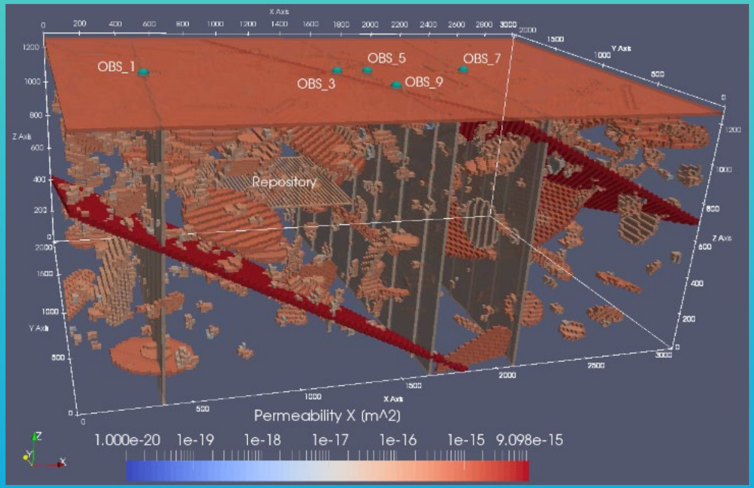

Stress Corrosion Cracking Research at Sandia National Labs

U.S. Nuclear Waste Technical Review Board
Winter 2022 Board Meeting (Virtual)
March 1-2, 2022

Charles Bryan, Rebecca Schaller, Andrew Knight, Brendan Nation, Ryan Katona, and Erin Karasz
Sandia National Laboratories

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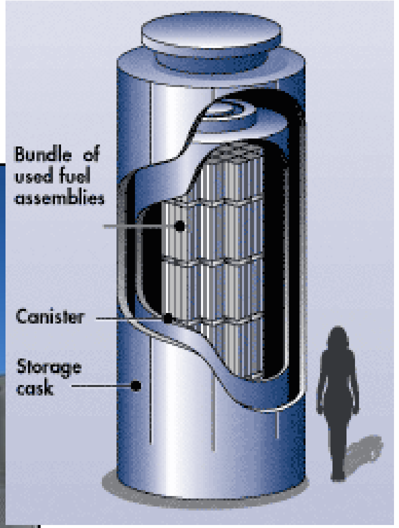
SAND2022-2045 PE

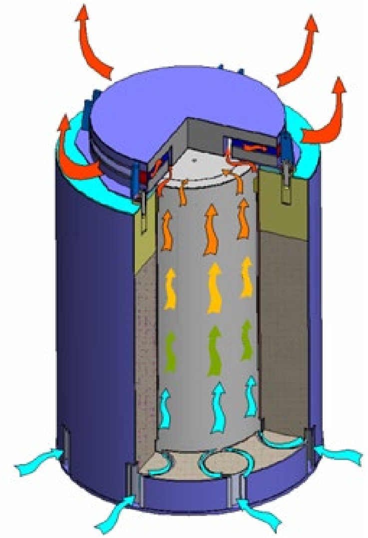
Background—Program Goals

- United States currently has over 86,000 metric tons of Spent Nuclear Fuel (SNF); about 50,000 metric tons in dry storage systems.
- The dry storage systems are intended as interim storage until a permanent disposal site is developed. However, lack of a repository pathway means that some SNF will remain in storage for decades beyond the original storage system specifications.
- In most systems, SNF is stored in stainless steel (304 SS) canisters. Canisters are stored in passively-ventilated overpacks, and accumulate surface dust over time. Deliquescence of chloride-rich salts could potentially lead to Stress Corrosion Cracking (SCC)
- Understanding SCC of interim storage containers has been determined to be a high priority data gap (EPRI 2011; DOE 2012; NRC 2012).
  - Timing and conditions of occurrence
  - Risk of canister penetration

Canistered SNF Dry Storage Systems
Two Standard Designs, with passive cooling

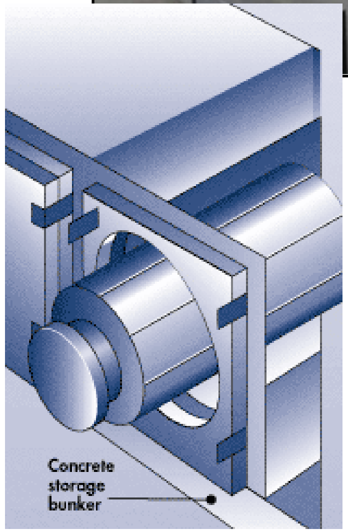
Vertical—In vertical systems, the welded stainless steel canister sits upright within a steel-lined concrete overpack.

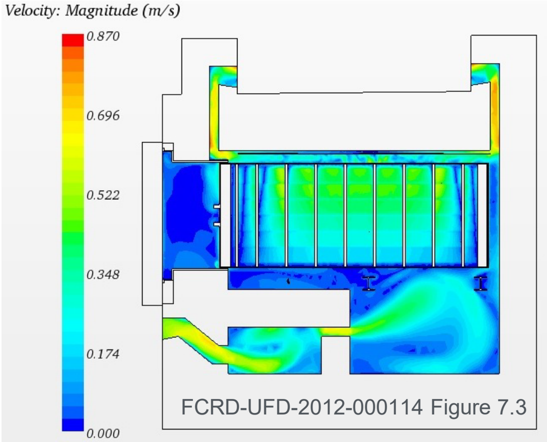




Pathway for air flow through the overpack.

Horizontal—In horizontal systems, the welded canister rests on its side upon rails within a concrete vault.



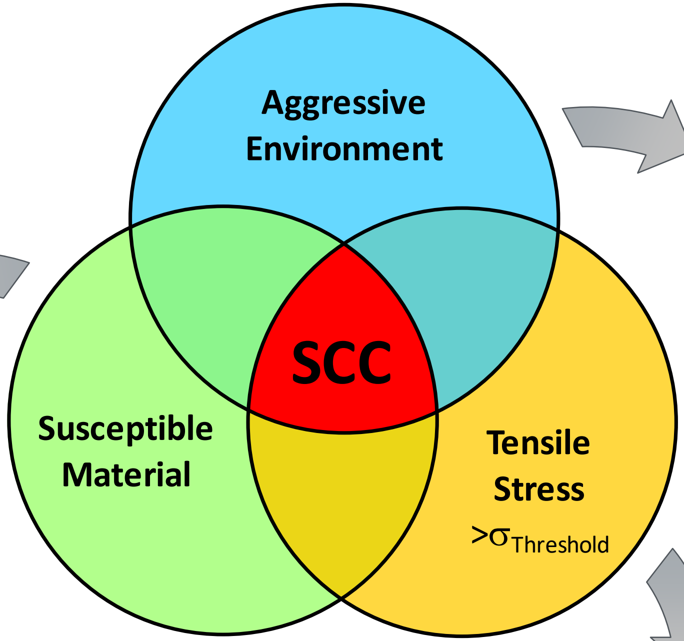


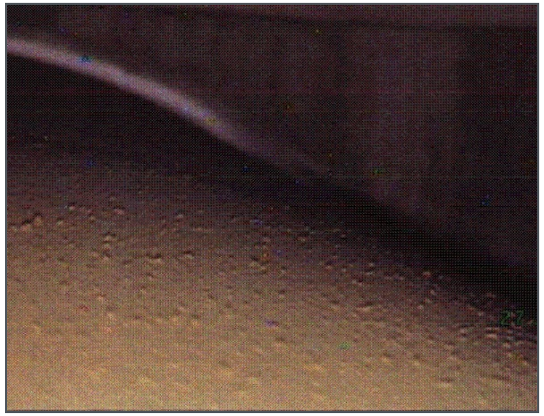
FCRD-UFD-2012-000114 Figure 7.3

Criteria for Stress Corrosion Cracking

To evaluate the potential for canister SCC, each must be considered

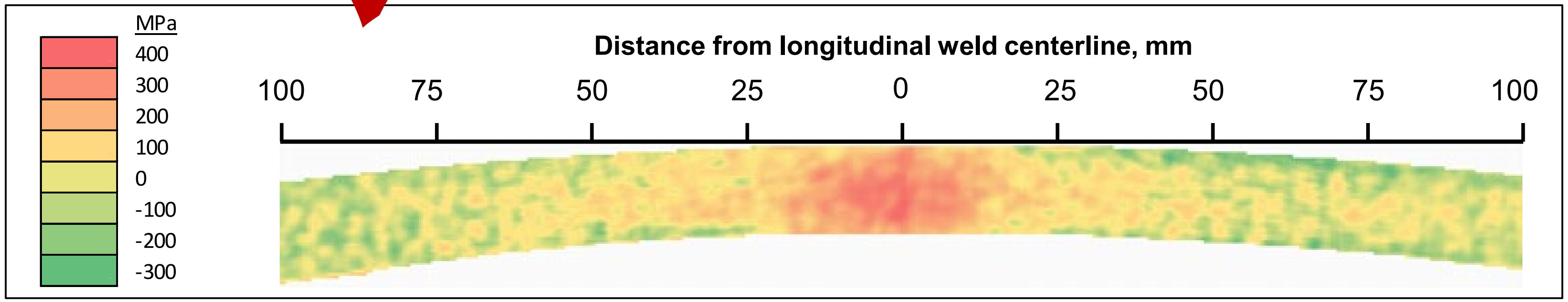
Weld zone, Ranor 304 SS plate





Dust on canister surface at Calvert Cliffs (EPRI 2014)

Chloride salts present at some sites. As canisters cool, corrosive brines may eventually form.



Measured weld residual stresses (SNL 2016)

Goal: Evaluating the risk

- What sites are at risk?
- When will corrosion initiate?
- Evolution of corrosion damage?
- Timing of crack initiation?
- Rates of crack growth and timing of potential penetration?

Overview Slide: CISCC Program

DOE CISCC Program

PNNL	SNL	SRNL	ORNL
Dust Deposition Analysis: Modeling	Canister Environment: Sampling and Measurement	CGR Validation: Large Plate Test	Crack Consequence
Parametric crack growth studies as f(materials properties and environment)	Brine stability and thermodynamic modeling		
Crack Consequence	Corrosion as f(environment and material)		
Cold Spray Development	Pitting model for parameterization		
	Single effect CGR studies		
	Crack Consequence		
	Cold Spray Evaluation		

DOE Collaborations

NEUP

Cold Spray/Welding
- Purdue
- The Ohio State
- Univ. of Cincinnati
- Univ. of Idaho
- Univ. of Wisconsin

CGR
- Univ. of Virginia

Coatings and Mitigation
- N. Carolina State
- Univ. of Nebraska
- Univ. of Nevada Reno
- Univ. of S Carolina
- Univ. of Wisconsin Madison
- Virginia Tech

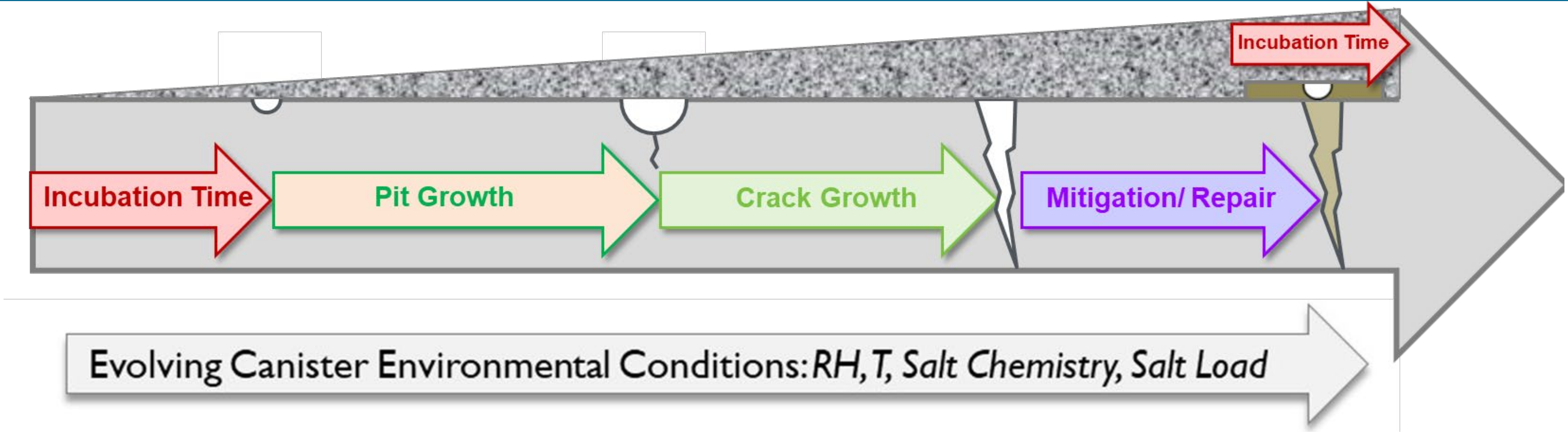
Other Collaborations

CGR and Pitting
- Univ. of Virginia
- The Ohio State
- Univ. of New Mexico
- DNV-GL

- EPRI
- EPRI
- EPRI

***CGR = crack growth rate**

Timeline, Stress Corrosion Cracking of SNF Dry Storage Canisters



Sandia's role:

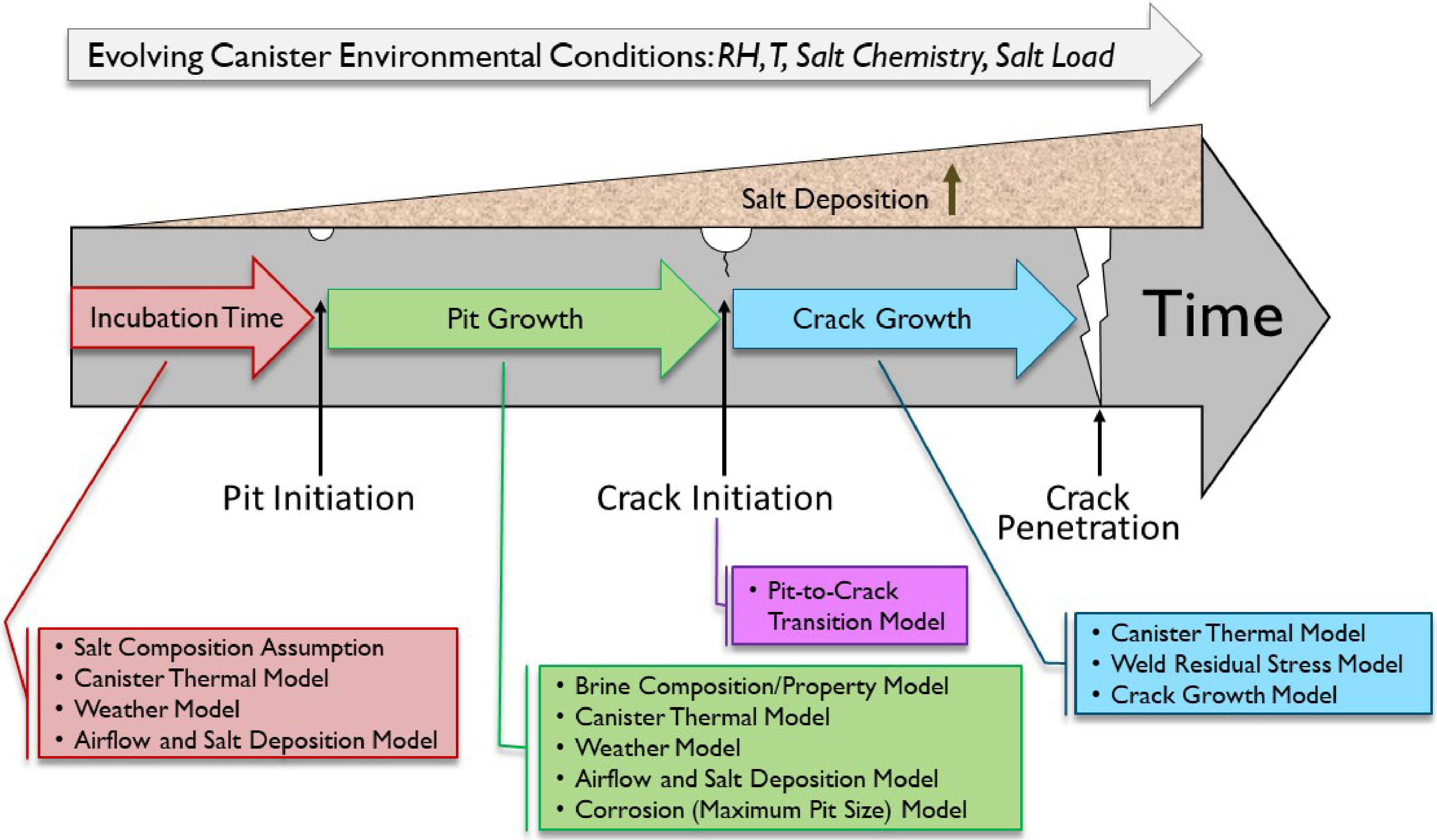
- **Defining the canister surface environment**
- **Importance of canister environment for pitting/SCC**
  - Dust, diurnal cycles, salt and brine chemistry/composition
  - Environmental influences on pit morphology and implications
    - Pit-to-crack transition
  - Pitting kinetics
    - Brine composition and cathodic limitations – predicting maximum pit size
- **Crack growth rate studies**
- **Mitigation and Repair—cold spray and coatings**

Probabilistic Model for Canister SCC

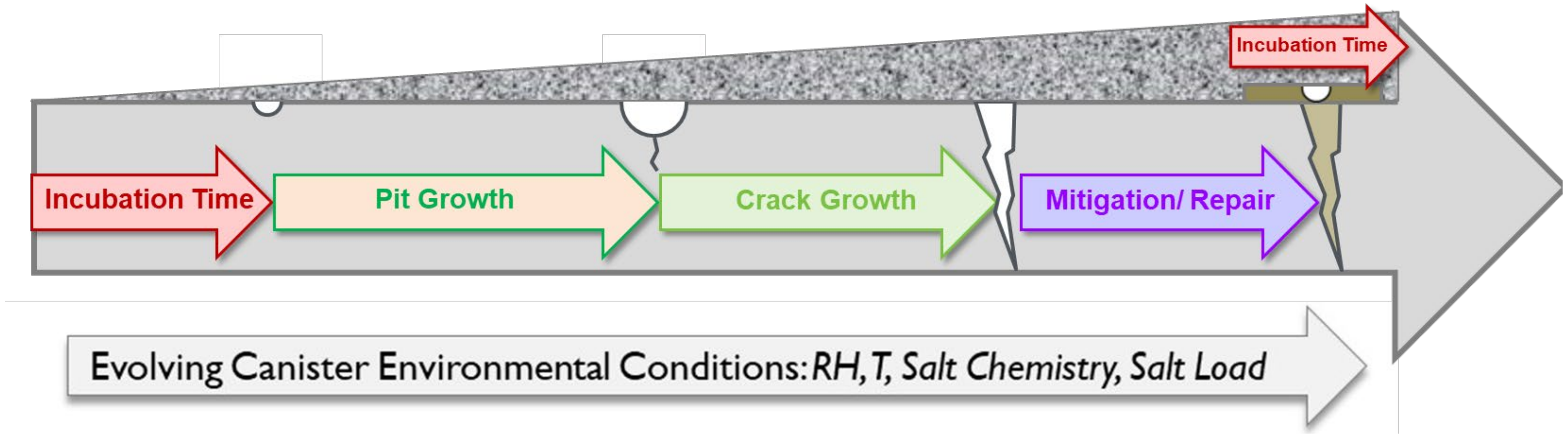
Provides the Framework for Experimental Studies

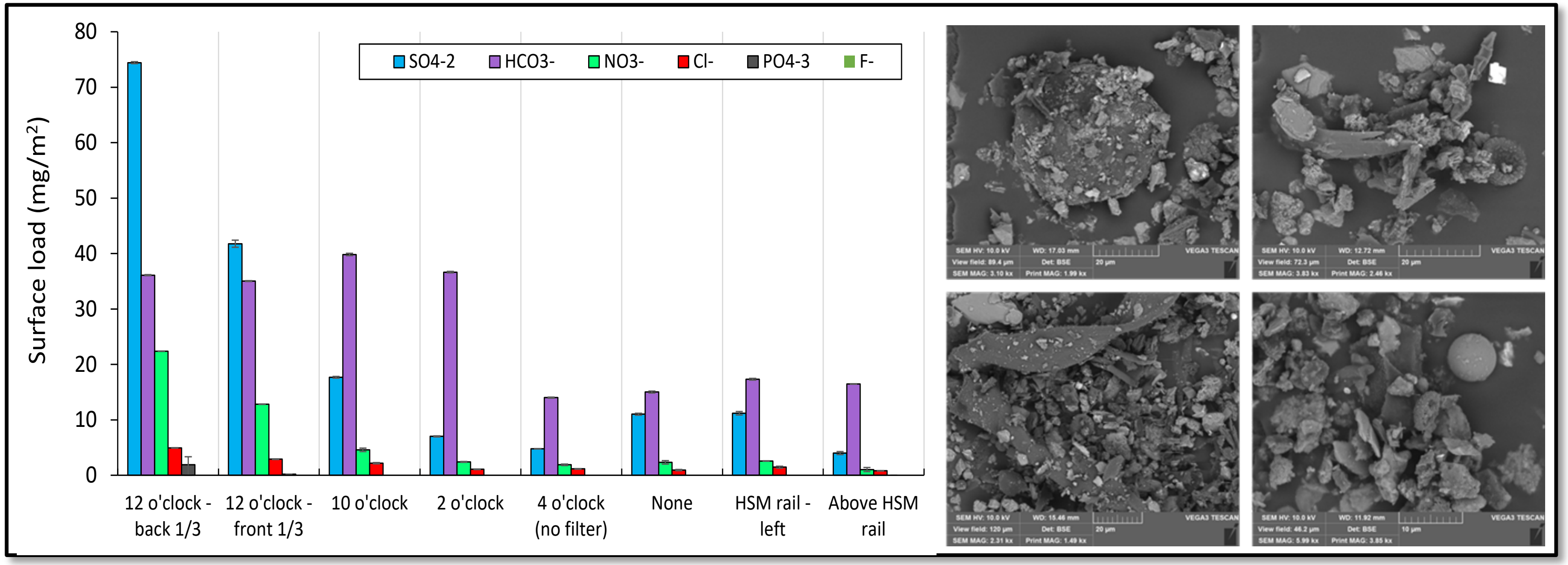
Evaluating timing of canister SCC initiation and penetration

- Incorporates many submodels for different features, events, and processes
- Used to evaluate model sensitivities, to focus research on reducing uncertainties for highest-impact parameters



Dry Storage Canister SCC: Current work





Current focuses:

1. Deposited salt characteristics/compositions
2. Mg-chloride brine evolution
3. Canister Deposition Field Demonstration
4. Corrosion in more realistic environments
   - Diurnal cycles in T/RH
   - Inert dust
   - Additional anions (e.g., NO_3, SO_4)
5. Pit-to-crack transition—environmental and material dependencies
6. CGR –moving towards atmospheric testing
7. Cold spray/coatings

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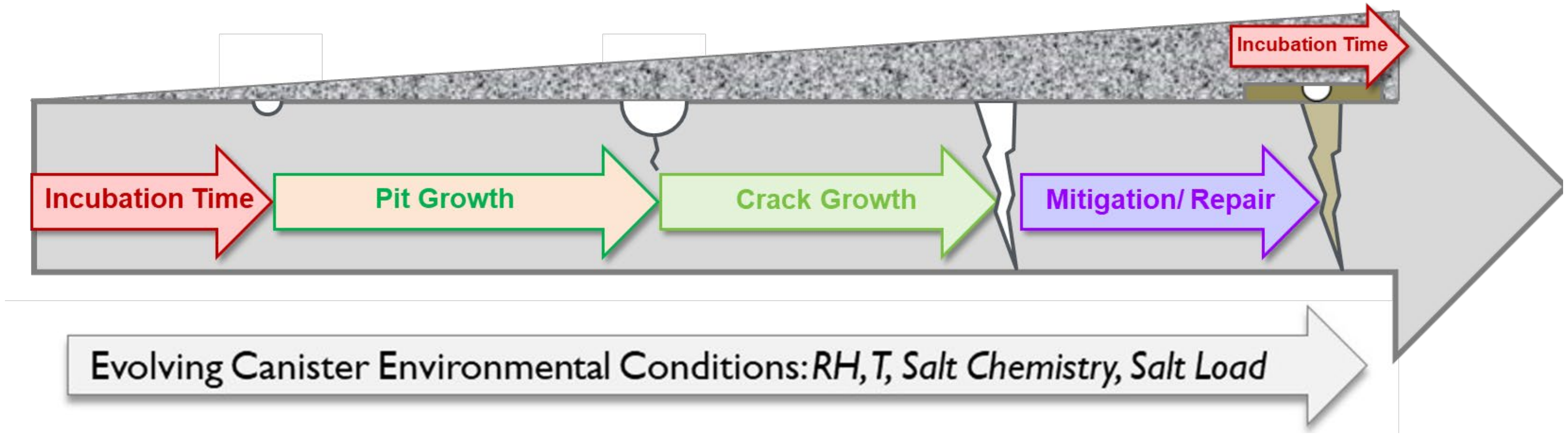

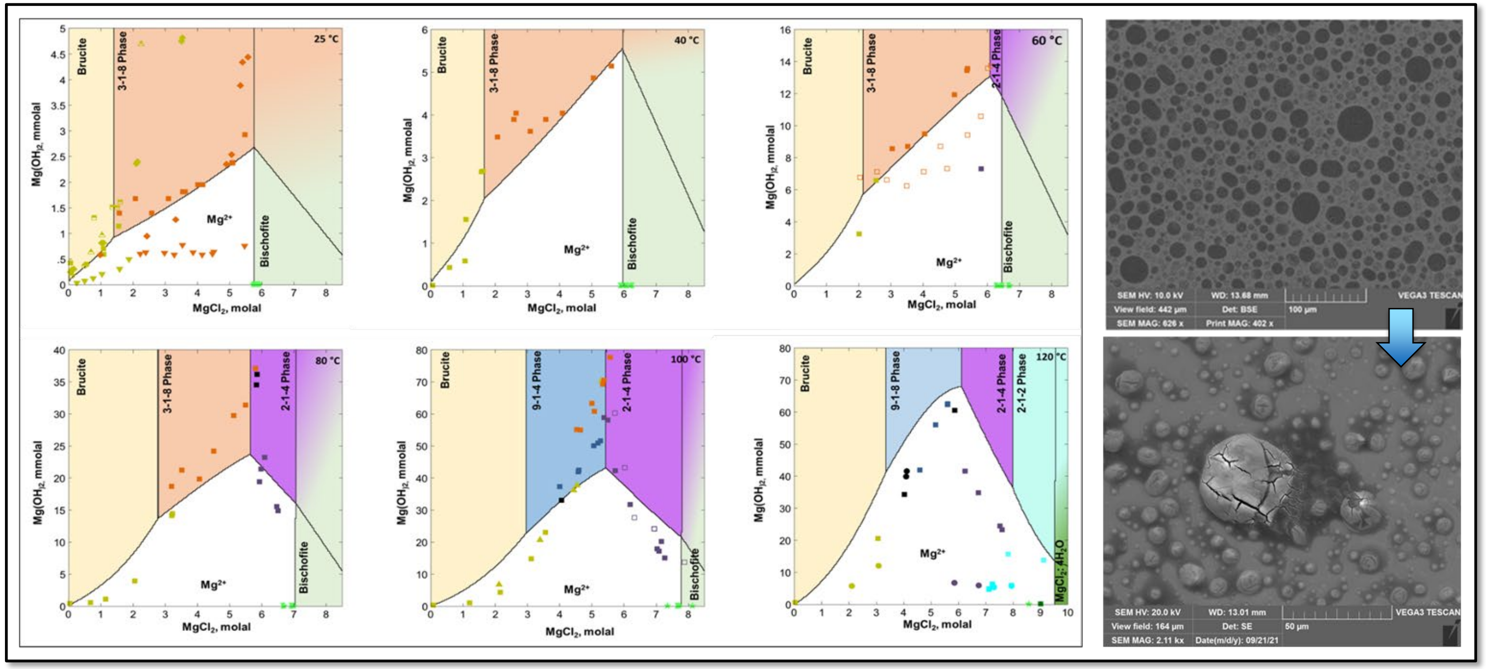

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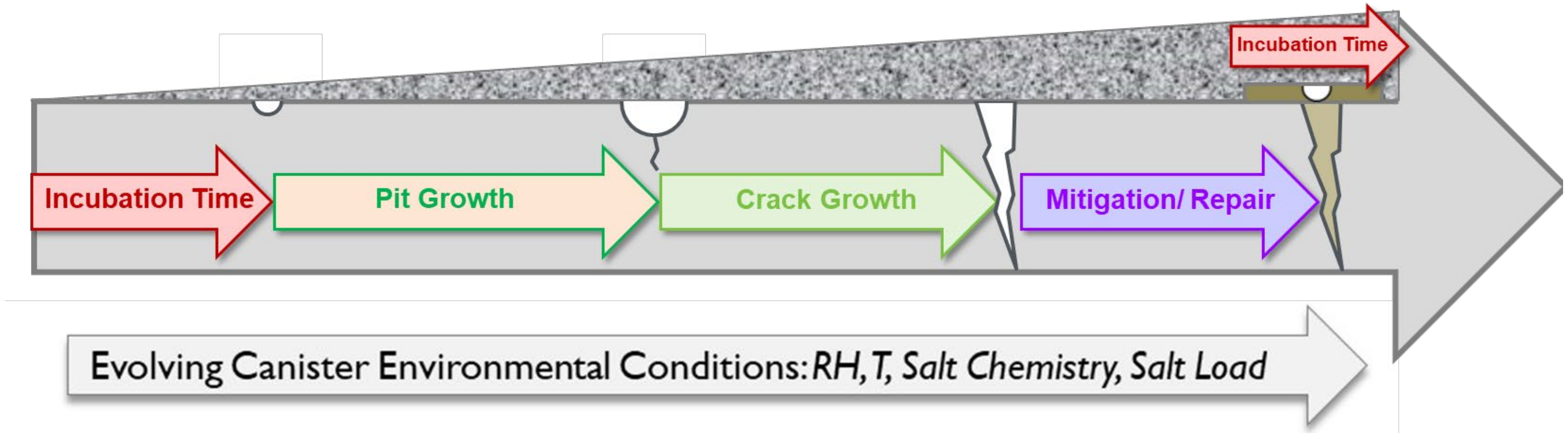

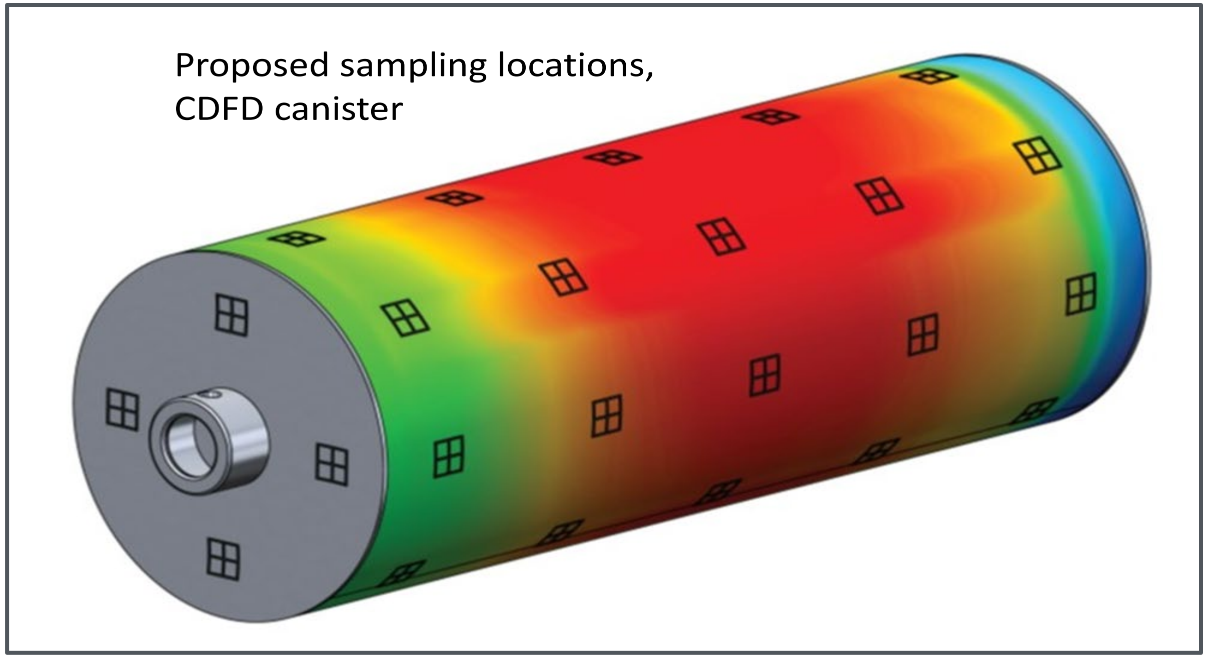

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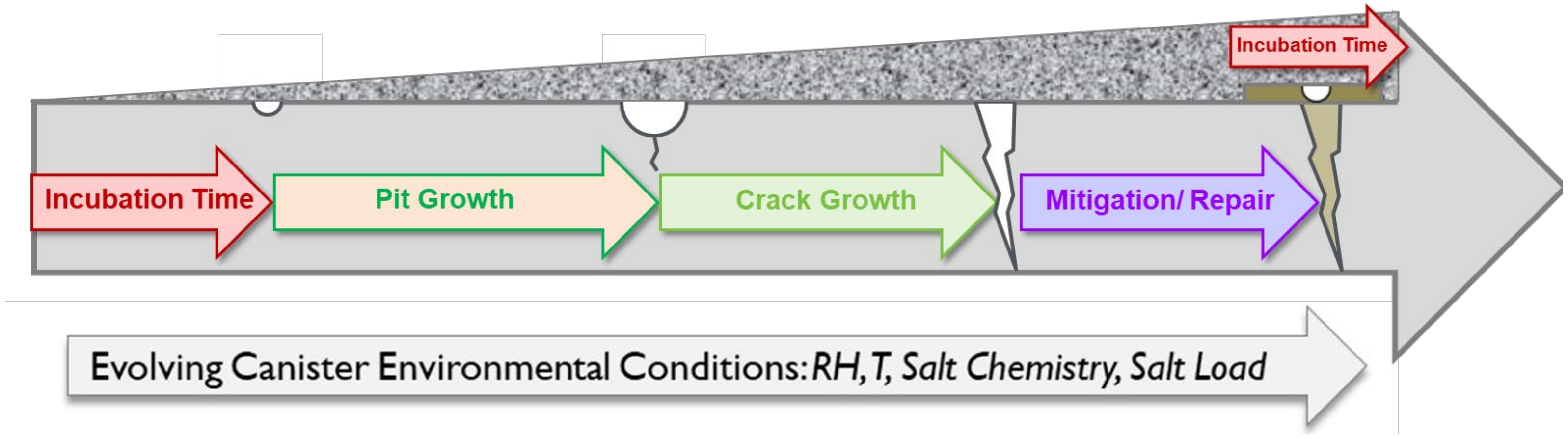
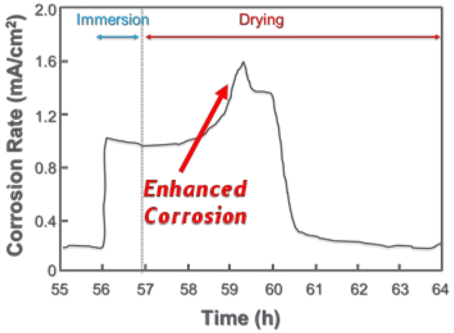


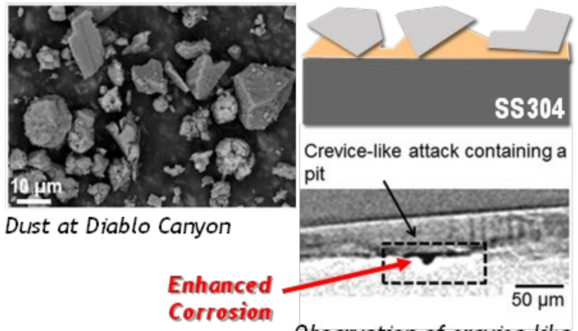
2 yr exposure underway

Diurnal Cycles



Sample preparation for 2 yr exposure

Dust/Precipitates

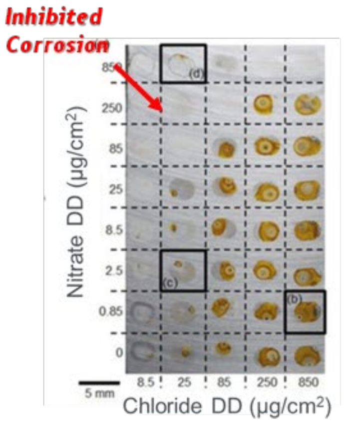


Dust at Diablo Canyon

Observation of crevice-like on a SS304 sample with surface deposits[2]

Electrochemical evaluation

Chemistry



304L plate with mixed droplets of $MgCl_2$ + $Mg(NO_3)_2$.[3]

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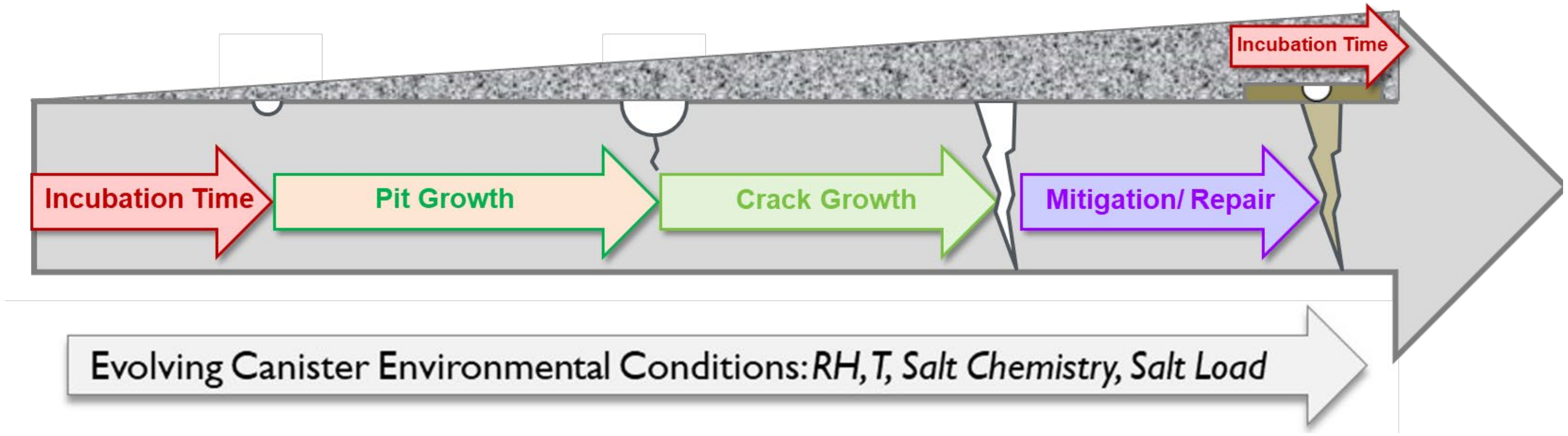
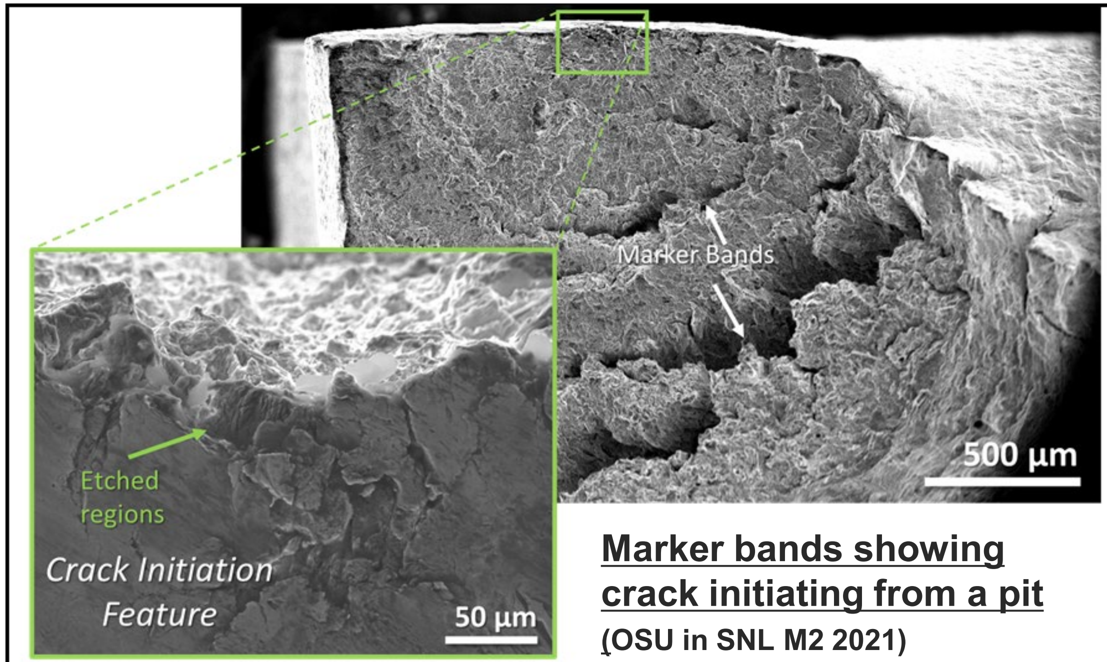
Dry Storage Canister SCC: Current work





Marker bands showing crack initiating from a pit
(OSU in SNL M2 2021)

Current focuses:

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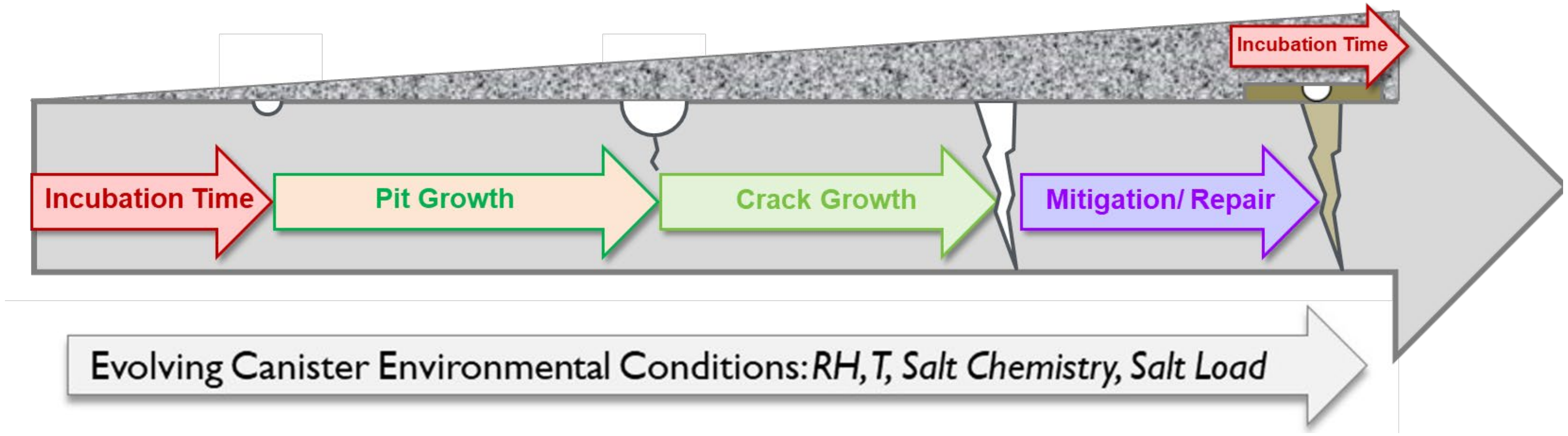
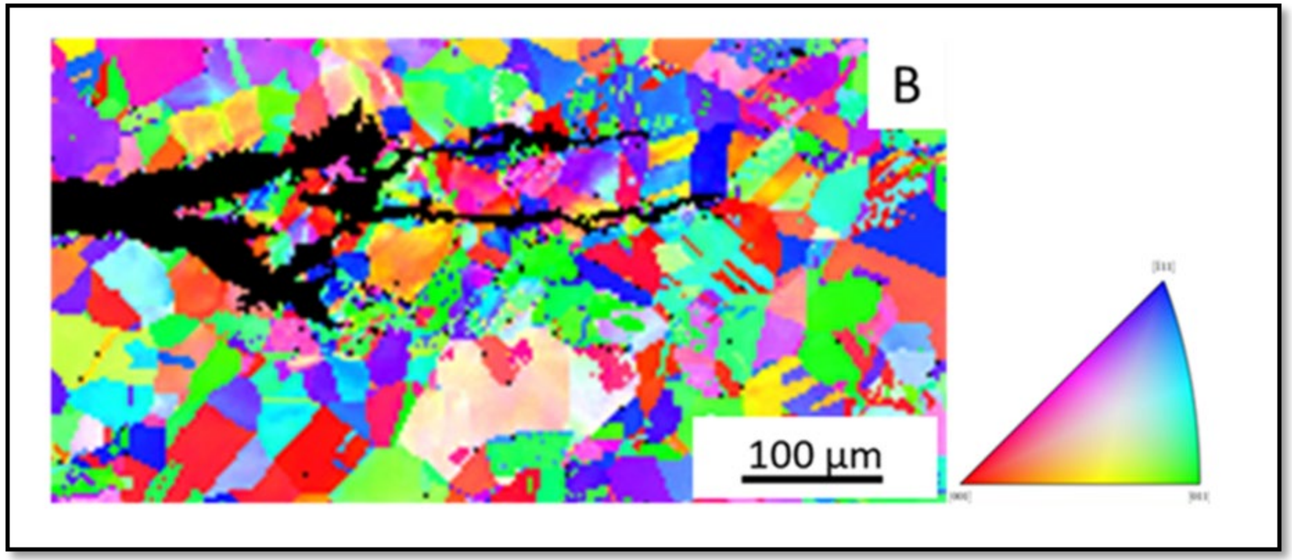
Dry Storage Canister SCC: Current work





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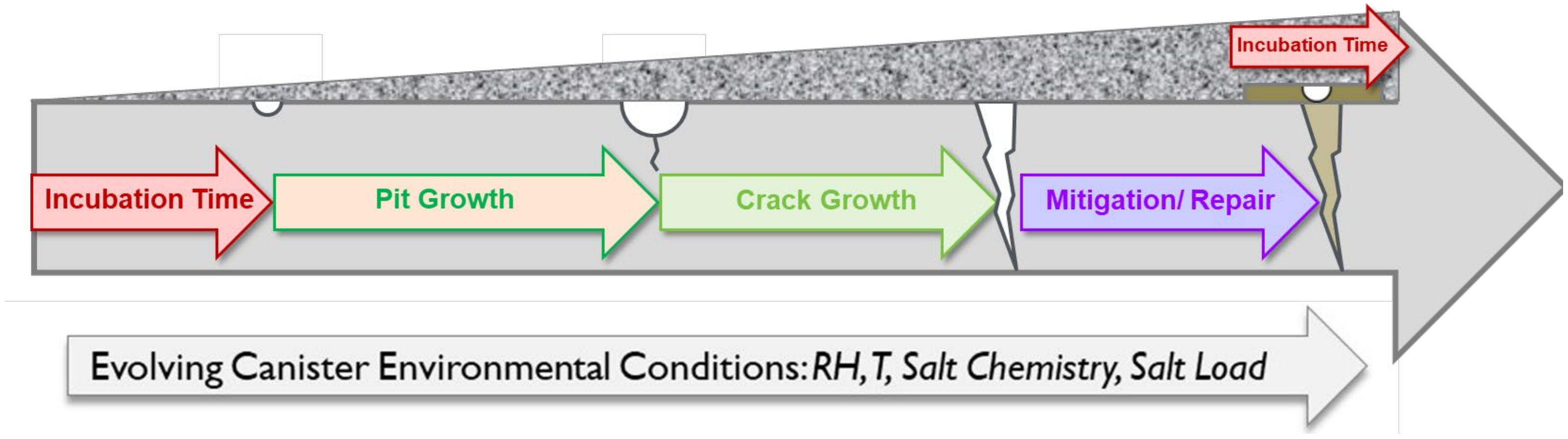

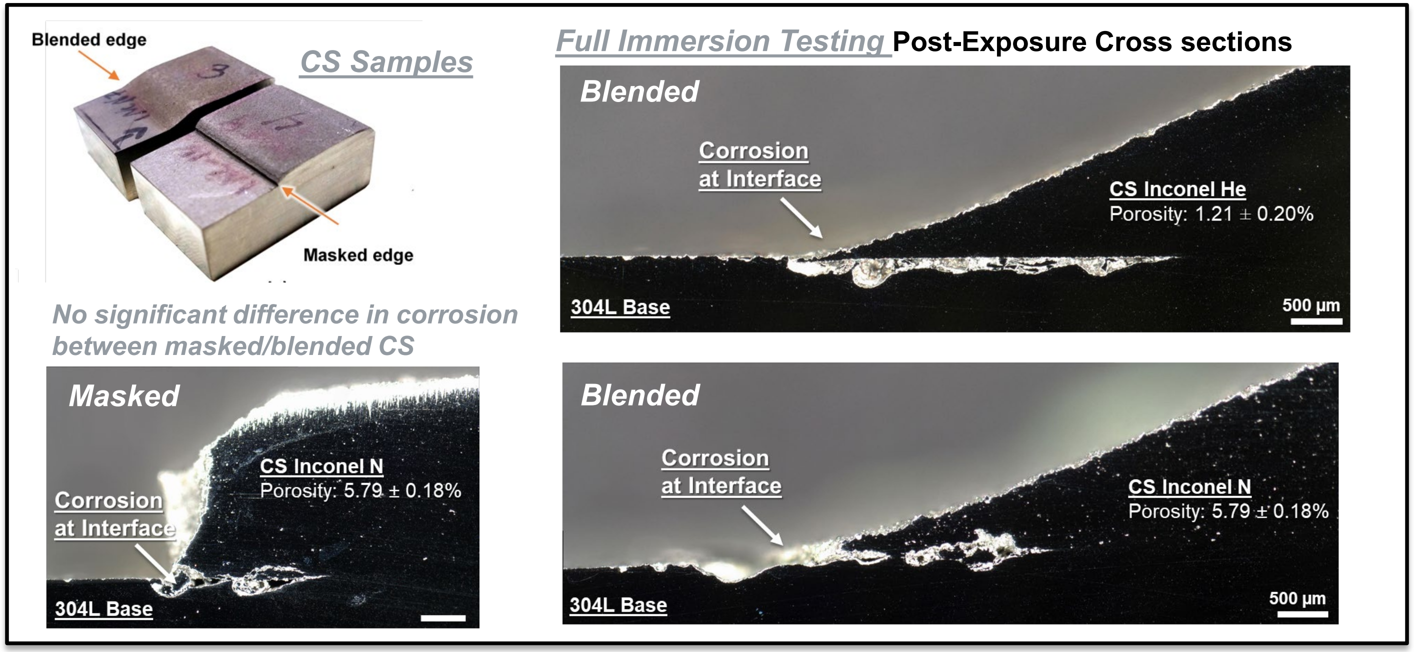

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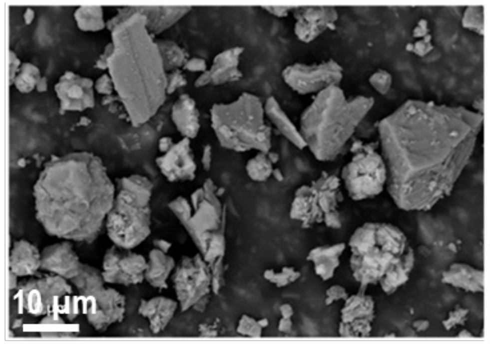
Defining the Canister Surface Environment

Site sampling and thermodynamic modeling

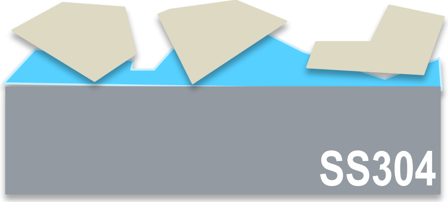
Why significant? → Influence on Corrosion:

Dust/Precipitates

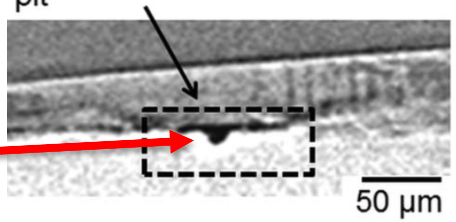


Dust at Diablo Canyon

Dust may act to spread water layer/ enhance corrosion



Crevice-like attack containing a pit

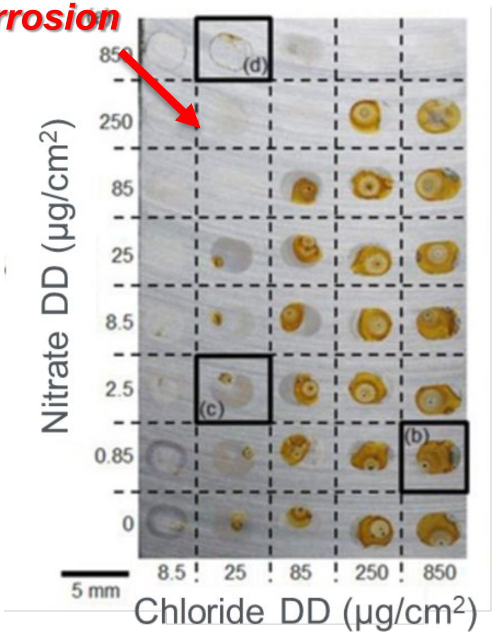


Enhanced Corrosion

Observation of crevice-like on a SS304 sample with surface deposits[2]

Chemistry

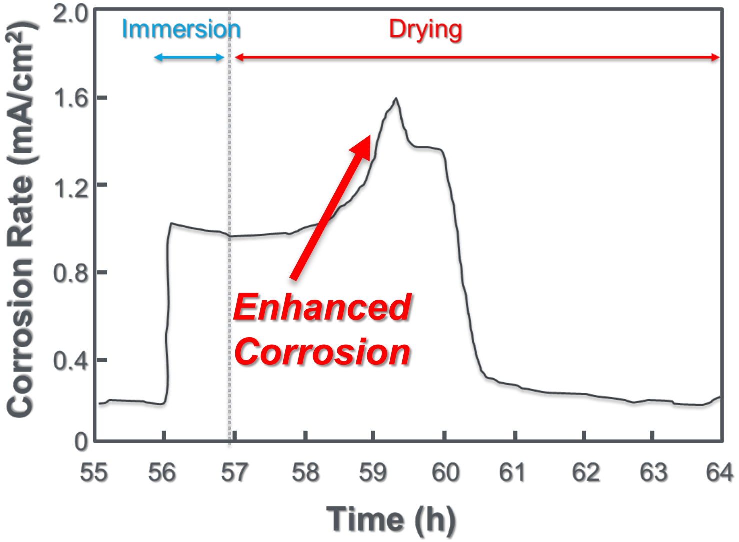
Inhibited Corrosion



304L plate with mixed droplets of $MgCl_2 + Mg(NO_3)_2$.[3]

Other chemistries may mitigate corrosion

Diurnal Cycles



Changes in the corrosion rate, i_{corr}, and potential during a wet/dry cycle of carbon steel.[1]

Corrosion rate increases upon initial drying (highly concentrated brine)

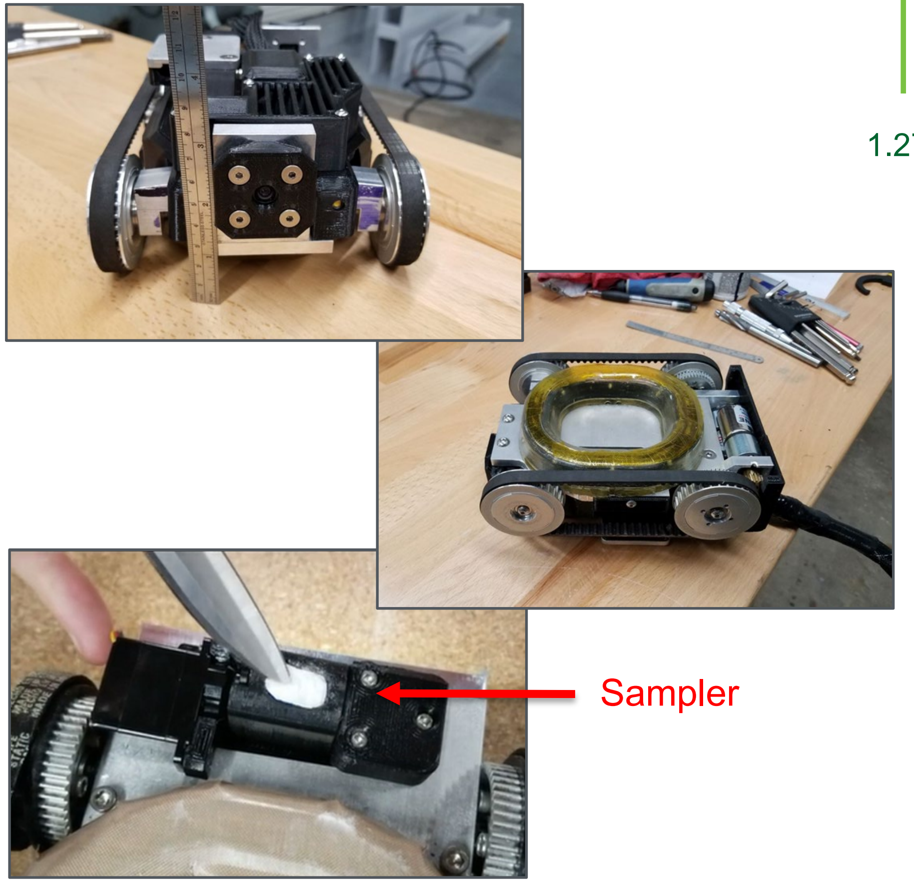
[1] Nishikata, A., Yamashita, Y., Katayama, H., Tsuru, T., Tanabe, K., & Mabuchi, H. (1995). *Corrosion science*, *37*(12), 2059-2069.
[2] Guo, L., Mi, N., Mohammed-Ali, H., Ghahari, M., Du Plessis, A., Cook, A., ... & Davenport, A. J. (2019).
[3] Cook, A. J., Padovani, C., & Davenport, A. J. (2017). *Journal of The Electrochemical Society*, *164*(4), C148.

Canister Surface Environment

ISFSI Site Sampling – Orano Sites "A" and "B"

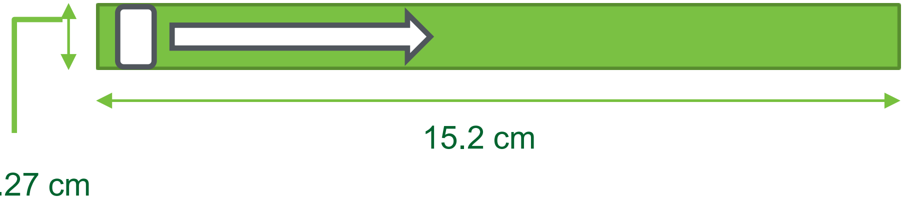
First dust and salt data available from inland sites.

Samples were collected using the RTT vacuum crawler.

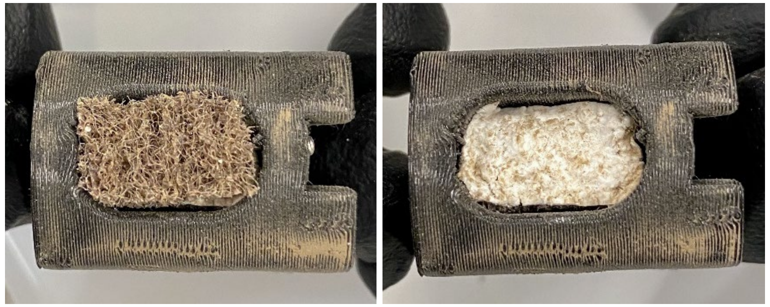


Sampler

Sample was collected by moving crawler 6". Sampled area = 19.35 cm^2

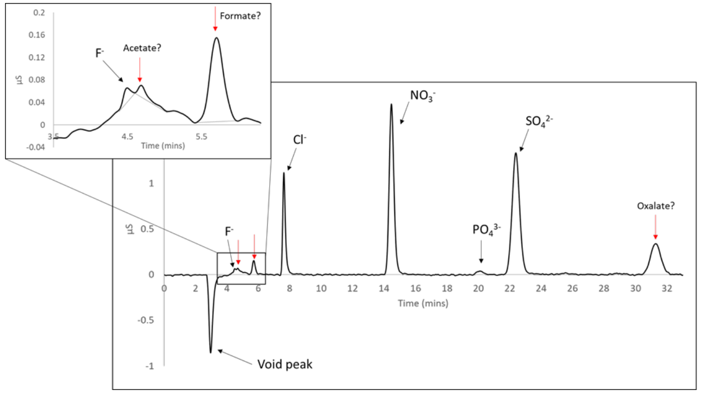


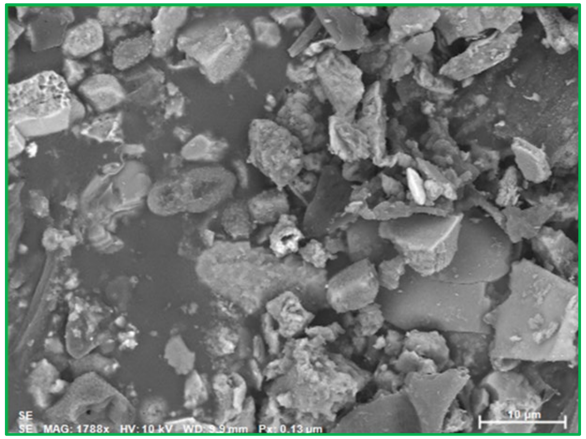
12 o'clock position – front third



2 o'clock position

Chemical analysis by IC and TIC analyzer/SEM analysis



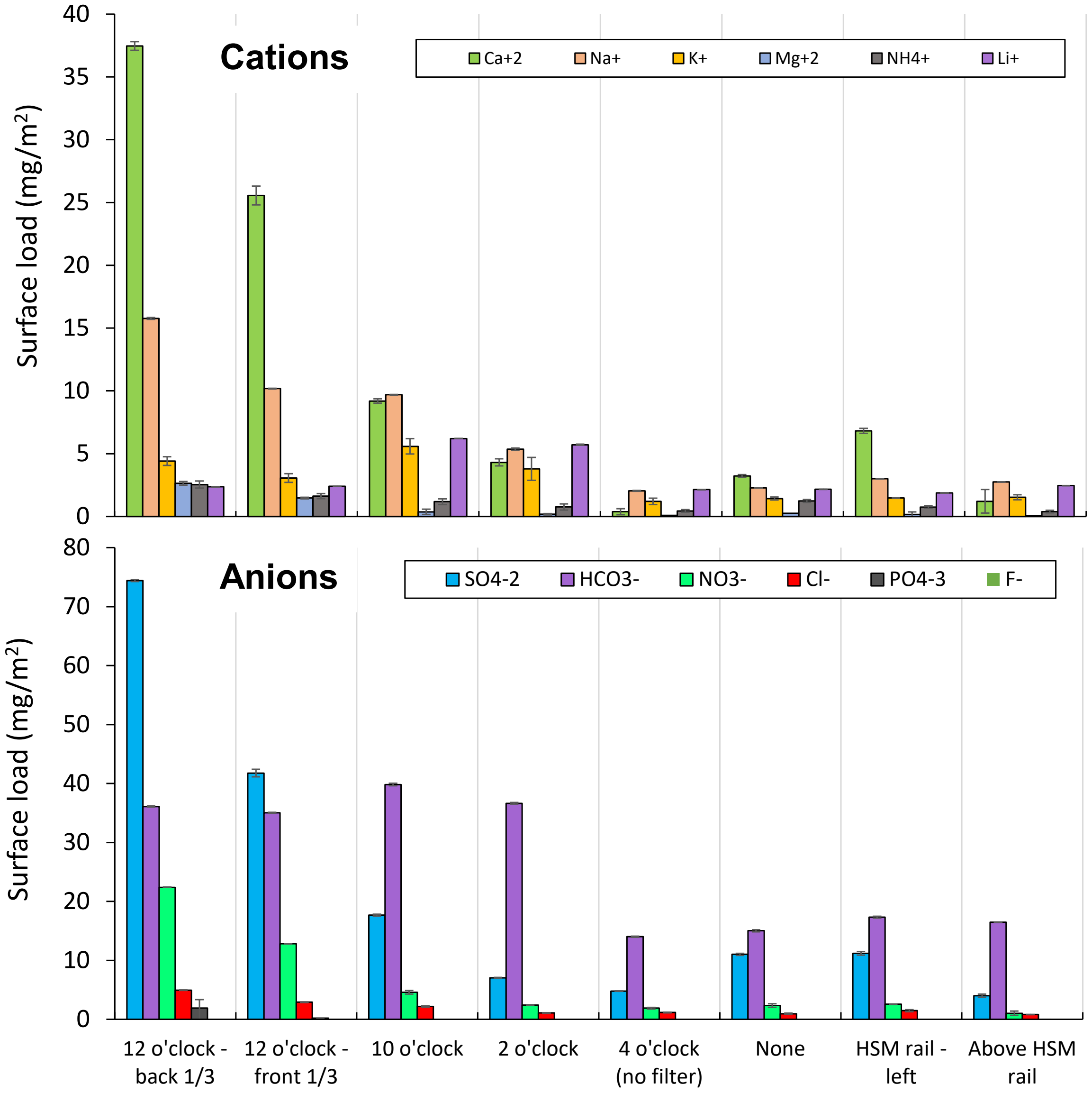


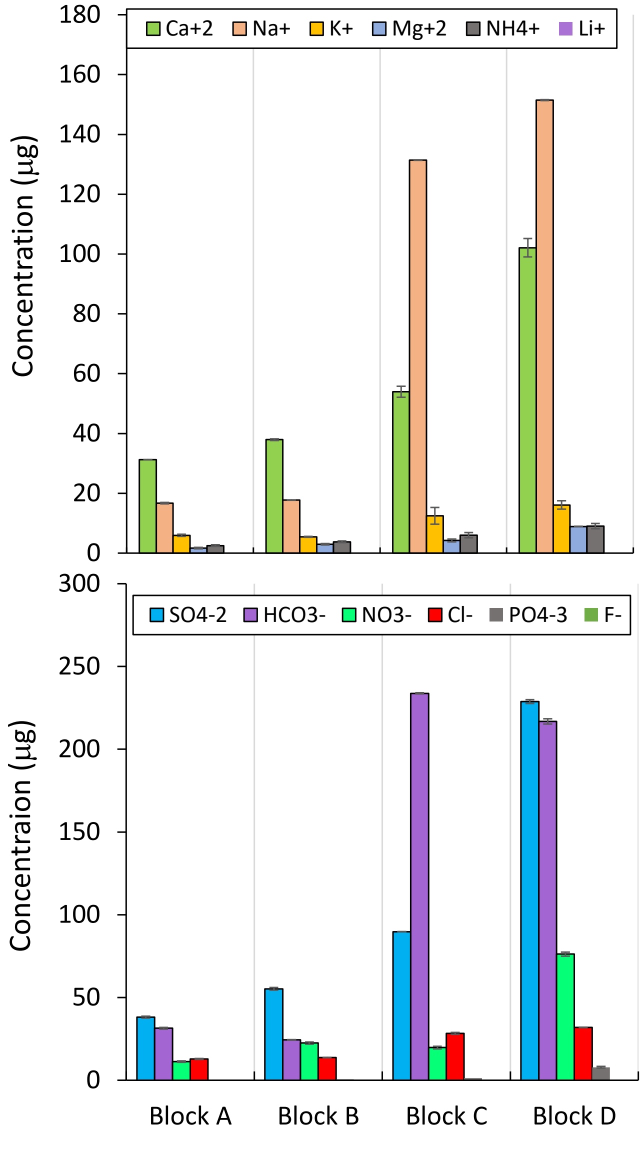
Canister Surface Environment
ISFSI Site Sampling – Orano Sites “A” and “B”

Site “A”

Chemistry:

- Li leached from the Scotchbrite® pads. Not present in dust!
- Cations: $Ca^{+2} >> Na^{+} > K^{+} > NH_4^{+} > Mg^{+2}$
- Anions: $SO_4^{-2} > HCO_3^{-} >> NO_3^{-} > Cl^{-}$
- Chloride concentrations all < 5 mg/m²





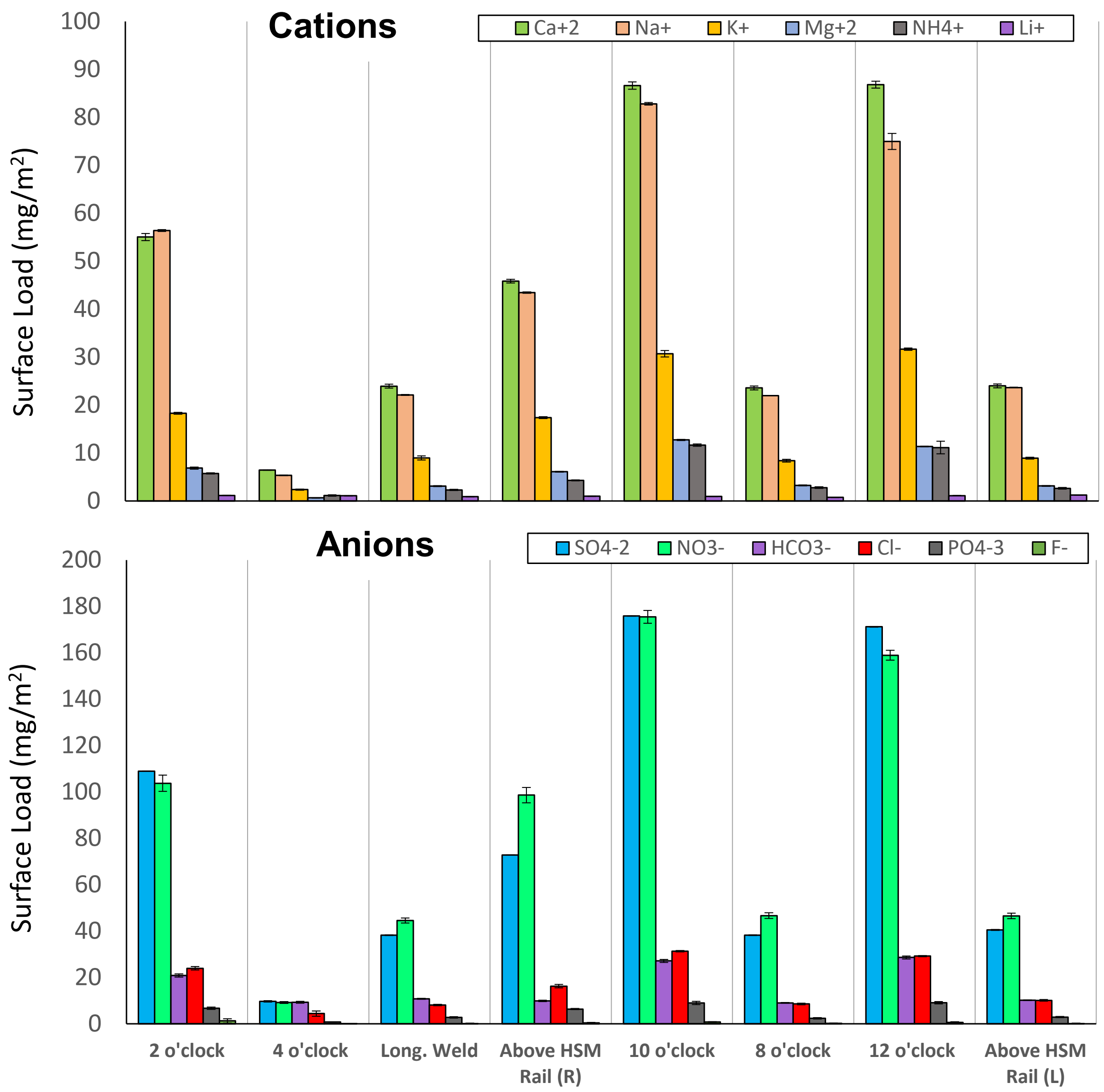
Canister Surface Environment

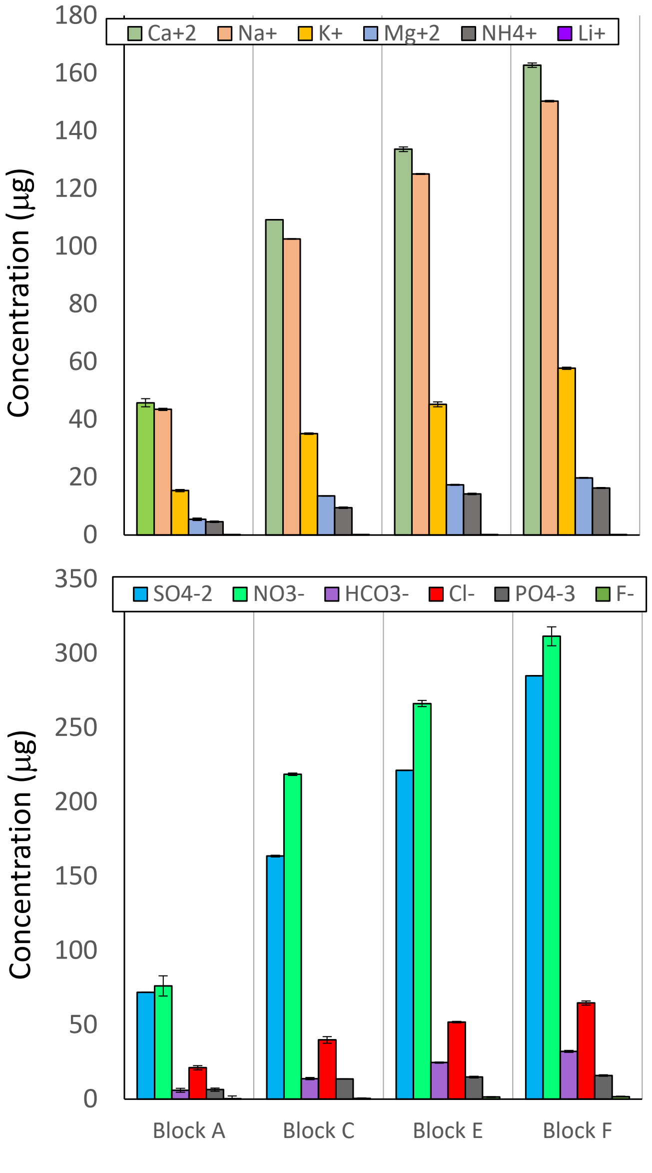
ISFSI Site Sampling – Orano Sites “A” and “B”

Site “B”

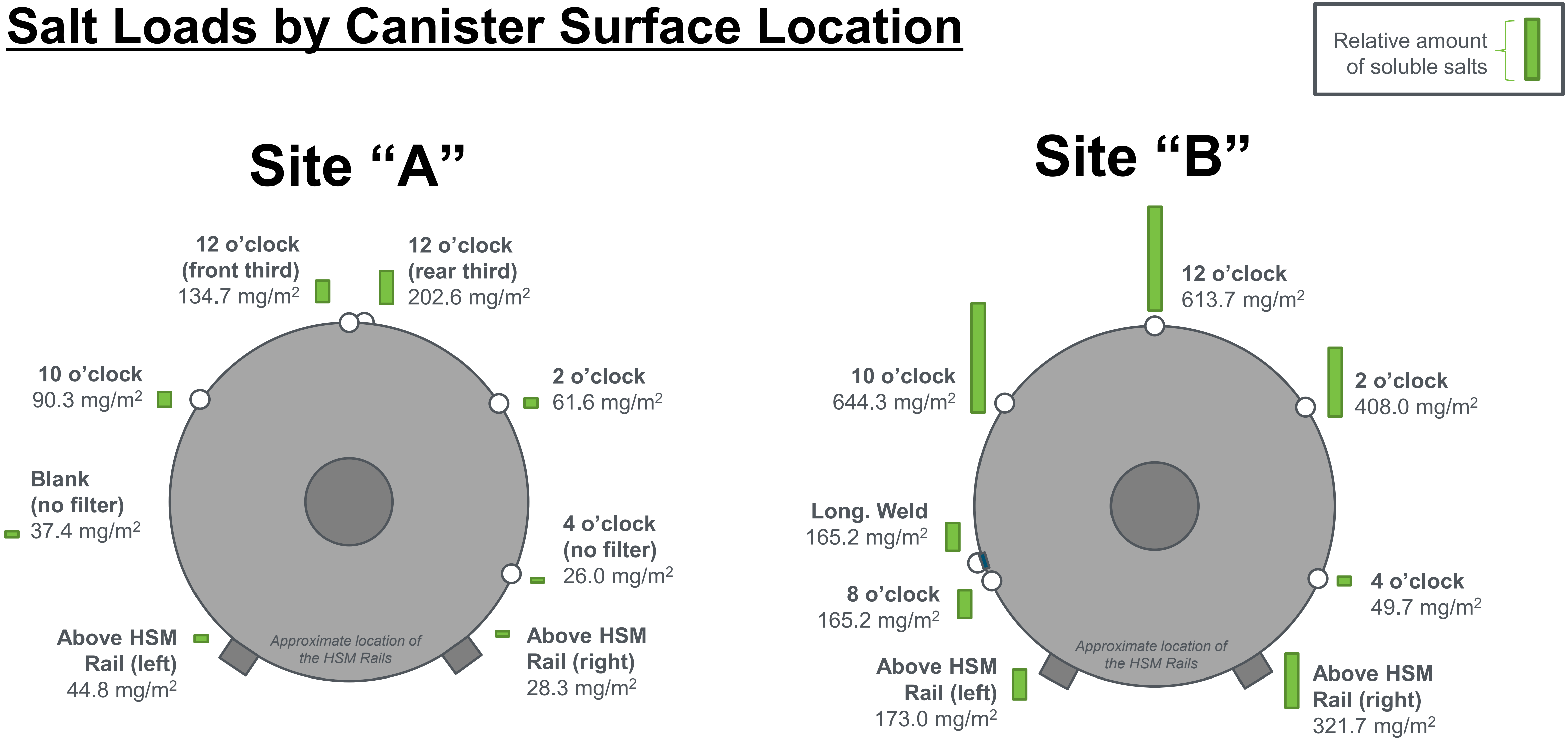
Chemistry:

- Li leached from the Scotchbrite® pads. Not present in dust!
- Cations: $Ca^{+2} \approx Na^{+} > K^{+} > Mg^{+2} > NH_4^{+}$
- Anions: $SO_4^{-2} > NO_3^{-} >> Cl^{-} > HCO_3^{-}$
- Chloride concentrations all $< 30\ mg/m^2$





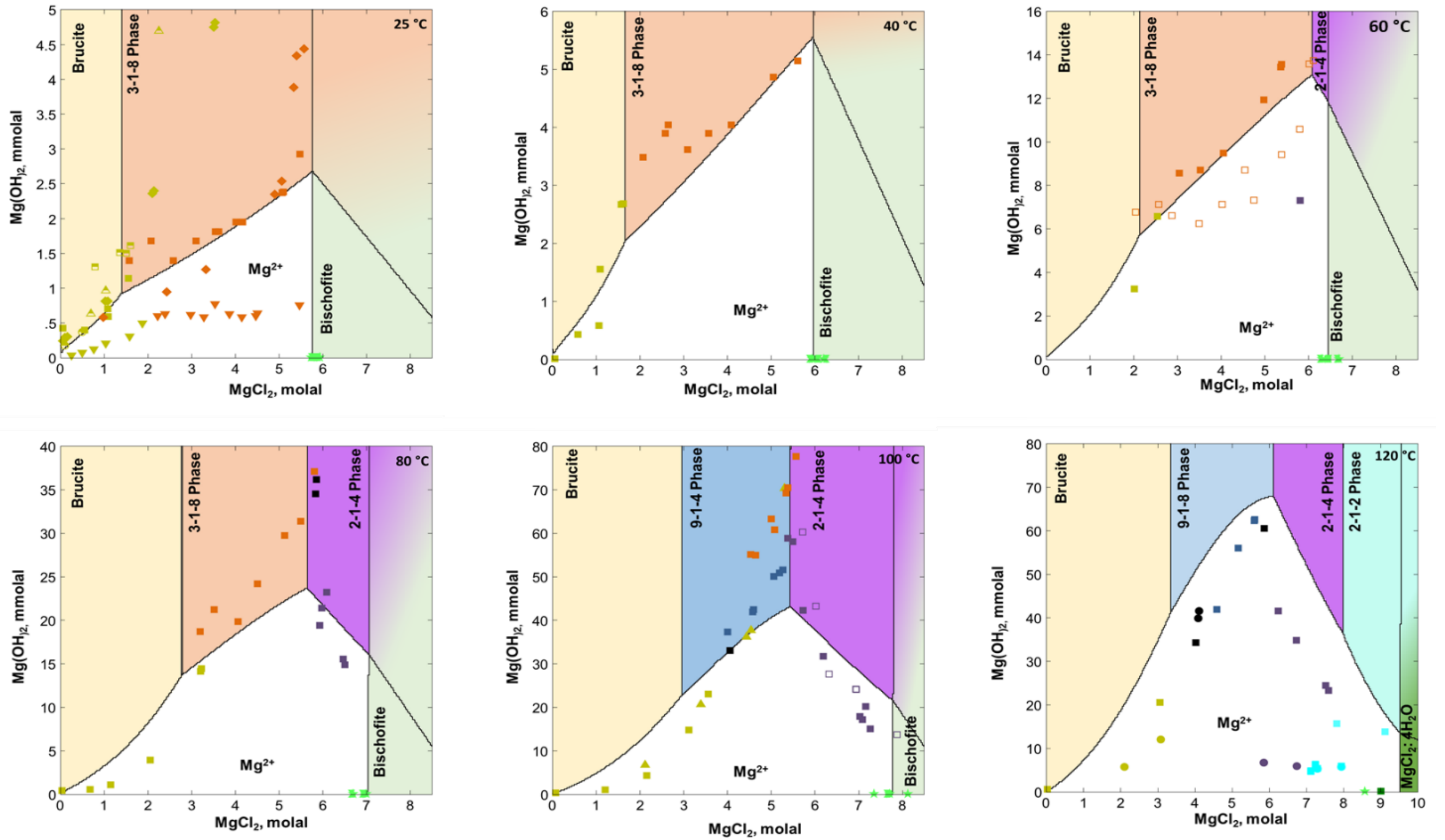
Salt Loads by Canister Surface Location



Canister Surface Environment

Mg-Chloride Brine Stability

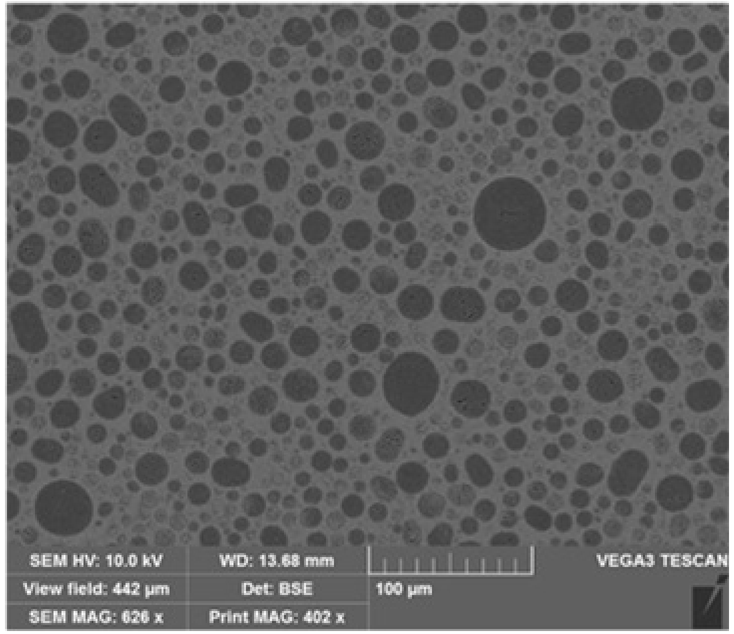
Thermodynamic model for Mg-Cl-(OH)-H_2O system: Consistent thermodynamic data is necessary to model $MgCl_2$ brine stability in at different T, RH, P_{HCl}



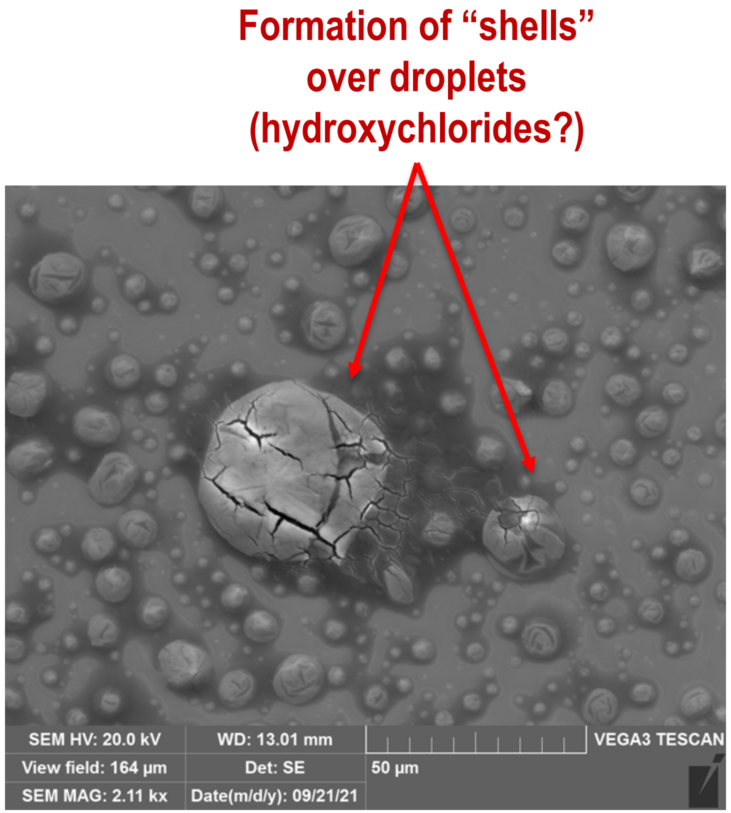
Draft of journal article is in development

$MgCl_2$ brine degassing experiment

- Exposed at 48ºC, 40% RH (near upper T for deliquescence on a canister)
- Very small dispersed droplets (high surface area to increase extent of reaction)
- High air flow (9 L/min)
- Exposed for 2, 4, 8, 16 weeks



Exposure



Why important?

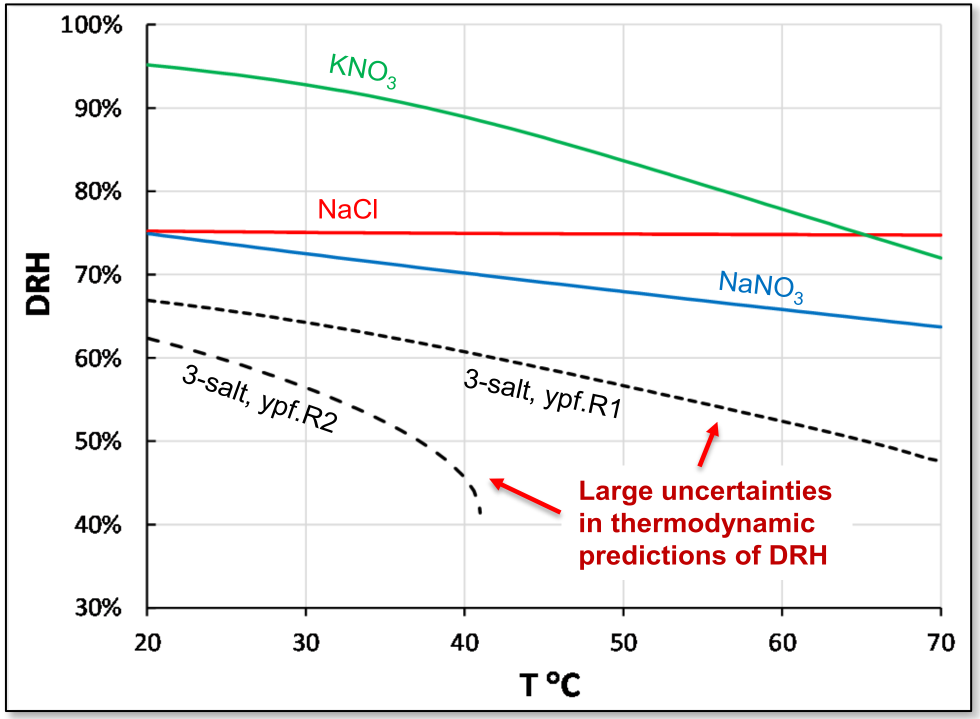
Mg-chloride brine stability may impact:

- Timing of corrosion initiation on canisters
- Brine volumes and corrosion extent/ evolution
- Corrosion morphology
- Interpretation of experimental results and extrapolation to field conditions

Canister Surface Environment
Deliquescence of Salt Mixtures

Evaluate deliquescence of multi-component nitrate-containing salt assemblages

- *Deliquescence RH (DRH) for nitrate-containing salt assemblages is poorly predicted by thermodynamic models*
- We will measure deliquescence RH (DRH) of typical salt mixtures.
- Why? Accurate DRH provides improved prediction of temperature and timing of brine formation/potential corrosion initiation.
  - Define range of conditions for laboratory testing
  - Assess timing of brine formation at individual sites.
- Measure deliquescence of salts in dusts collected from actual sites?
  - Methodology: quartz crystal microbalance (QCM) and/or other instruments

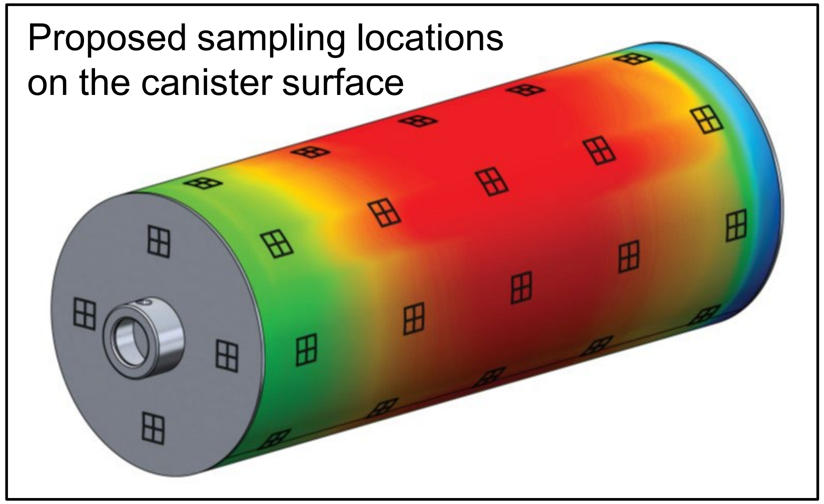


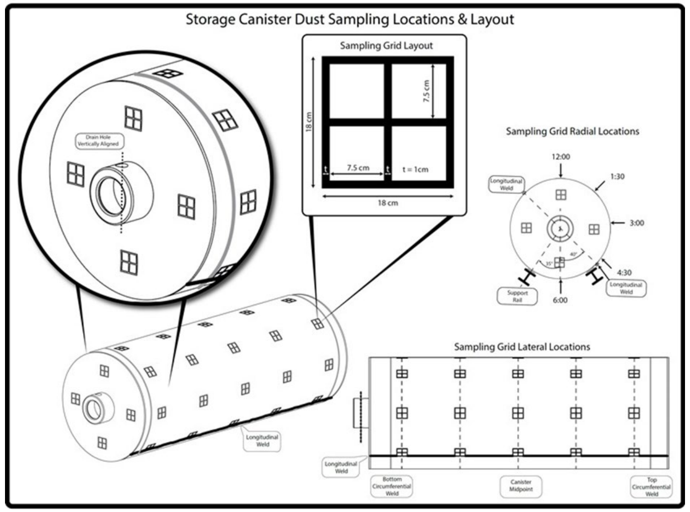
Canister Surface Environment

Canister Deposition Field Demonstration (CDFD)

Principal goal: Evaluate dust/salt deposition on canister surfaces under realistic storage conditions, in part to parameterize and validate PNNL dust deposition model

- Canisters: 32PTH2 NUHOMS (Orano/TN)
- Vaults: horizontal storage modules (HSMs)
- Heater rods used to simulate fuel heat loads. Heat loads:
  - 0 kW
  - 10 kW
  - 40 kW
- Duration: up to 10 years





Canister-Relevant Environments for Laboratory Corrosion Testing

Dust Exposures

- Atmospheric Exposure – 3 conditions

Chemistry

- Immersed scoping measurements

Cyclic Exposures

- Atmospheric Exposure – diurnal cycle

Canister-Relevant Environments for Laboratory Corrosion Testing

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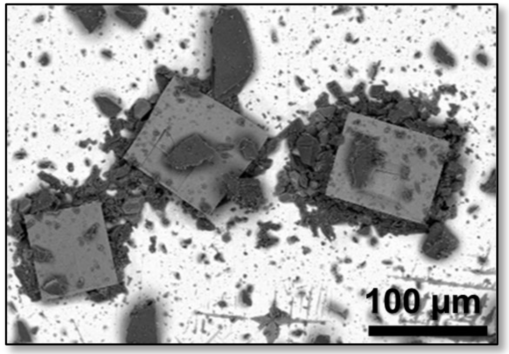
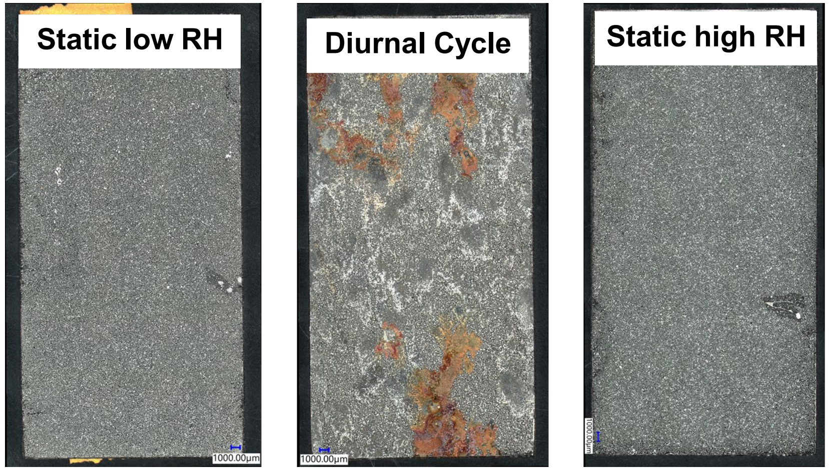
74 µm dust deposited with seawater



1 month exposure – 304 coupons with seawater & dust



Canister-Relevant Environments for Laboratory Corrosion Testing

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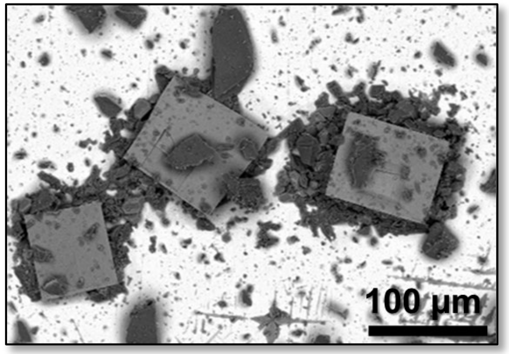
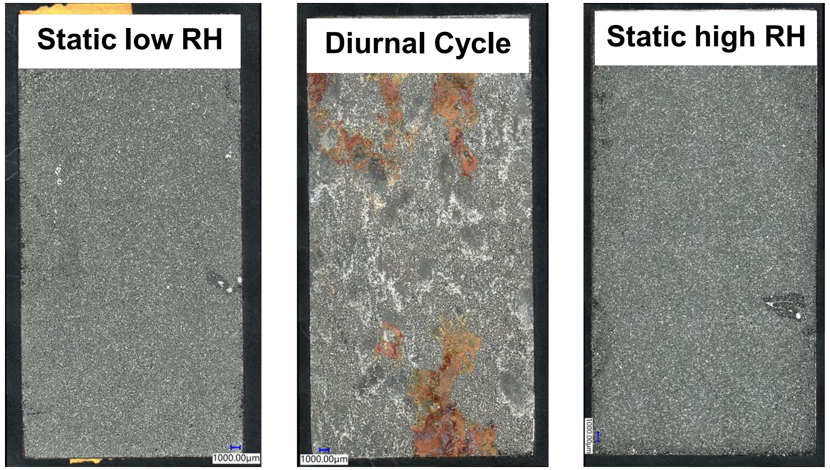
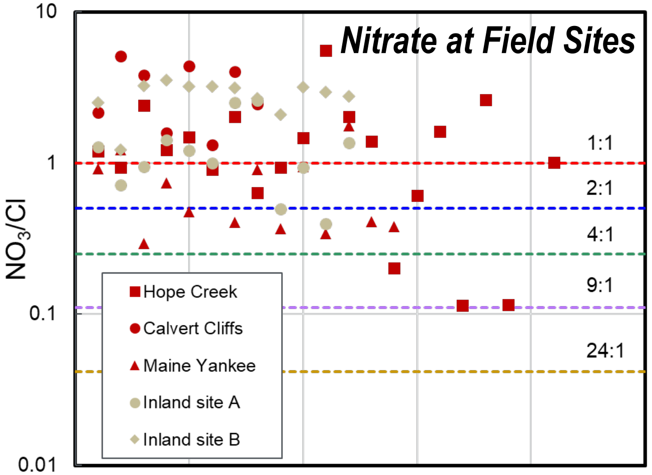


1 month exposure – 304 coupons with seawater & dust

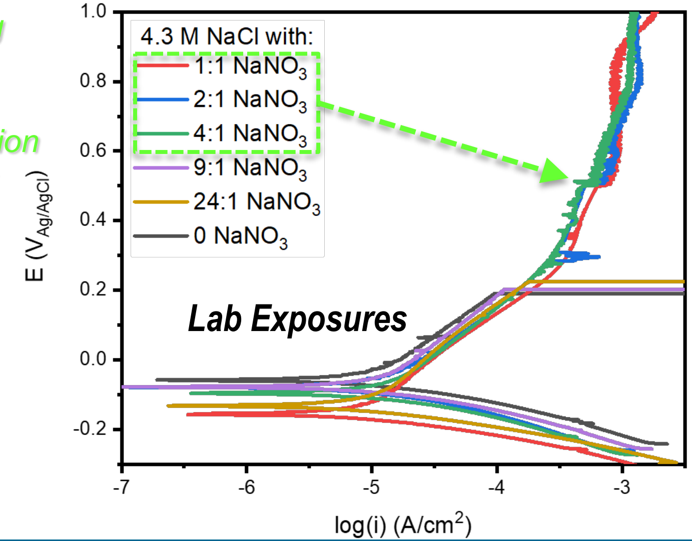


Chemistry

- Immersed scoping measurements



Passivating effects of Nitrates: Concentration Dependent



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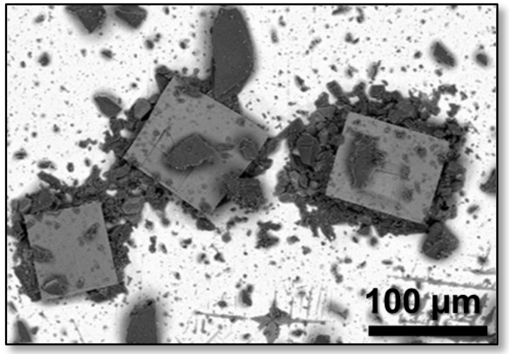
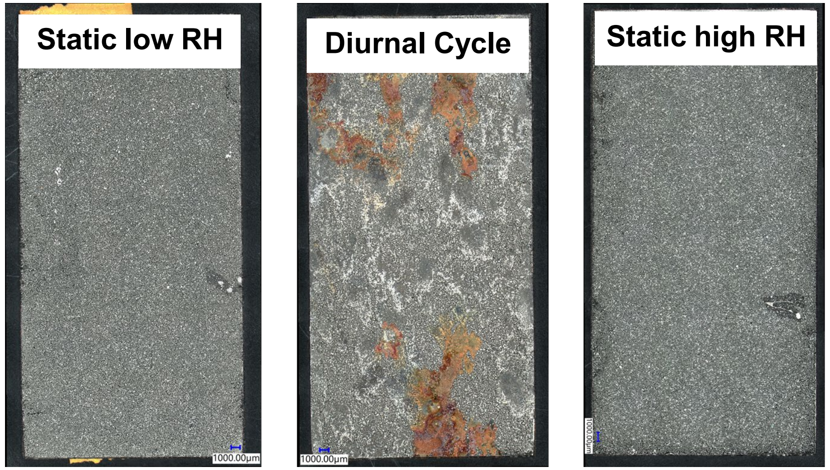
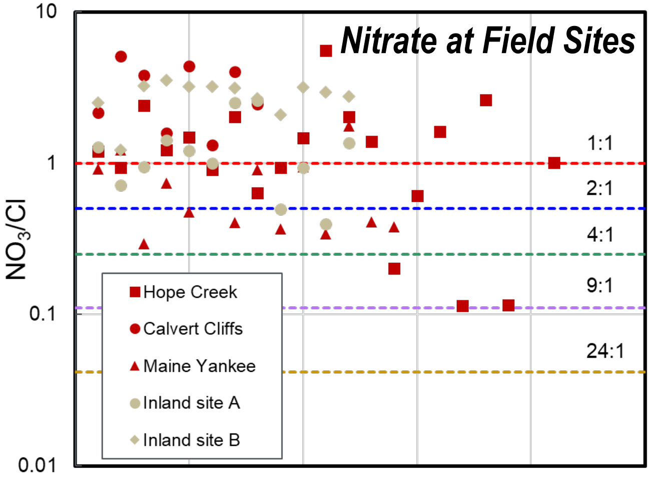


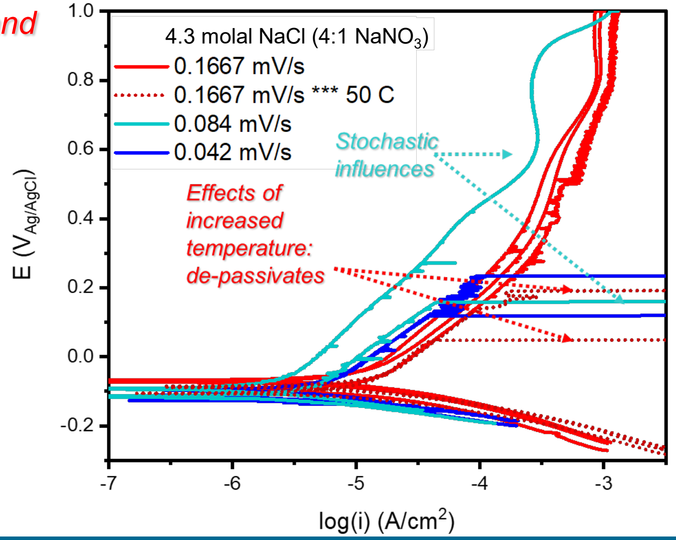
1 month exposure – 304 coupons with seawater & dust



Chemistry
- Immersed scoping measurements



Stochastic and variable dependent



Cyclic Exposures
- Atmospheric Exposure – diurnal cycle

Canister-Relevant Environments for Laboratory Corrosion Testing

Dust Exposures

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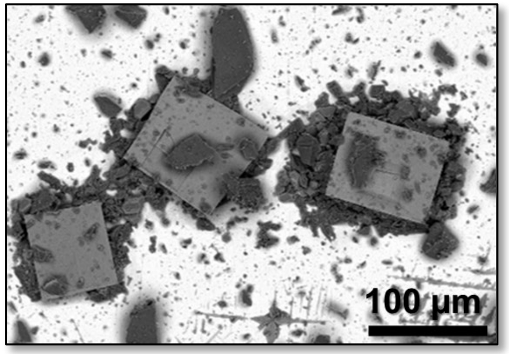
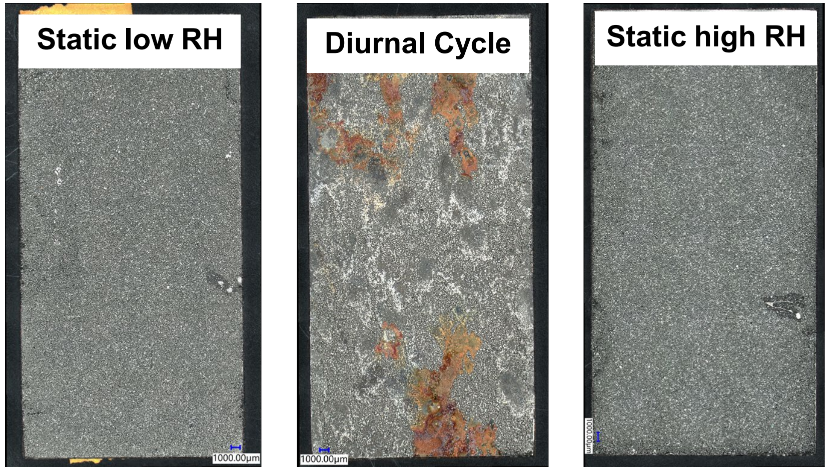
74 µm dust deposited with seawater

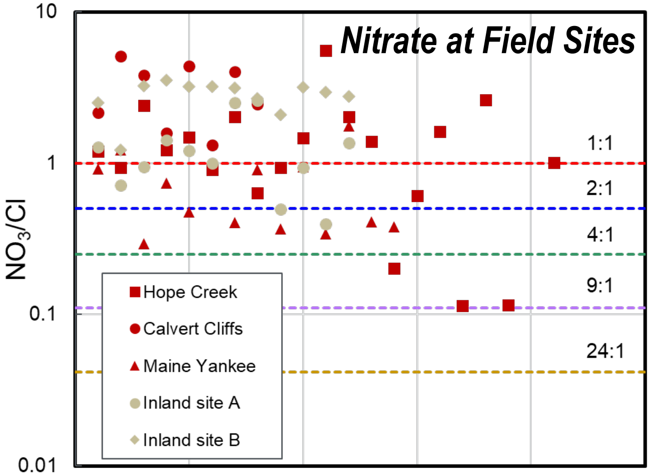


1 month exposure – 304 coupons with seawater & dust

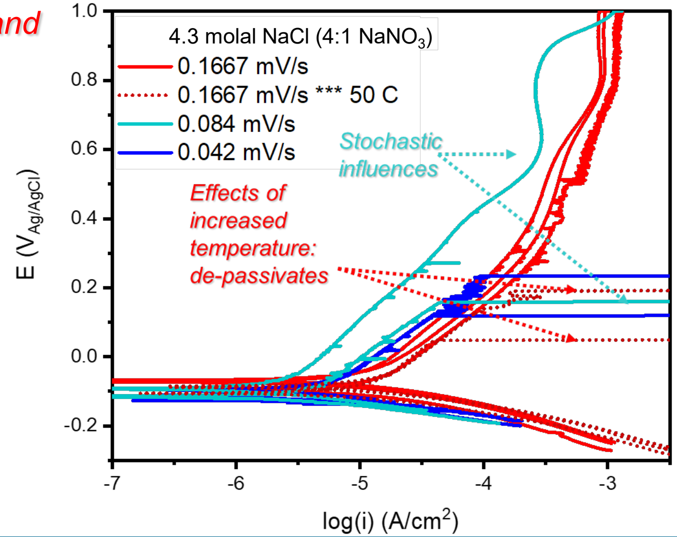


Chemistry

- Immersed scoping measurements



Stochastic and variable dependent



Cyclic Exposures

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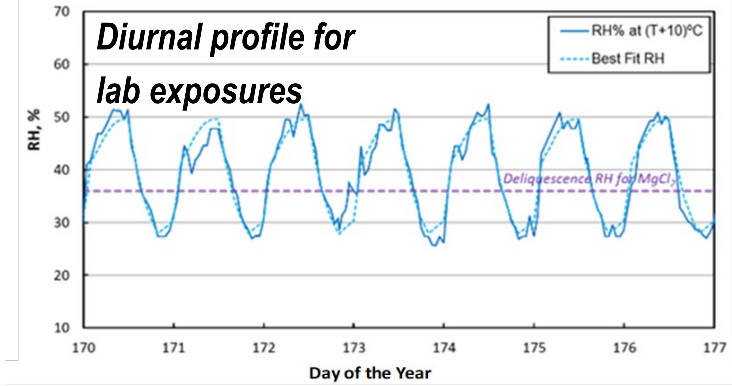
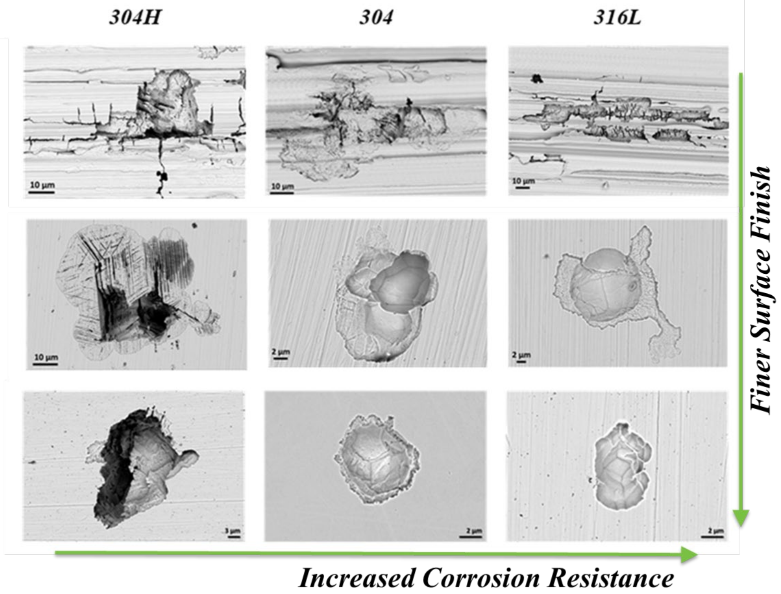
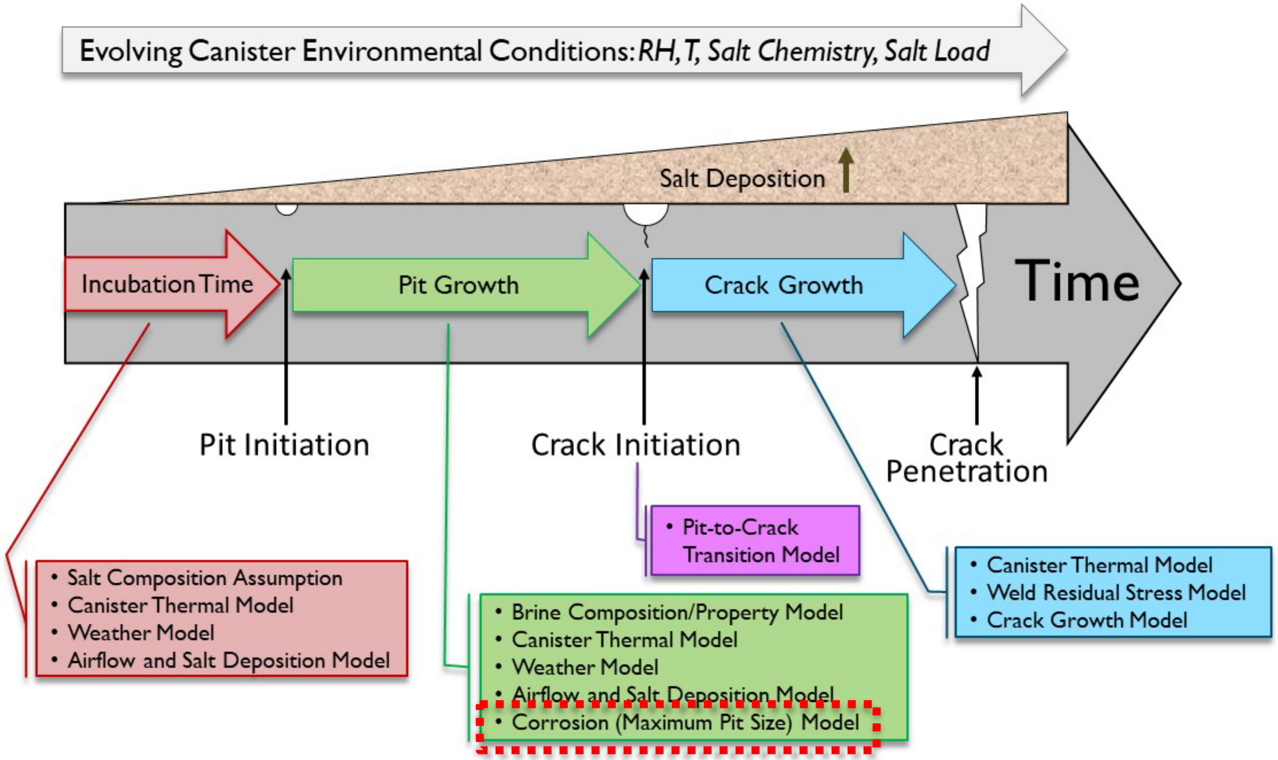


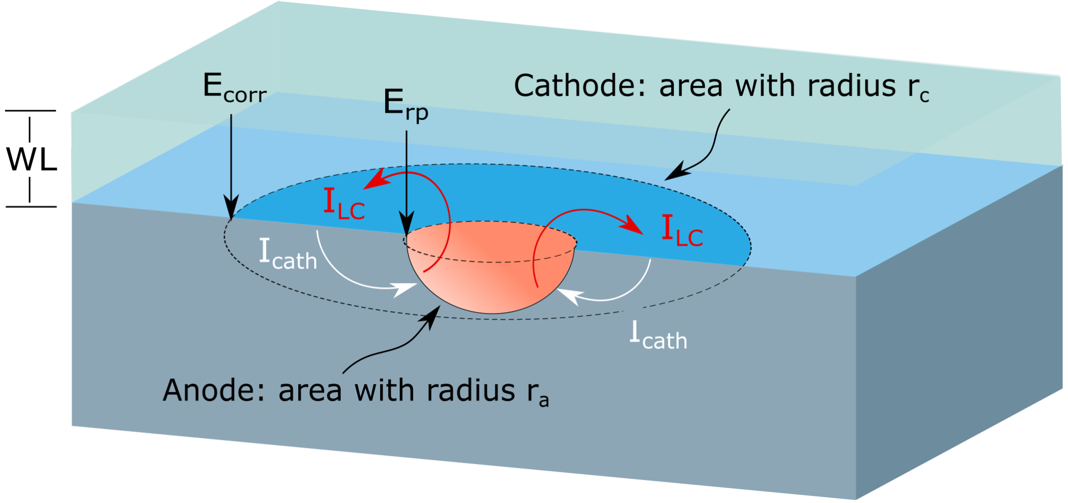
Post Exposure Corrosion Damage



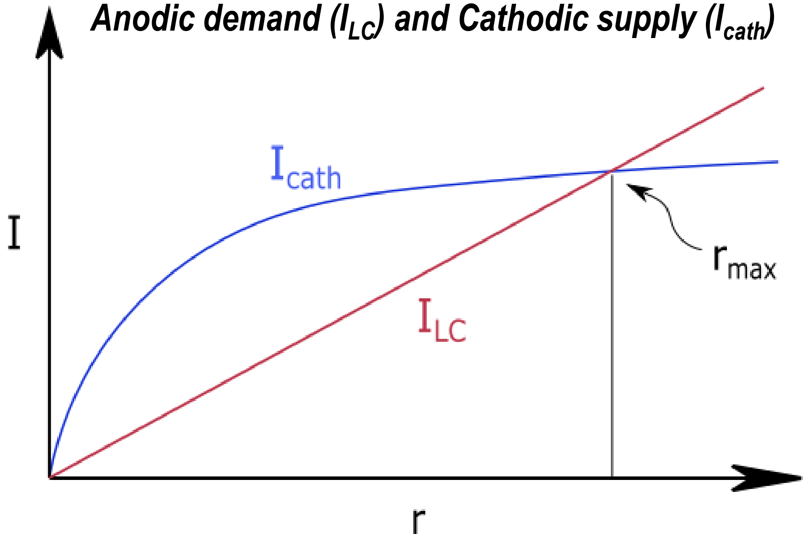
Prediction of Maximum Pit Sizes



Pitting under thin electrolyte layer



Anodic demand (I_{LC}) and Cathodic supply (I_{cath})



- Pit (anode) must be supported by cathodic reduction reaction forming an inherent galvanic couple
- In finite water layers, cathode limited by ohmic drop
  - Finite cathode → Finite anode → Finite pit

Chen, Z. Y., & Kelly, R. G. (2009). Computational modeling of bounding conditions for pit size on stainless steel in atmospheric environments. *Journal of the Electrochemical Society*, *157*(2), C69.

Prediction of Maximum Pit Sizes

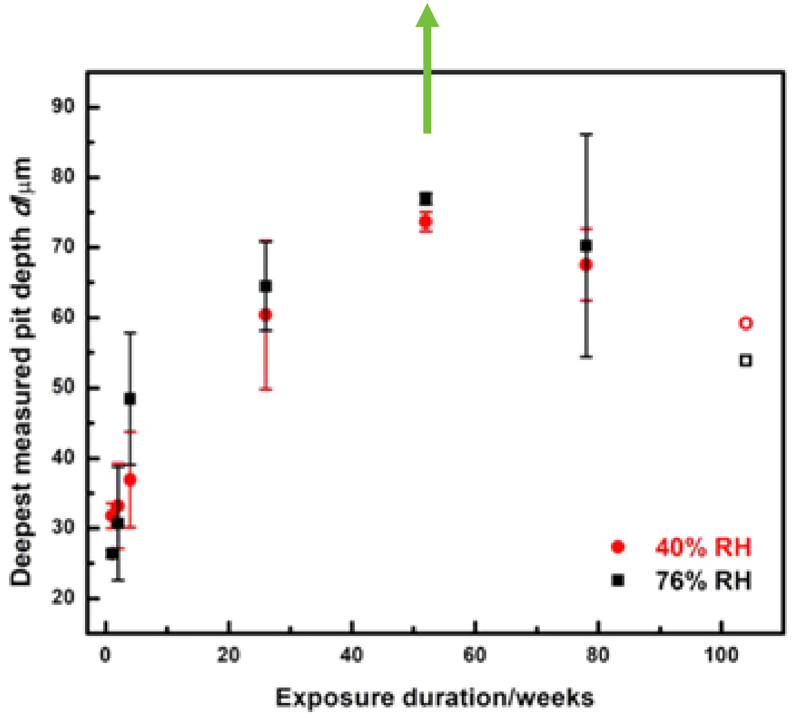
Comparison to long-term pitting exposures

<u>Conservative estimates of the maximum pit</u>

Roughly 1.5 x larger estimate

76 % Max Pit ~ 230 µm

40 % Max Pit ~ 110 µm



Srinivasan, J., Weirich, T. D., Marino, G. A., Annerino, A. R., Taylor, J. M., Noell, P. J., ... & Schindelholz, E. J. (2021). Long-Term Effects of Humidity on Stainless Steel Pitting in Sea Salt Exposures. *Journal of The Electrochemical Society*, *168*(2), 021501.

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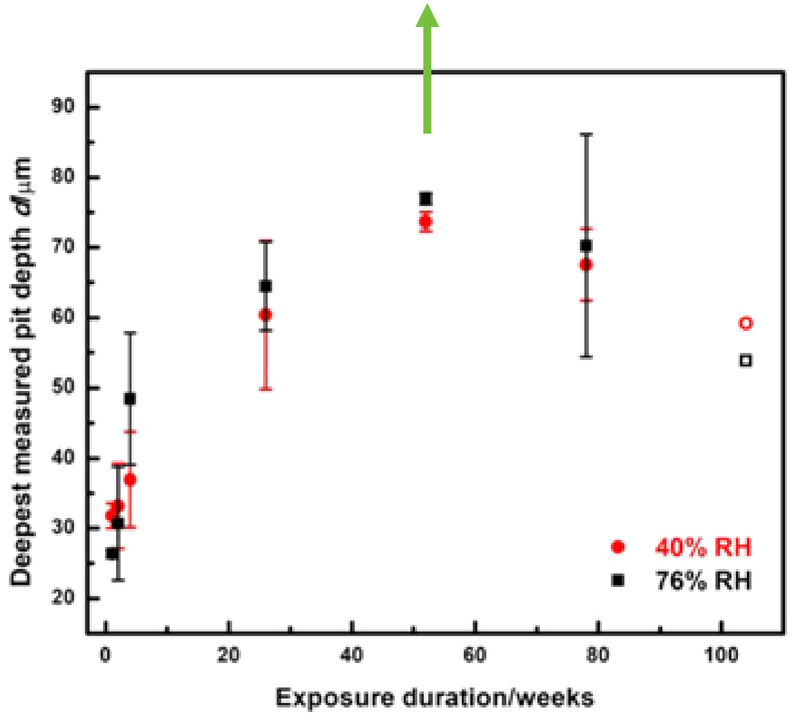
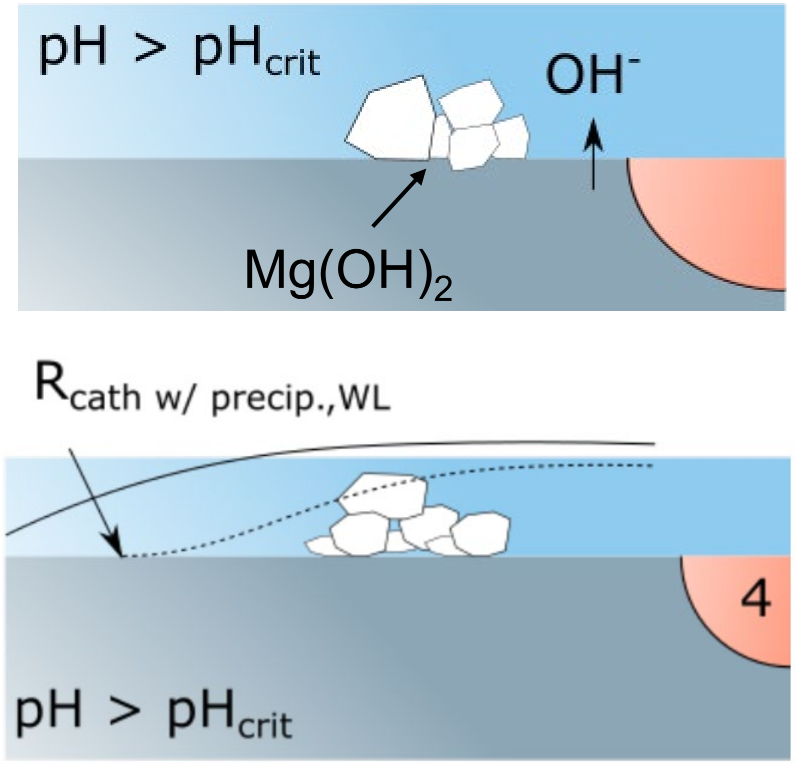


Potential influences on cathode



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Katona, R. M., Kelly, R. G., Bryan, C. R., Schaller, R. F., & Knight, A. W. (2020). Use of in situ Raman spectroelectrochemical technique to explore atmospheric corrosion in marine-relevant environments. *Electrochemistry Communications*, *118*, 106768.

Prediction of Maximum Pit Sizes

Comparison to long-term pitting exposures

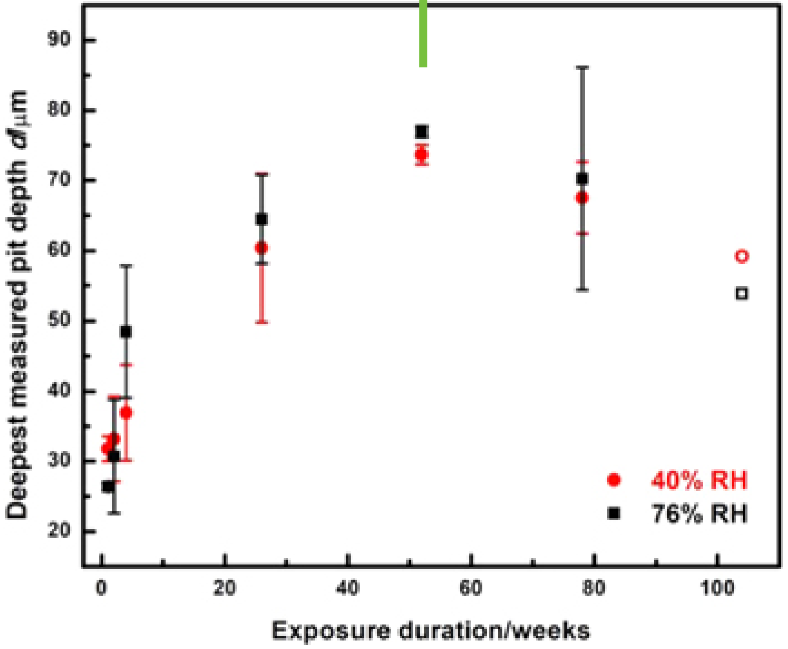
When comparing to exposures, prediction of maximum pit sizes with precipitation is ***directly inline for 40% RH***

Conservative estimates of the maximum pit

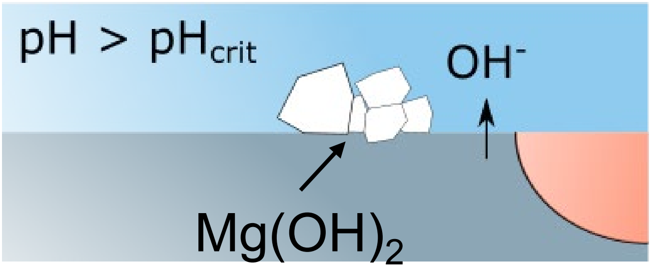
Roughly 1.5 x larger estimate

76 % Max Pit ~ 230 μm

40 % Max Pit ~ 110 μm

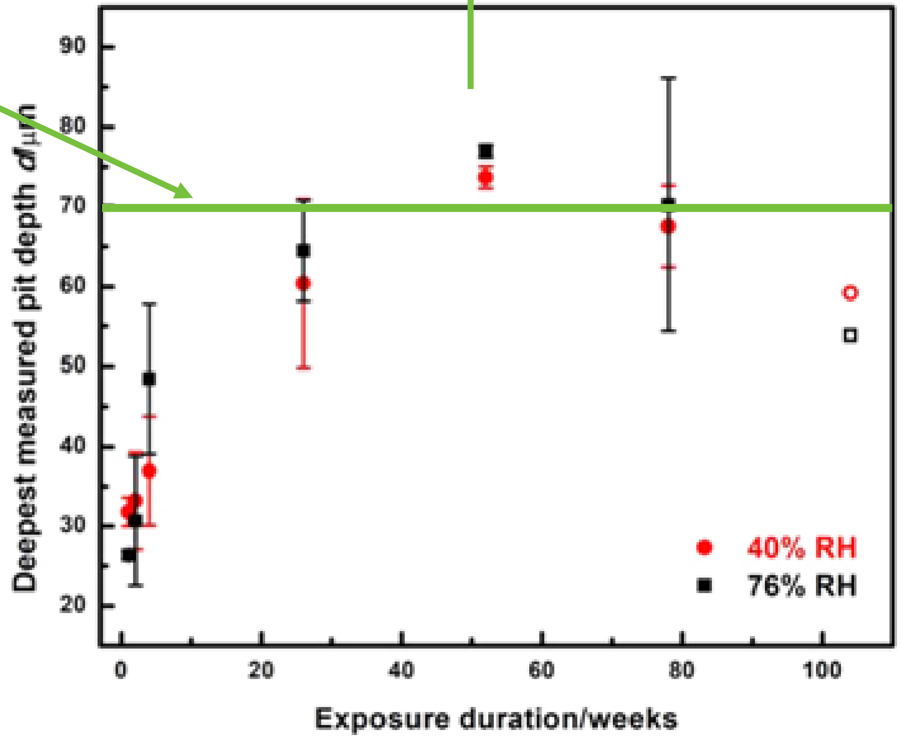


40 % Max Pit ~ 70 μm



Katona, R. M., Knight, A. W., Schindelholz, E. J., Bryan, C. R., Schaller, R. F., & Kelly, R. G. (2021). Quantitative assessment of environmental phenomena on maximum pit size predictions in marine environments. *Electrochimica Acta*, *370*, 137696.

76 % Max Pit ~ 190 μm



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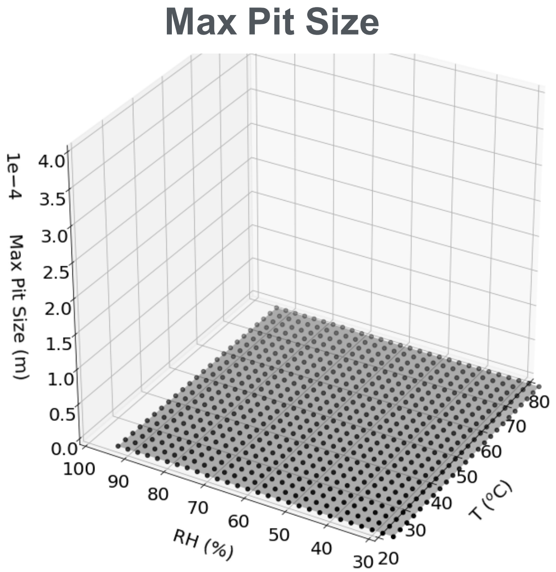
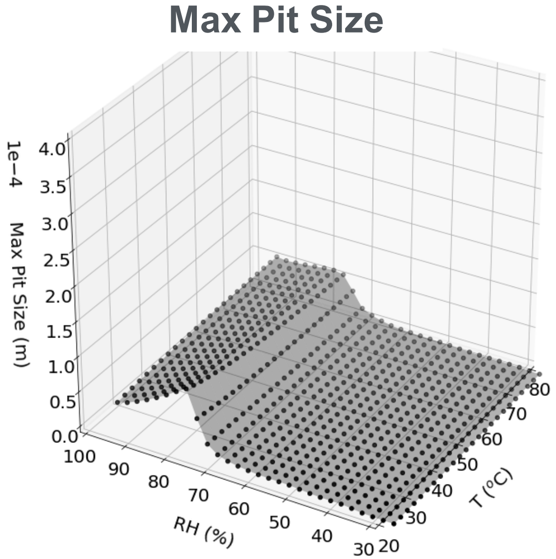
Prediction of Maximum Pit Sizes

Parameterization of the model

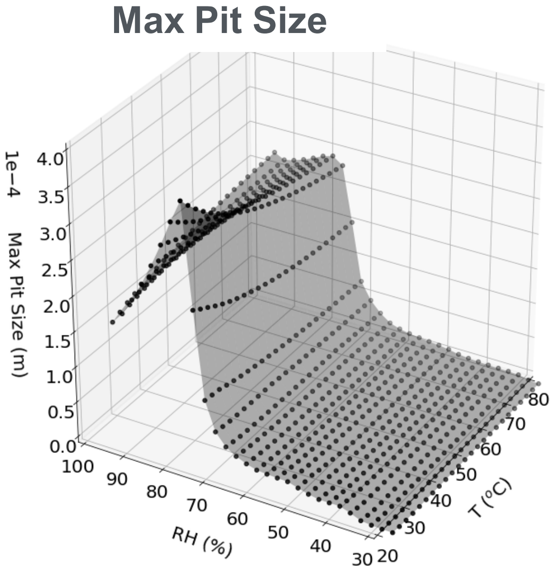
Increasing Salt Load (LD)



LD = 0.1 g/m^2



LD = 3 g/m^2



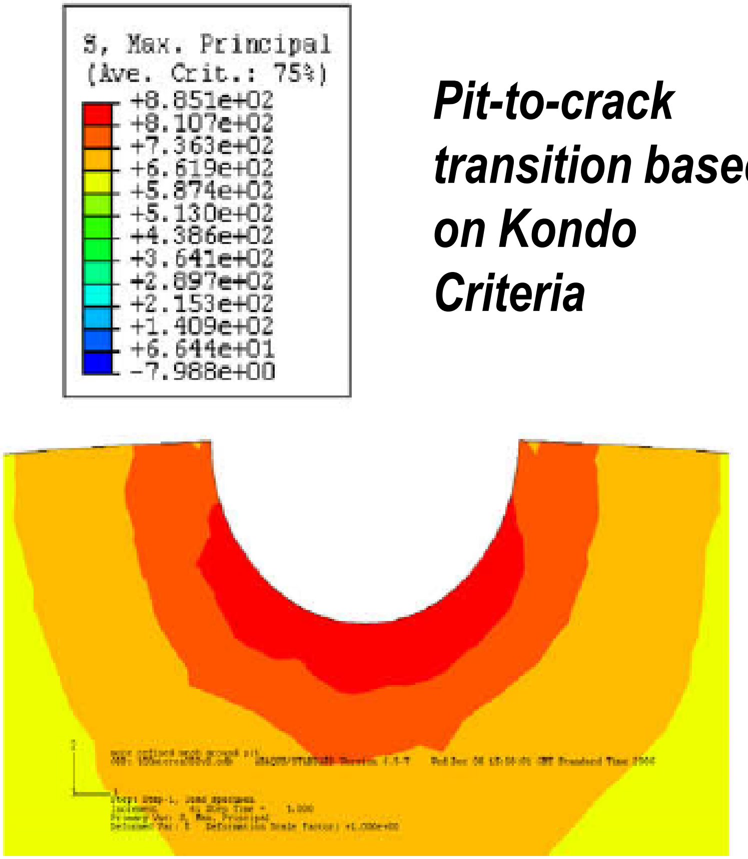
LD = 10 g/m^2

Environmental influences on corrosion damage (maximum pit size)

- ***Decreasing RH increases maximum pit sizes*** to a maximum at ~ 75 % RH
- ***Increasing temperature*** slightly ***decreases maximum pit*** sizes
- ***Increasing salt deposition increases maximum*** pit size

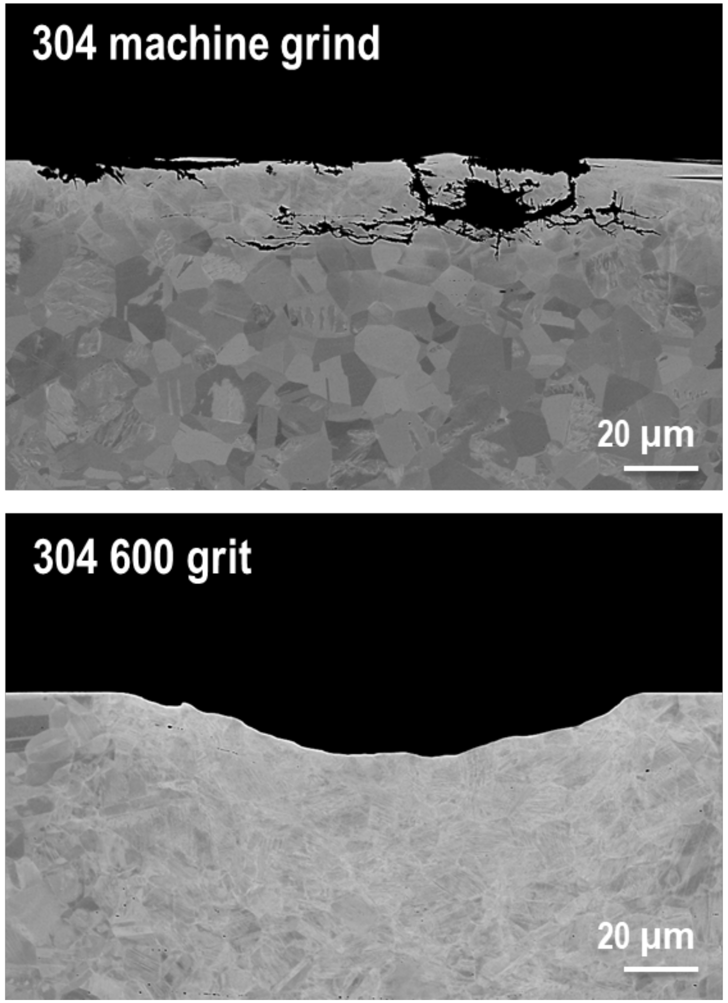
Environment and material influence on pit shape – why significant?

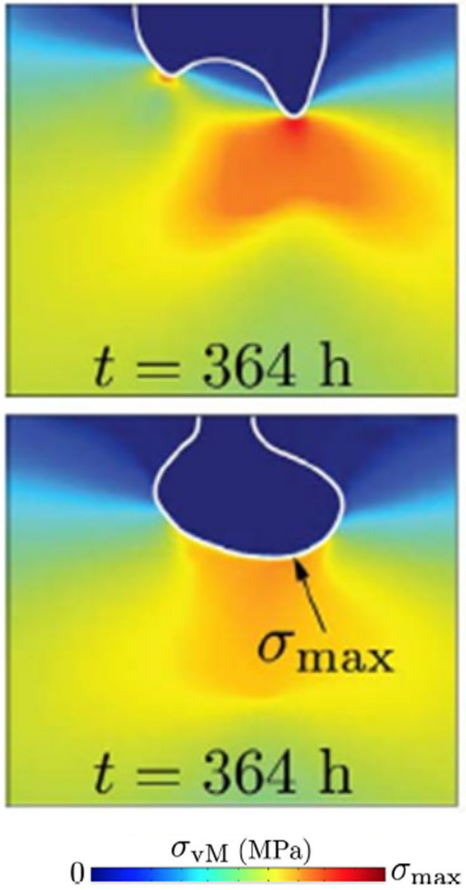
Why is pit shape significant?



Pit-to-crack transition based on Kondo Criteria

Turnbull, A., Wright, L., & Crocker, L. (2010). New insight into the pit-to-crack transition from finite element analysis of the stress and strain distribution around a corrosion pit. *Corrosion Science*, *52*(4), 1492-1498.





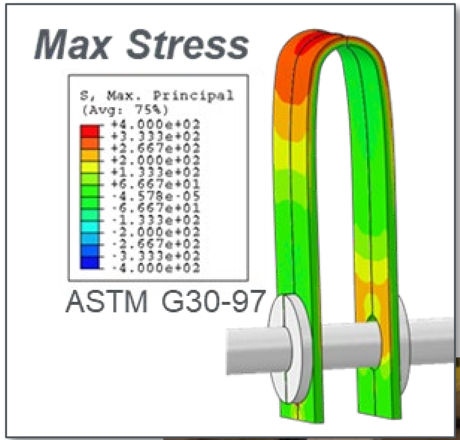
Mai, W., & Soghrati, S. (2017). A phase field model for simulating the stress corrosion cracking initiated from pits. *Corrosion Science*, *125*, 87-98.

Canister-Relevant Testing Environments: Pit to Crack

Large Scale Exposure Testing: U-bend coupons to examine pit to crack transition

Initial Exposures:

- #4 Machine Finish (60 grit) 304L
- 300 µg/cm^2 artificial seawater
- or 500 µg/cm^2 $MgCl_2$
- Exposure: diurnal cycle and static 40% RH



Example stress modeling

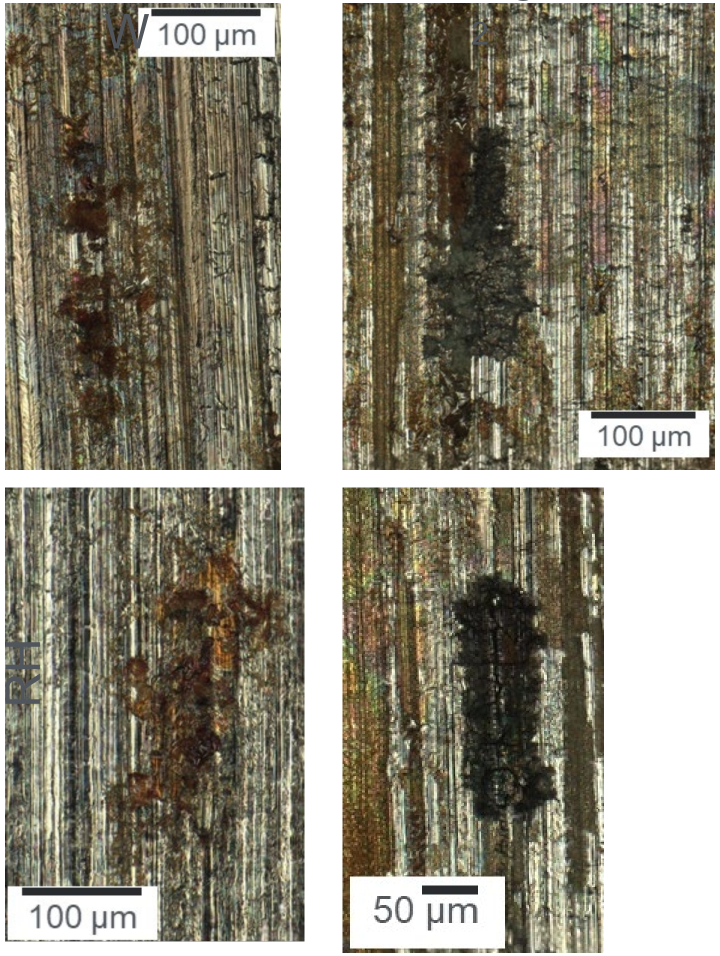
Salt Deposition

Initial Optical Observations

AS MgCl

cyclic

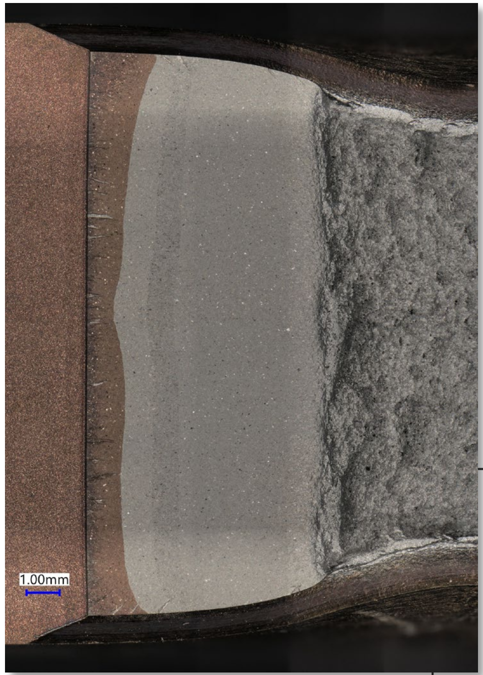
40%



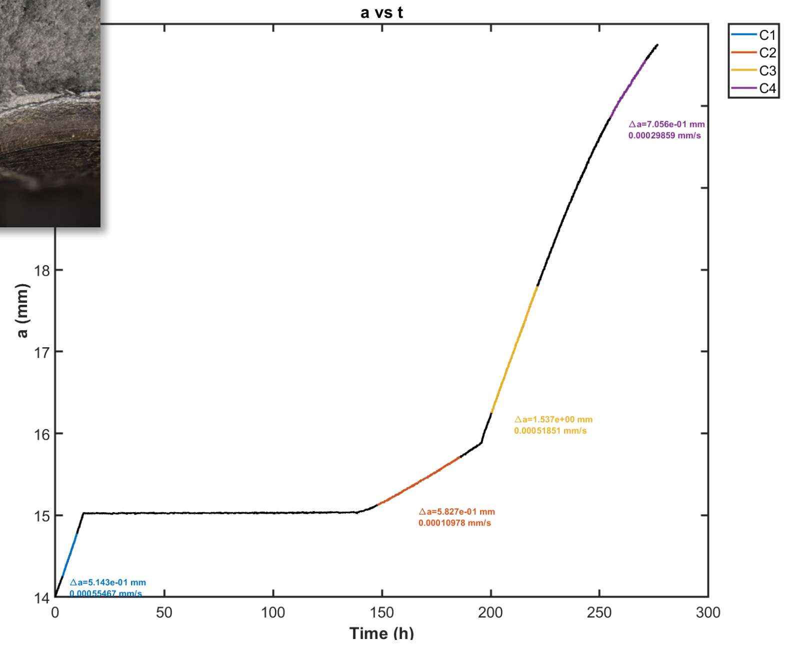
Crack Growth Rate – Lab setup and Calibration

- ***Final CGR lab setup complete***
- ***DCPD testing in air underway***

Example: DCPD testing in air



Crack length vs time

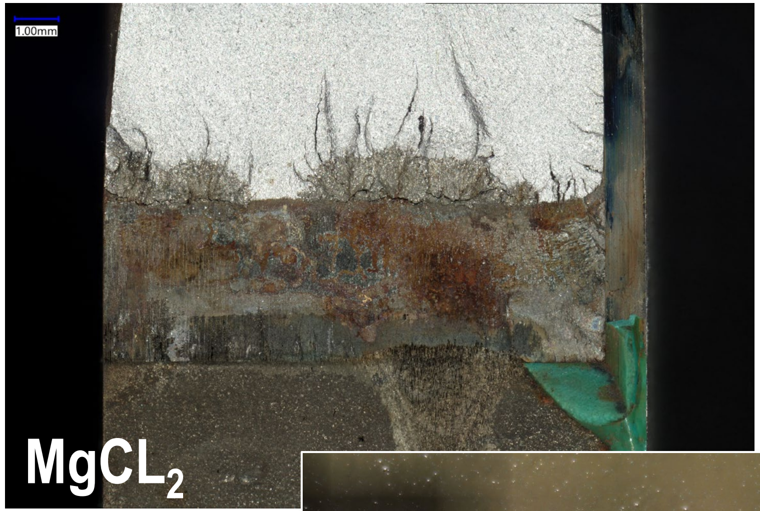


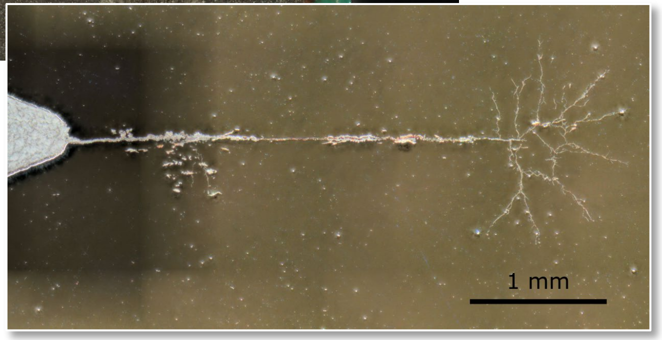
Crack Growth Rate in Relevant Brine Environments

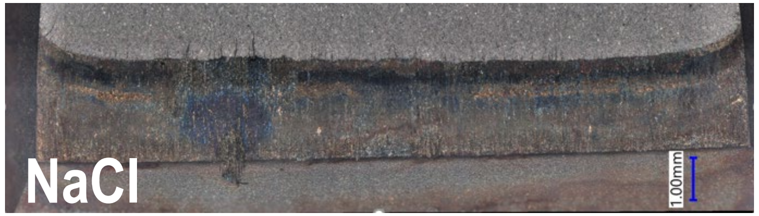
- ***Final CGR lab setup complete***
- ***DCPD testing in air underway***

Developing an understanding of DCPD and fractography in saturated salt solutions

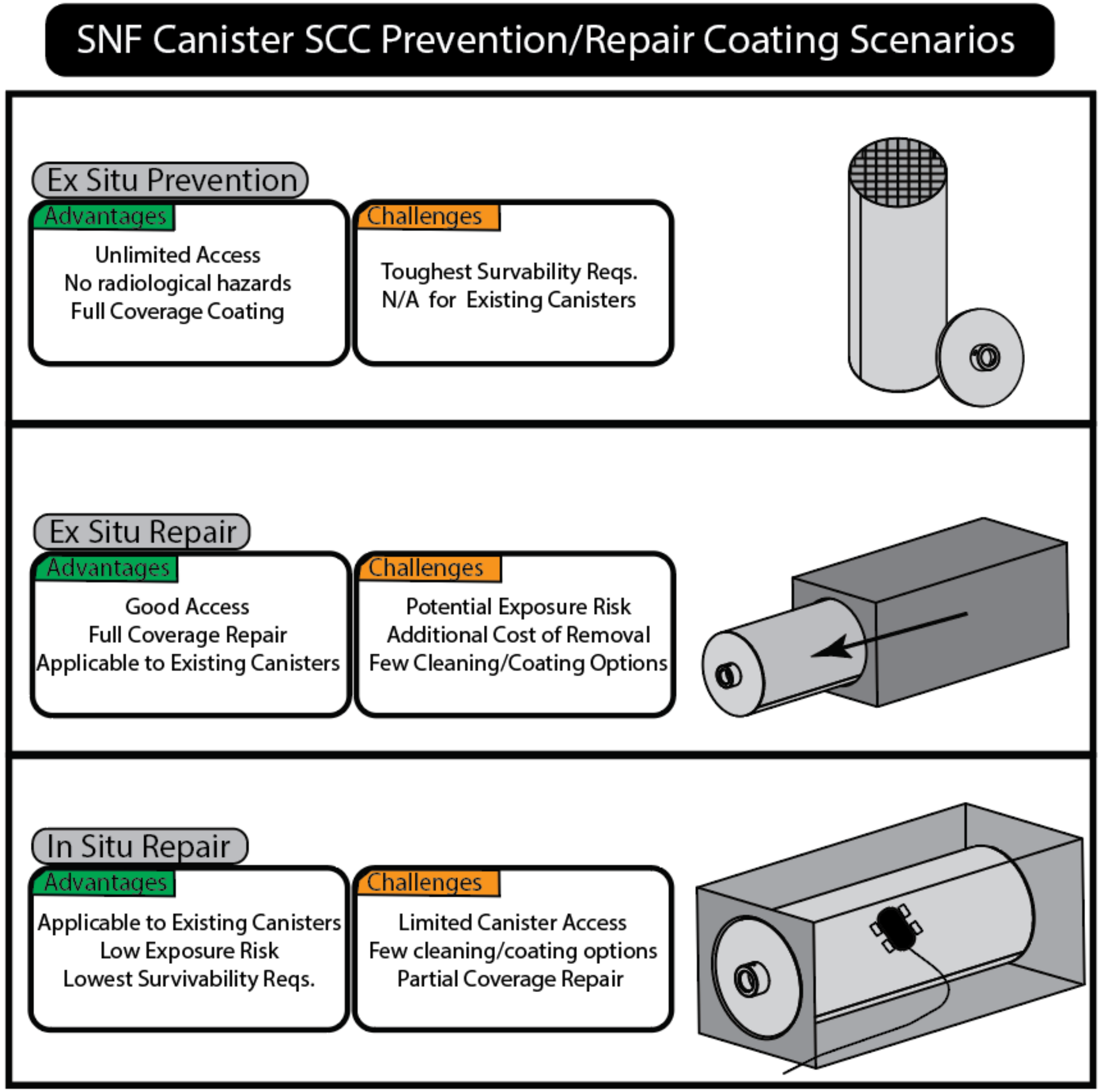
Example: Saturated $MgCl_2$ Tests Compared to NaCl





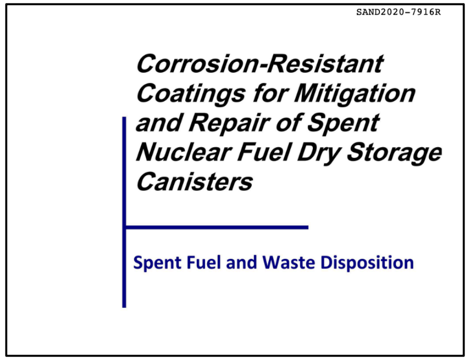


Mitigation and Repair: Canister Coatings Evaluation



1. Collaborative effort with industrial partners
   - *Based on FY20 coatings report*

Initial Scoping Report


SAND2020-7916R

Corrosion-Resistant Coatings for Mitigation and Repair of Spent Nuclear Fuel Dry Storage Canisters

Spent Fuel and Waste Disposition

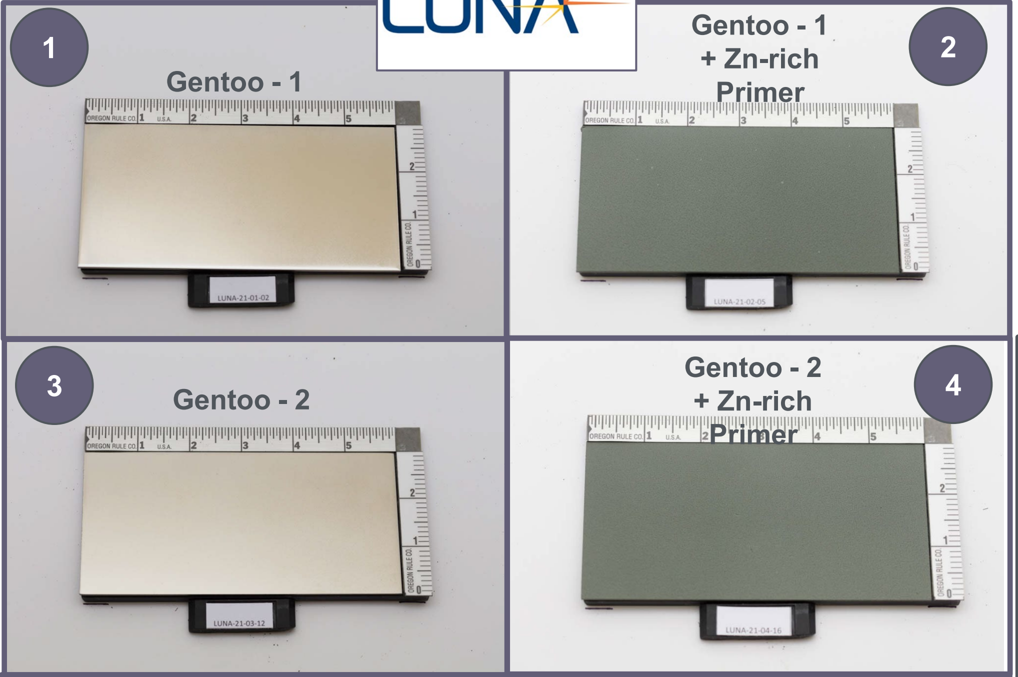
2. Collaboration with PNNL to evaluate cold spray as a potential mitigation and repair strategy

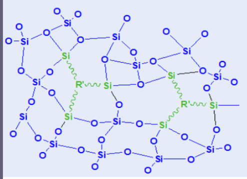
SNL-Industrial Collaboration– Initial coatings for evaluation

Coating types:

4 collaborating companies, 11 variants

LUNA

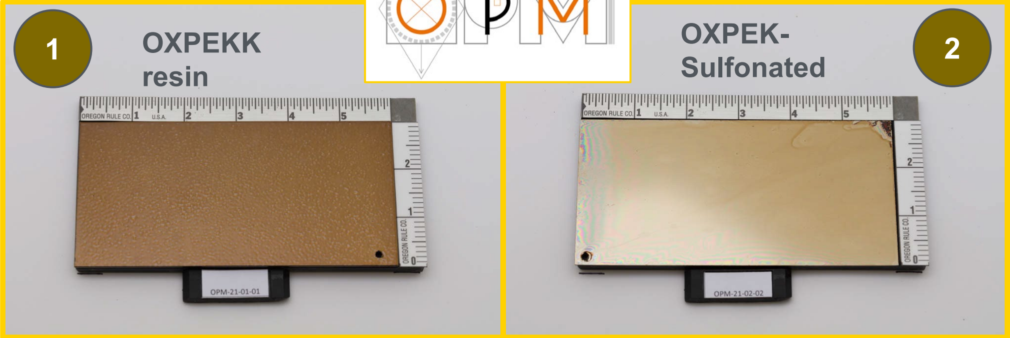


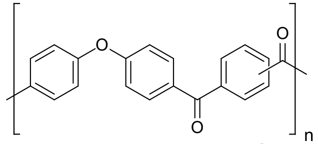


5- variants of Gentoo with and without Zn-rich primer

Durable ceramic hybrid inorganic/polymer coating with/without galvanic protection

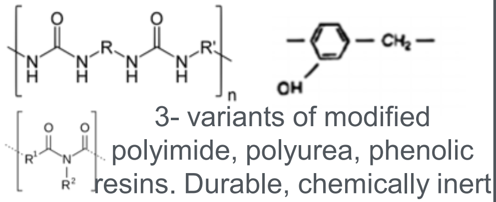
OPM





2- variants of Polyetherketoneketone (OXPEKK).
High temperature thermoplastic with high radiation resistance

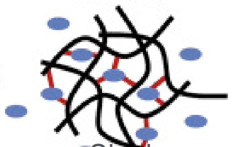
White Horse



3- variants of modified polyimide, polyurea, phenolic resins. Durable, chemically inert and can include additives to increase corrosion resistance

FLORA Coatings

An Innovation Driven Company



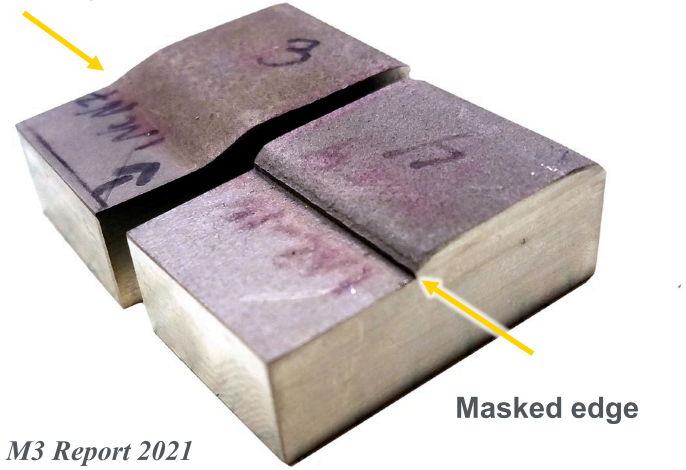
Single component hybrid inorganic/modified polyurethane coating resulting in a quasi-ceramic structure.

SNL – PNNL collaboration:
Cold Spray – Accelerated Corrosion Testing

Cold Spray Matrix

CS Material	Interface	Process Gas
Inconel 625	Blended	He
Inconel 625	Blended	N
Inconel 625	Masked	N
Nickel	Blended	N
Nickel	Masked	N
Super C	Blended	N

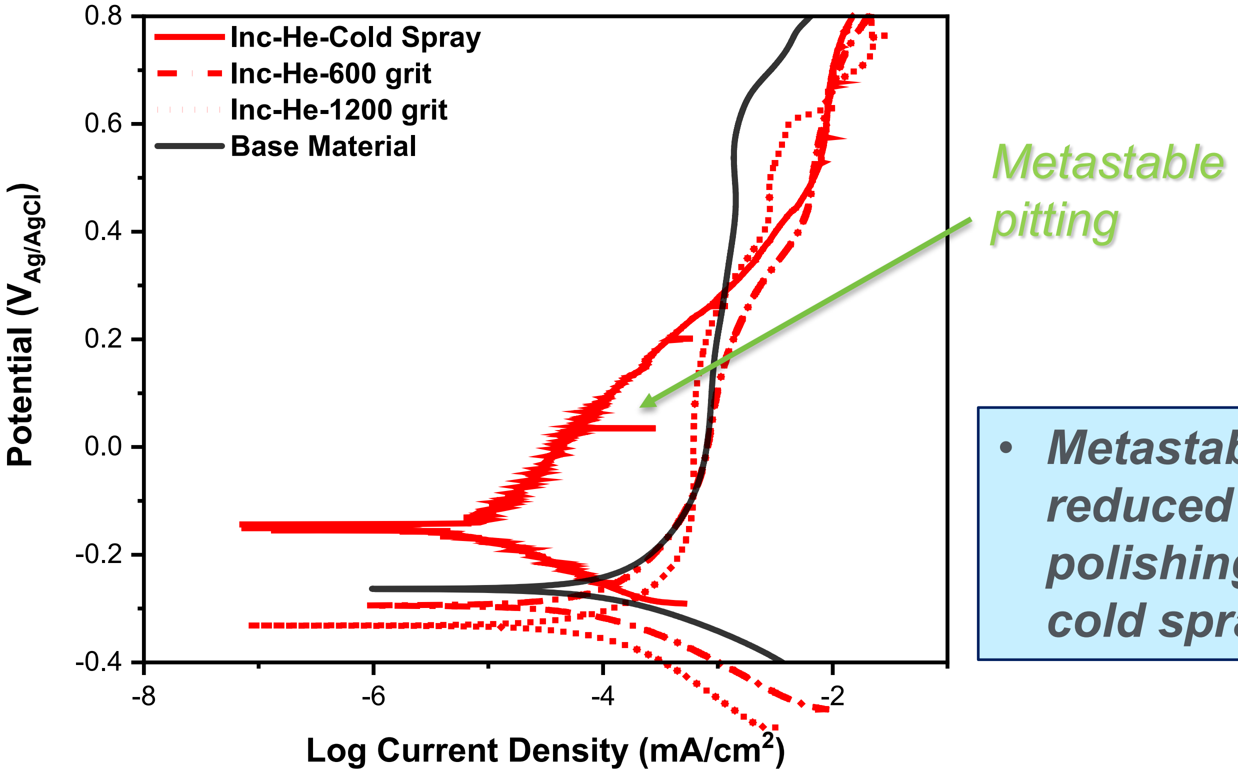
Cold Spray Samples with Edge Processing



PNNL M3 Report 2021

Accelerated Corrosion Testing for Cold Spray Optimization:

ASTM G-5: potentiodynamic polarization in 0.6 M NaCl



- *Metastable pitting reduced by polishing/grinding cold spray surface*

SNL – PNNL collaboration:
Cold Spray – Accelerated Corrosion Testing

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Inconel 625	Blended	He
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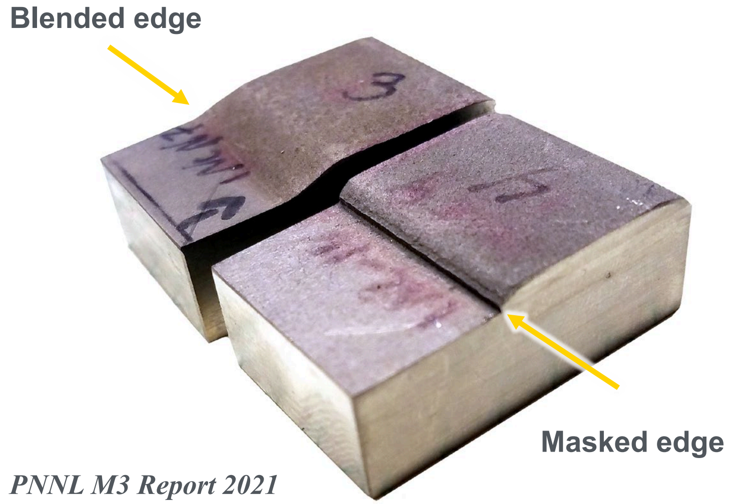
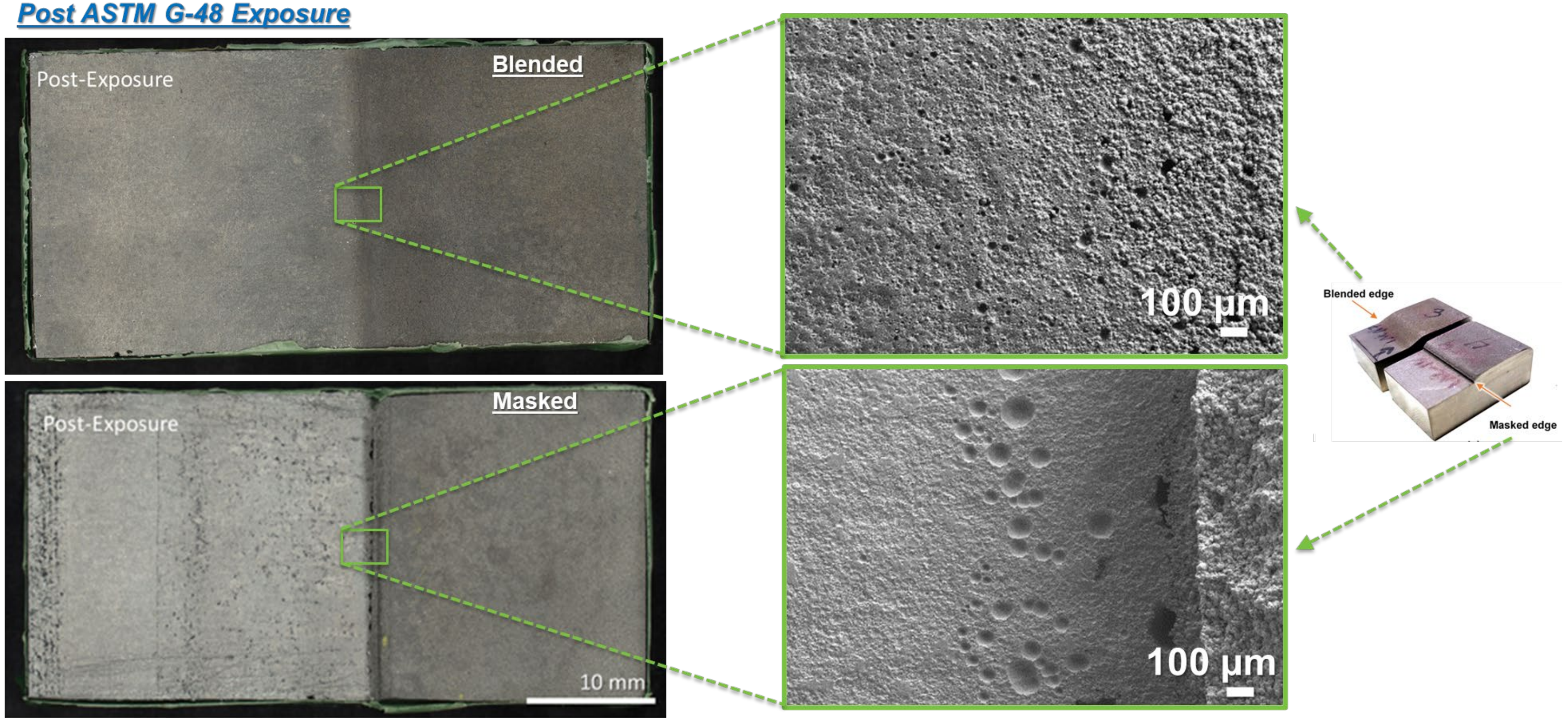


PNNL M3 Report 2021

Accelerated Corrosion Testing for Cold Spray Optimization:

ASTM G48: full immersion pitting 6% by weight $FeCl_3$

Post ASTM G-48 Exposure



- ***Majority of attack at interface and influenced by edge type***

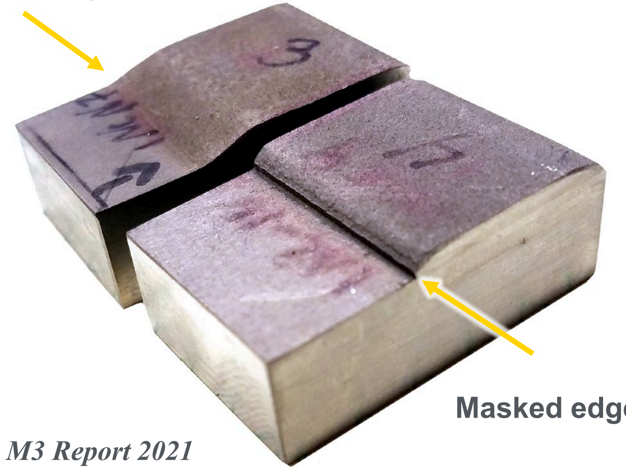
SNL – PNNL collaboration:
Cold Spray – Accelerated Corrosion Testing

Cold Spray Matrix

CS Material	Interface	Process Gas
Inconel 625	Blended	He
Inconel 625	Blended	N
Inconel 625	Masked	N
Nickel	Blended	N
Nickel	Masked	N
Super C	Blended	N

Cold Spray Samples with Edge Processing

Blended edge



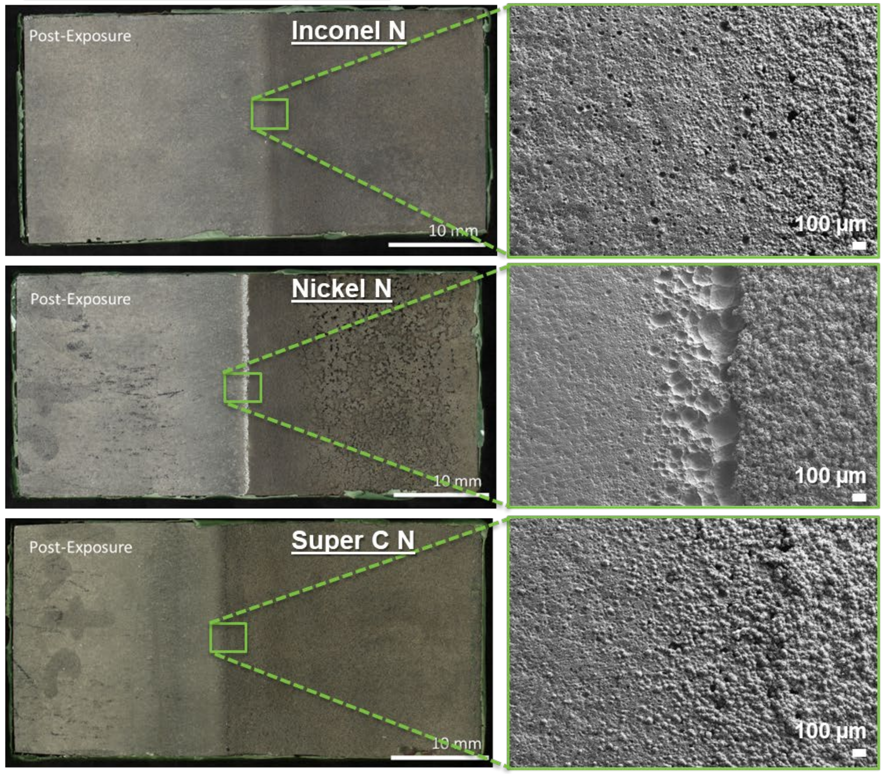
Masked edge

PNNL M3 Report 2021

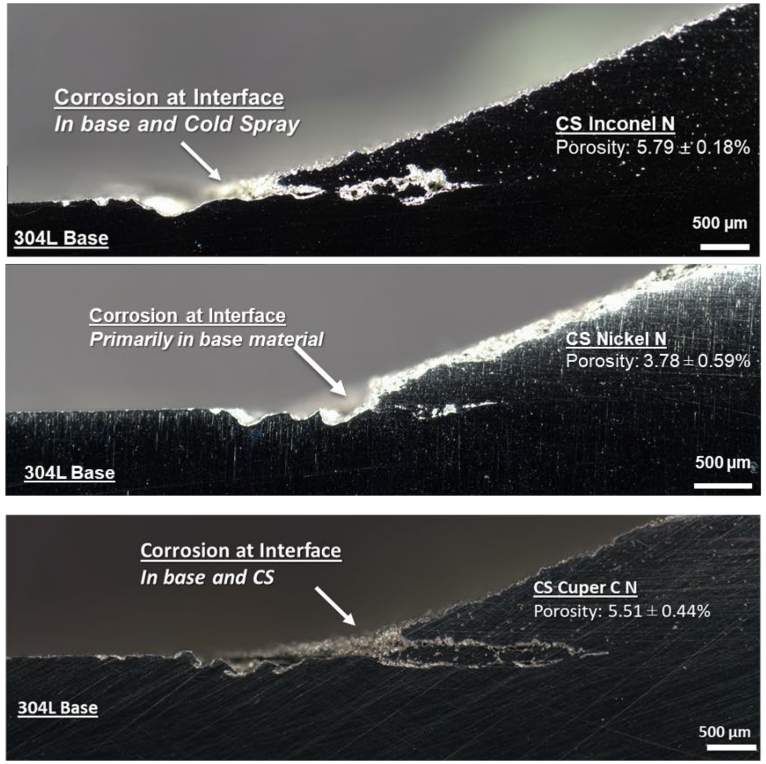
Accelerated Corrosion Testing for Cold Spray Optimization:

ASTM G48: full immersion pitting 6% by weight $FeCl_3$

Post ASTM G-48 Exposure



Cross section Post Exposure



- ***Attack influenced by material type and process gas/porosity of cold spray.***

Summary: Primary Goals of Current and Future Work

- **Environmental Studies:**
  - Analysis of dust from in-service canisters—characterization of canister surface environments for corrosion testing
  - Mg-chloride brine stability (timing/temperature of corrosion, extent and morphology of corrosion, etc.)
  - Brine DRH as a function of salt composition (timing/temperature of brine development)
  - Dust/salt deposition (CDFD)
- **Corrosion testing and modeling in canister relevant environments**
  - Examining influence of canister-relevant environments on corrosion (pitting and pit to crack)
  - Expanding modeling efforts to account for non-static brine/corrosion conditions to better predict pitting and SCC initiation
- **Crack growth rate**
  - Installed, calibrating, and reviewing initial tests in varied brine environments to explore potential effects on CGR
- **Coatings**
  - Developed MOU with industry partners, received initial coatings for evaluation at SNL
  - Collaborated with PNNL for accelerated corrosion evaluation of CS coatings

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Questions?