due 30 days before trip start date. Please complete the light shaded area.

## TRAVEL INFORMATION

• <b>Traveler Name(s):</b> (include Office/Division)	Robert E Einziger - NMSS/SFST	• <b>Phone #(s):</b>	301-492-3283
• <b>E-mail Address(es):</b>	robert.einziger@nr.gov	• <b>Location(s):</b>	EBB -3B08
• <b>Multiple Travelers:</b>	<input checked="" type="checkbox"/> Less than 4 <input type="checkbox"/> 4 or more (see below)		
If 4+, Coordinating Office:			
If 4+, Office Director Approvals:	(1)	<b>CHOOSE ONE OF THE FOLLOWING</b>	Date:
(Office Director approves travelers	(2)	<b>CHOOSE ONE OF THE FOLLOWING</b>	Date:
from his/her office only)	(3)	<b>CHOOSE ONE OF THE FOLLOWING</b>	Date:
	(4)	<b>CHOOSE ONE OF THE FOLLOWING</b>	Date:
ADAMS Accession Number: [If 4+, Submit NRC Daily Note with ML# of pre-trip notification 30 days before trip start date]			
• <b>Travel Dates</b> [mm/dd/yyyy]:	06/05 -6/10/2011		
• <b>Destination(s)</b> [City, Country]:	Berlin, Germany		
• <b>Framework:</b>	<input type="checkbox"/> <b>Export and Import Licensing</b> <input type="checkbox"/> <b>Multilateral Cooperation and Assistance</b> <input type="checkbox"/> <b>Treaties, Agreements, and Conventions</b> <input type="checkbox"/> <b>International Cooperative Research</b> <input type="checkbox"/> <b>Bilateral Cooperation and Assistance</b> <input checked="" type="checkbox"/> <b>Other (specify)</b>		
• <b>International Organization:</b>	<input type="checkbox"/> NEA/CSNI <input type="checkbox"/> NEA/CNRA <input type="checkbox"/> NEA/RWMC <input type="checkbox"/> NEA/MDEP <input type="checkbox"/> NEA/CRPPH <input type="checkbox"/> IAEA/NS(TRANSSC) <input type="checkbox"/> IAEA/NS(WASSC) <input type="checkbox"/> IAEA/(NUSSC) <input type="checkbox"/> IAEA/NS(RASSC) <input type="checkbox"/> IAEA/Safeguards <input type="checkbox"/> IAEA/NS <input type="checkbox"/> IAEA/NE <input checked="" type="checkbox"/> IAEA/Technical Cooperation <input checked="" type="checkbox"/> Other: <u>BAM</u>		
• <b>Purpose of Travel:</b>	Participate in EPRI Extended Storage Cooperative Program (ESCP) meeting hosted by BAM that has the goal of defining; 1) the International gaps for extended storage and transportation, and 2) what research and demonstrations international participants will do to fill those gaps.		
• <b>Desired Outcome:</b>	The goal of the meeting is for the International Community to define the gaps they envision for licensing extended storage and transportation (EST), and describing the research they intend to do to fill the gaps. The NRC is in the process of identifying the gaps it sees in regulating EST. It is the travelers goal to determine if the international community has different gaps and the technical reason why. The travel also hopes to influence the direction and scope of the foreign research programs so they might fulfill gaps that the NRC has identified. Synergistically working with the international participants will 1) reduce the amount of research the NRC must conduct, and 2) increase the size of the data base supporting our regulatory decisions.		

• <b>Traveler Role(s):</b>	participant	
• <b>Is this an NRC Core or Non-Core Trip?</b> [Core means NRC-Funded. Non-Core is externally funded or travel to Canada]	<input checked="" type="checkbox"/> Core	<input type="checkbox"/> Non-Core
• <b>Is there a speech or presentation to be given?</b> [If yes, send ADAMS ML# of presentations in an EDO One Week Look Ahead]	<input type="checkbox"/> Yes	<input checked="" type="checkbox"/> No
• <b>Are policy issues or other items of Commission interest to be raised?</b>	<input type="checkbox"/> Yes	<input checked="" type="checkbox"/> No
If yes, how will the Commission be informed?		



**From:** Helton, Donald  
**To:** Pires, Jose; Esmaili, Hossein  
**Subject:** FW: TEPCO : Press Release | Report on the Steel Beam Falling into Unit 3 Spent Fuel Pool at Fukushima Daiichi Nuclear Power Station to the Nuclear Regulatory Authority  
**Date:** Wednesday, October 03, 2012 9:04:00 AM

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We've seen most of these pictures before, but there were a couple of schematics that were new. Just an FYI, I don't think this helps us much in addressing the debris questions...

-----Original Message-----

From: Marksberry, Don  
Sent: Wednesday, October 03, 2012 6:27 AM  
To: Helton, Donald  
Subject: TEPCO : Press Release | Report on the Steel Beam Falling into Unit 3 Spent Fuel Pool at Fukushima Daiichi Nuclear Power Station to the Nuclear Regulatory Authority

[http://www.tepco.co.jp/en/press/corp-com/release/2012/1221393\\_1870.html](http://www.tepco.co.jp/en/press/corp-com/release/2012/1221393_1870.html)

Some photos and diagrams in the Japanese report

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