U.S. Department of Energy
Office of Civilian Radioactive Waste Management

www.ocrwm.doe.gov

Plans for Long-Term Corrosion Testing and Recent Results

Presented to:
Nuclear Waste Technical Review Board

Presented by:
Paige Russell (DOE)
Doug Wall (Lead Lab)
Neil Brown (Lead Lab)

January 16, 2008
Las Vegas, NV

Outline of Material Presented

- **Corrosion test plans**
  - **Objectives of long-term testing plan**
  - **Approaches to near-term and long-term corrosion testing**
  - **Tasks included in corrosion test planning**
- **Recent Results**
  - **Behavior of Alloy 22 and witness materials under deliquescent conditions**
  - **High Temperature, Controlled Dewpoint Exposure System**

Objectives of Long-Term Corrosion Testing Plan

Long-Term Corrosion Testing Plan. **SAND2007-7027. Albuquerque, NM: Sandia National Laboratories.**

- **Reduce model uncertainty**
- **Improve model defensibility and build consensus in the scientific community**
- **Establish estimates of testing needs to inform planning for the next-generation long-term corrosion test facility**
- **Performance confirmation planning will identify a subset of the testing activities and perhaps additional tests that will be become part of the performance confirmation**

Approaches to Near-Term and Long-Term Testing

- **FY08**
  - **Reduce uncertainty in current models**
  - **Improve confidence in the screening justification for screening out localized corrosion of the waste package under deliquescent conditions**
  - **Improve confidence in stress corrosion cracking (SCC) model through continuation of ongoing testing**
- **FY09/FY10**
  - **Systematic investigation of underlying corrosion processes**
- **FY11-FY17**
  - **Long term testing**
  - **Secondary issues (e.g., crevice material)**

Corrosion Test Planning

Task Description	FY08	FY09-FY10	FY11+
Task 1 - Weight Loss for Alloy 22 and Titanium and Surrogates	• Analysis of baseline materials from 9.5 year exposures	• Analysis of surrogate materials from 9.5 year exposures	• Long-term exposures
Task 2 - Crevice Corrosion of alloy 22, Titanium Alloys and Surrogates	• Analysis of baseline materials from 9.5 year exposures	• Crevice behavior at open-circuit conditions to evaluate propagation / stifling	• Long-term exposures • Secondary effects (e.g., crevice material, force)
Task 3 - Critical Potential of Alloy 22	• Reproduce excluded data, improve statistics	• Determine threshold values of Cl^-, NO_3^-	• Continue threshold value assessment • Secondary effects (e.g., surface finish, force, crevice material)
Task 4 - Open Circuit Potential of Alloy 22	• Improve statistics • Cathodic kinetics	• Systematic study as a function of Cl^-, NO_3^-, temp and pH • Cathodic kinetics	• Long-term exposures • Secondary effects (e.g., other anions, cations, surface finish)
Task 5 - Temperature Dependence of Alloy 22 and Titanium Alloys	• Weight-loss and electrochemical testing of Alloy 22	• Studies in seepage and deliquescent environments	• Continue FY09/10 studies • Longer-term studies

Corrosion Test Planning (Continued)

Task Description	FY08	FY09-FY10	FY11+
Task 6 - Critical Potential of Titanium Alloys	• Systematic study as a function of F^-, Cl^-, and NO_3^-	• Continue FY08 study for Grades 28 and 29	• Effect of welds, other anions and contaminants
Task 7 - Characterization of Alloy 22 and Titanium Oxides	• Samples from 5 year exposures • Technique development	• Samples from 9.5 year exposures • Deliquescence exposure samples	• Samples from other FY11+ tasks • Effects of electrochemical and chemical stresses
Task 8 - Alloy 22, Deliquescence Testing	• Technique development • Evaluate stifling processes • Initiation studies	• Prepare samples for open circuit and oxide characterization studies	• Long-term general corrosion rate • Long-term crevice behavior of Alloy 22 and surrogate materials
Task 9 - Microbial Influenced Corrosion on Alloy 22	• Characterization of existing MIC samples	• Design and procurement of MIC testing apparatus	• Long-term MIC testing
Task 10 - Aging and Phase Stability of Alloy 22	• Expand model range to include Cr- and Mo- rich compositions	• Phase stability of surrogate materials • Phase stability of welded Alloy 22	• Long-term, low temp stability studies • Minor alloying elements and impurities

Corrosion Test Planning (Continued)

Task Description	FY08	FY09-FY10	FY11+
Task 11 – Stress Corrosion Cracking	• Creep-rupture of titanium • Examine T-threshold for SCC in Alloy 22 • Continue ongoing tests	• Continue FY08 testing	• Long-term U-bend, CT and constant load testing
Task 12 – Analysis of U-bend Samples from the LTCTF	• Examine samples from most aggressive environments	• Examine remaining samples from long-term exposures	-
Task 13 – Hydrogen Embrittlement	-	• Relate bulk H to mechanical properties	• Relate exposure conditions to mechanical properties and H content
Task 14 – Corrosion Behavior of Neutron Absorber Materials	-	-	• Long-term exposures
Task 15 – Analysis of Stainless Steel Corrosion Products	-	• Determine composition and structure of 316SS corrosion products from LTCTF	-

Corrosion Test Planning (Continued)

Task Description	FY08	FY09-FY10	FY11+
Task 16 – Corrosion Testing Under Dripping Conditions	-	• Design / identify test system	• Evaluate corrosion processes under dripping conditions to compare with results from inundated conditions
Task 17 – Open Circuit Potential of Titanium	-	-	• Long-term exposures • Secondary effects (e.g., thermal oxide, surface finish, contact with tuff)

Recent Test Results: Alloy 22 and Surrogate Materials Under Deliquescent Conditions

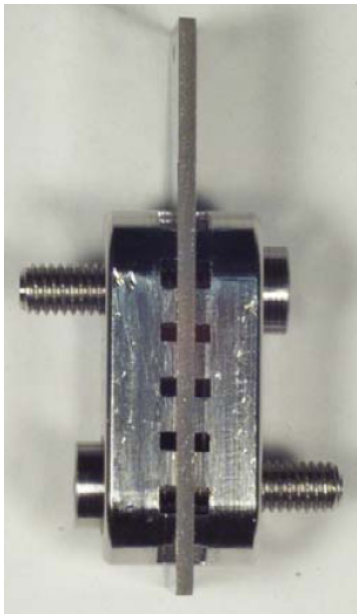
Materials: Alloy 22, 825, carbon steel, 304 SS, 316 SS

Salt Assemblages: $NaCl + NaNO_3 + KNO_3$
$NaCl + NaNO_3 + KNO_3 + Ca(NO_3)_2$ } eutectic compositions

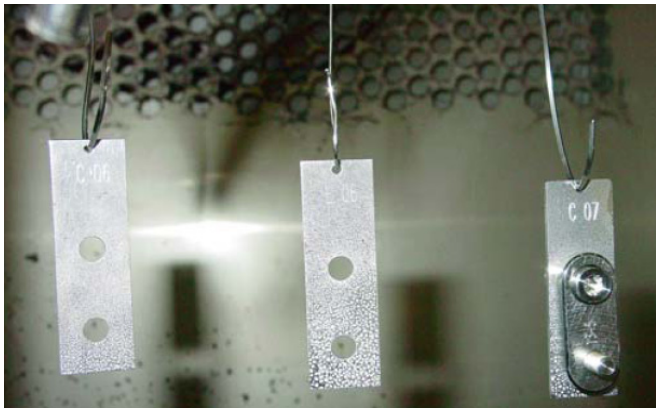
Temperature: 180ºC

Atmosphere: 100% Steam (~10%RH)

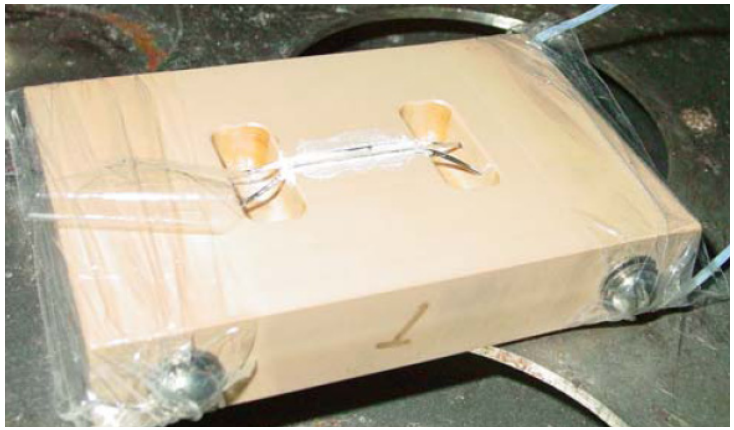
Test Duration: 50 days



Creviced sample assembly



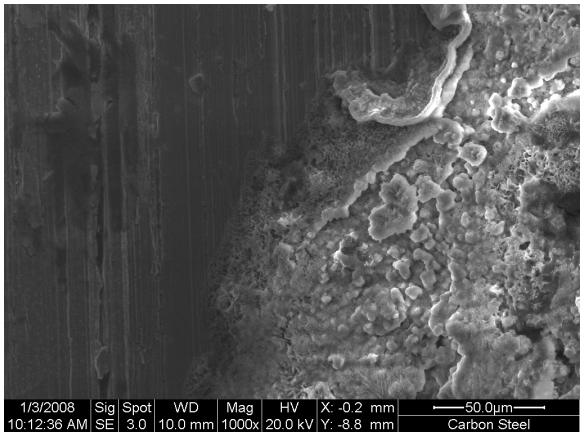
Samples decorated with salt assemblages hanging in test chamber



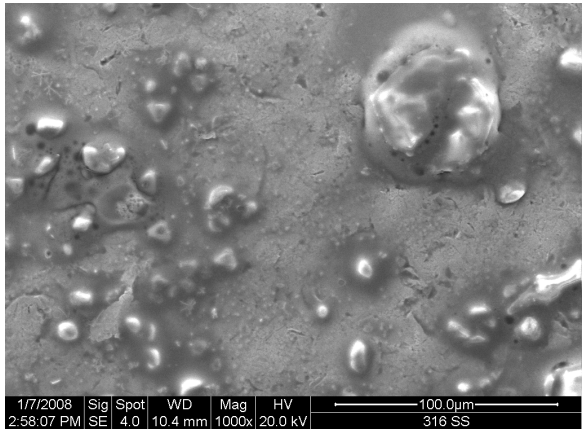
Parallel Pt wires used to detect deliquescence of salt assemblage

Recent Test Results: Alloy 22 and Surrogate Materials Under Deliquescent Conditions (Continued)

180ºC, Steam, 50 days 4-salt assemblage	
Carbon Steel	corrosion
304SS	corrosion
316SS	corrosion
825	no corrosion
Alloy 22	no corrosion

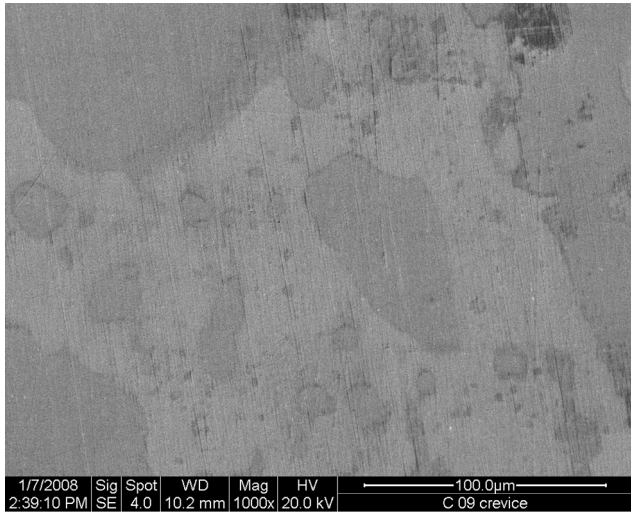


Uncreviced carbon steel



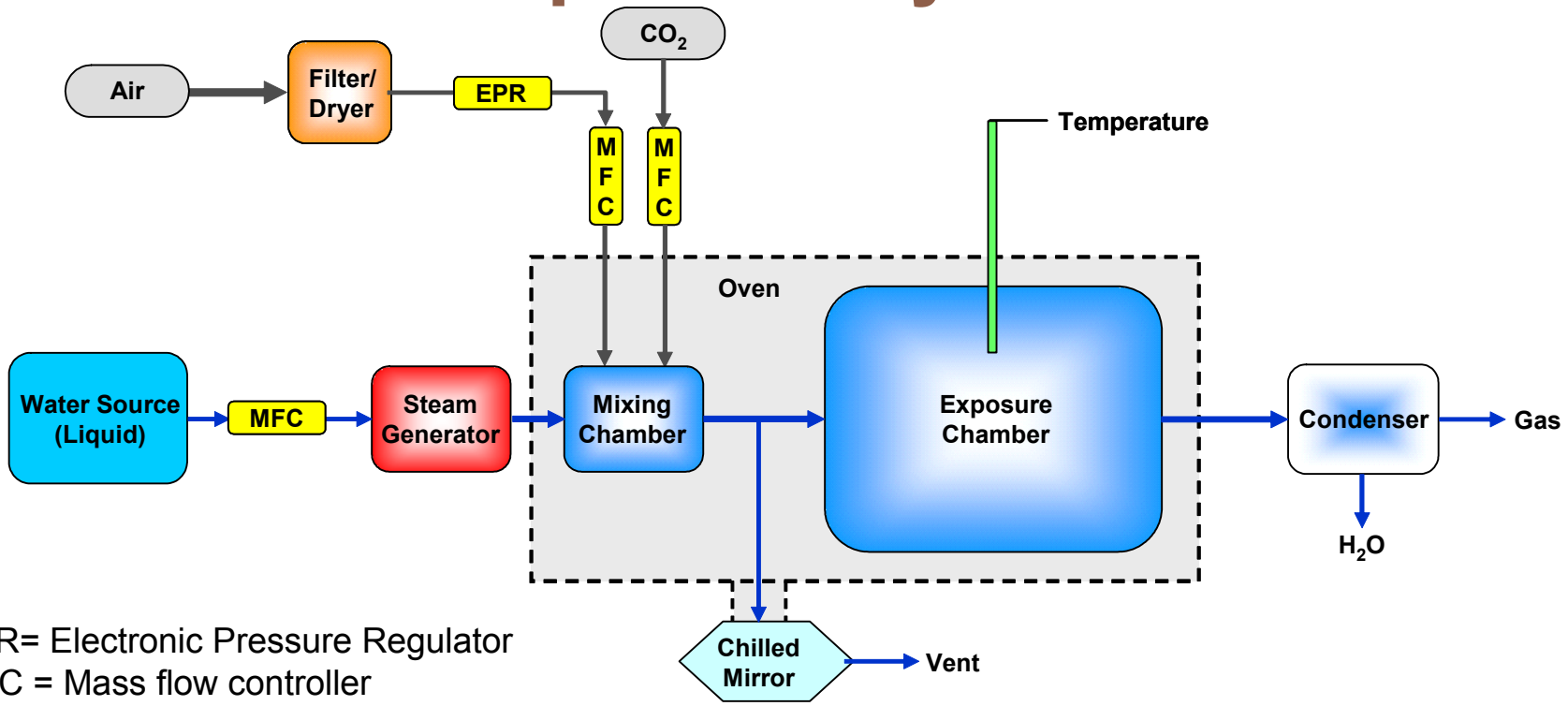
Uncreviced 316SS

- **3-salt assemblage:** evidence of deliquescence followed by dryout
- **4-salt assemblage:** sustained deliquescence detected by low resistance between Pt wires



Creviced Alloy 22 sample

High Temperature, Controlled Dewpoint Exposure System

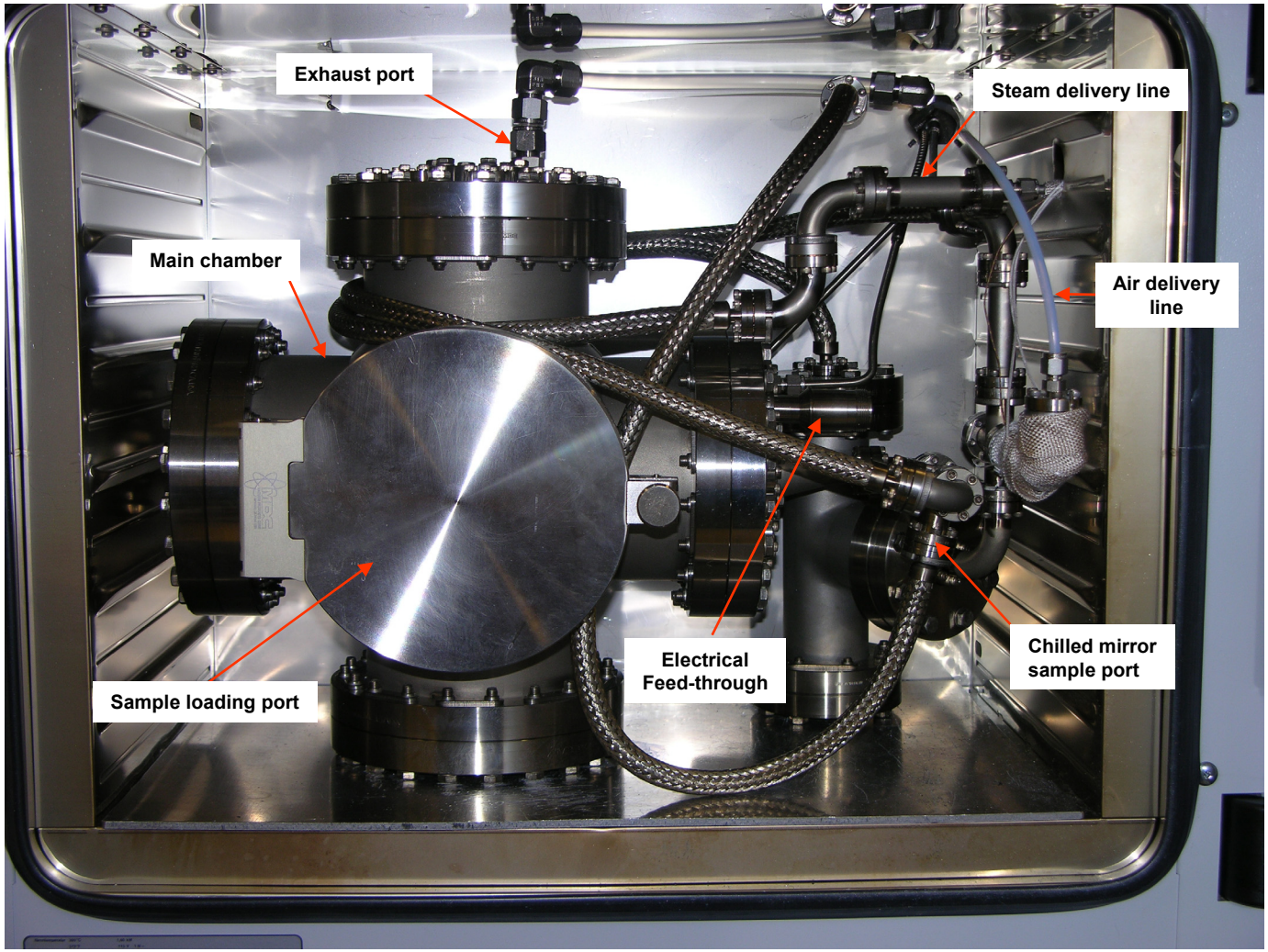


EPR= Electronic Pressure Regulator
MFC = Mass flow controller

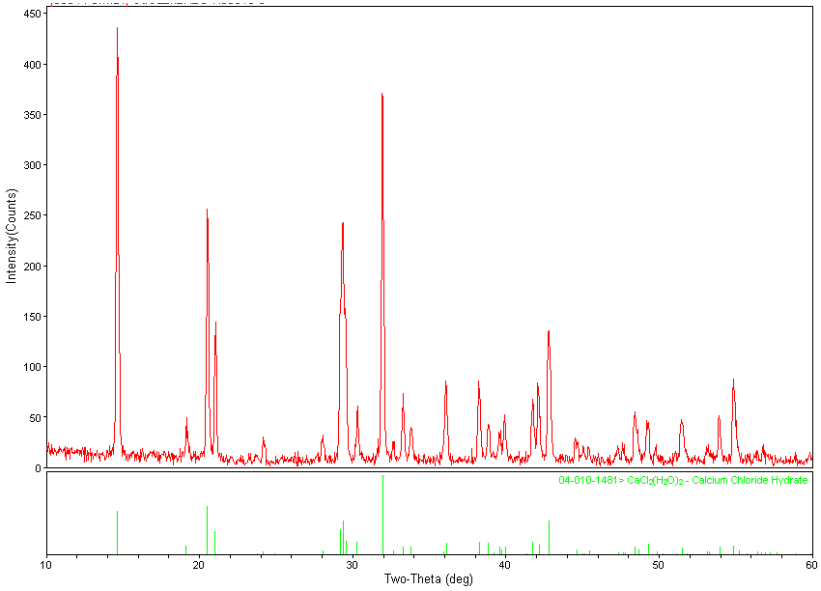
System Capability

- **Max Process Temperature >250°C**
- **Max Dewpoint = 95°C (at 1 atm)**
- **Electrical access to samples**

High Temperature, Controlled Dewpoint Exposure System (Continued)



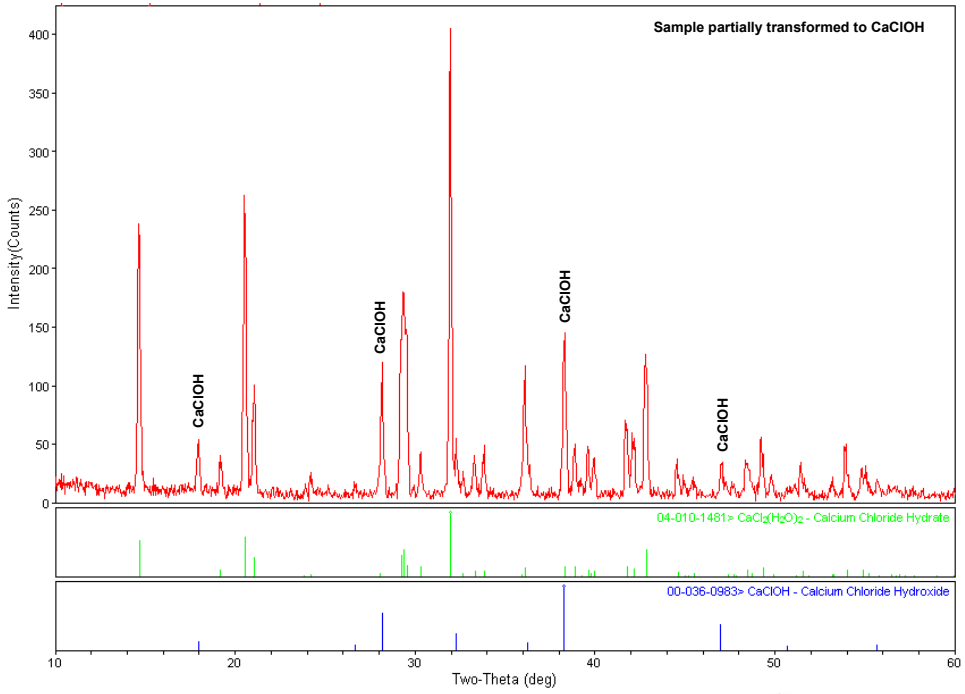
Initial Test Results: $CaCl_2$ Degassing



Temperature: 150ºC
Dewpoint: 91ºC
Test Duration: 1 week

$CaCl_2 \cdot 2H_2O$

$CaClOH$

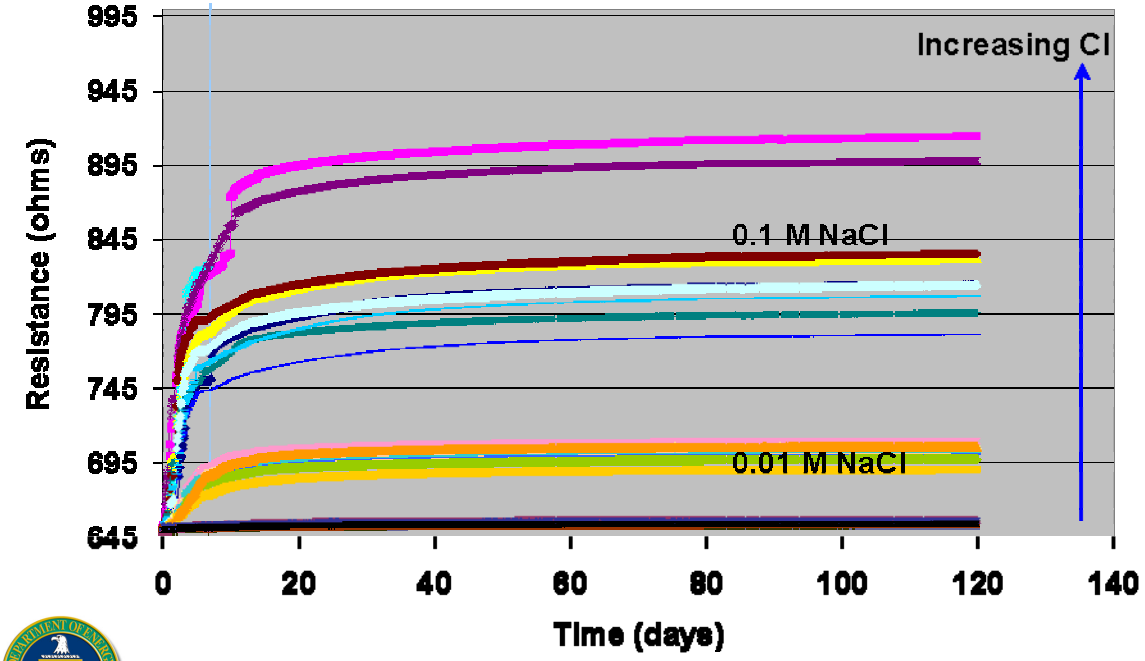


In-Situ Electrical Resistance Technique is Under Development for Monitoring Corrosion

Direct-Current Potential Drop is Used to Detect Resistance Change Due to Material Loss or Conversion to Corrosion Products

- Initiation
- Damage accumulation (relative rates)
- Stifling

Example: corrosion stifling on nichrome resistors at 60ºC and 85%RH (provided by Rob Sorensen, Sandia National Laboratories)



The resistance technique will be used to evaluate stifling and the relationship to initial levels of contaminant loading

Conclusions

- A multiyear plan for corrosion testing activities has been developed
- Recent test results support the conclusion that Alloy 22 will not undergo localized corrosion under deliquescent conditions in the presence of the three- and four-salt assemblages
- A new capability has been developed that enables testing across all temperature and humidity ranges expected during the thermal pulse