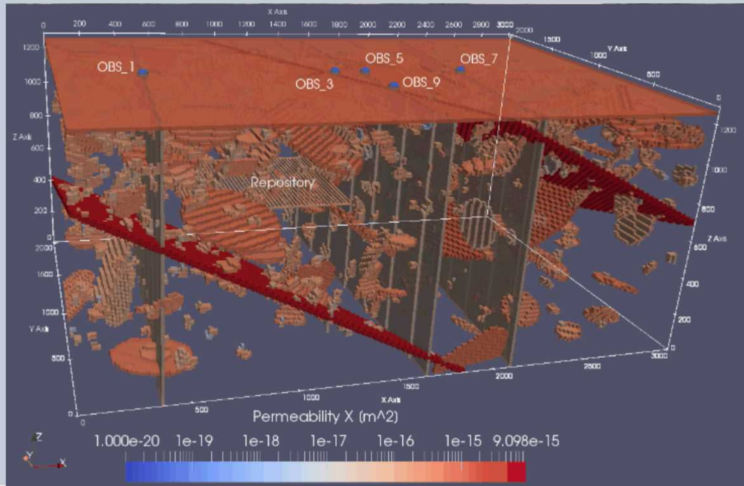
Corrosion Research at Sandia National Laboratories

CISCC Program Meeting

SAND2020-2857PE



SFWD

Spent Fuel & Waste Disposition

CISCC Program Meeting

March 4, 2020

Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International Inc., for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-NA0003525.

1. Goals of SNL Program: Corrosion and SCC

Overall objective

1. Improve ability to predict timing and location of potential canister penetration by stress corrosion cracking (SCC)

- *Probabilistic SCC model for improved prediction of canister performance.*
- *Provide guidance for aging management programs*

2. Provide input on SCC mitigation and repair strategies

Goals of experimental work

- 1) **BRINE**: Determine electrolyte composition and evolution with time
- 2) **CORROSION**: Determine the relationship between surface environment, material properties, mechanical environment, and damage distribution and rates
- 3) **PIT-TO-CRACK**: Determine the environmental and mechanical properties that influence when and where pit-to-crack transition occurs
- 4) **CGR**: Determine crack growth rate as a function of environment and material properties

2. How do we achieve this?

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1) <u>Material Properties:</u>

Characterization of Weld Metal Properties

- Evaluate sensitization, hardness, microstructure (metrics for degree of plastic deformation)
- How do these factors influence subsequent corrosion and SCC?

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2) Surface Environment:

- Concentrated Brines ($MgCl_2$)
  - Cathode size
  - Pit initiation and morphology
  - Change in cathodic reaction from ORR to HER
  - Effect of brine composition
- Do nitrates play an inhibitive role?
- Does inert dust effect brine distribution/ cathode properties?
- Other probable environmental effects:
  - Cyclic conditions (Diurnal variations)
  - Metal contamination

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3) **Damage Distribution and Rates:**

Pit-to-Crack Transition and CGR

- Factors controlling pit-to-crack transition
  - Pit morphology/ modeling stress (Kondo criterion)
  - Environment dependence f(T, RH, Cl^-, etc.)
  - Plastic strain, microstructure, residual stress
- CGR experiments:
  - Strain rate dependence
  - Environment dependence f(T, RH, Cl^-, cyclic conditions, etc.)
  - Material properties
  - Acoustic monitoring of CG (cyclic conditions, brine properties)
- Large Plates: Why don't these crack?
  - What are the surface stresses? (Center hole drilling)
  - Metallurgy: microhardness to determine work hardening

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4) **Mitigation Strategies:**

- Evaluation of FSW and Cold Spray techniques
- Development of qualification for mitigation strategies (based on CISCC knowledge thus far)

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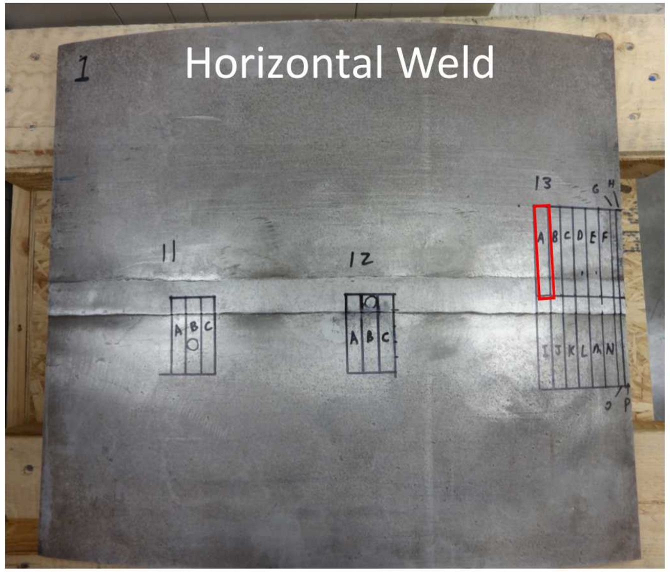
WELD METAL PROPERTIES

- Experiments:
  - **Weld sections from Big Plate test: longitudinal and circumferential**
    1. Corrosion Analysis
    2. Microstructural Etch
    3. EBSD map
    4. Evaluate sensitization around weld (DLEPR)
    5. Microhardness map
    6. Surface stress measurements (center hole drilling at SNL)
- Why:
  - Provides information on weld material properties, specifically:
    1. Degree of cold work due to weld shrinkage (hardness ∝ CW)
    2. Sensitization in weld and HAZ
    3. Residual stresses
- Who/What else it feeds
  - Information pertinent for:
    - SNL for SCC (pit-to-crack and CGR) testing inputs (*relevant material properties*)
    - PNNL (*direct inputs for material properties in CGR testing*)
    - SRNL (*validation/ additional information of large plate SCC testing*)
- What is needed?



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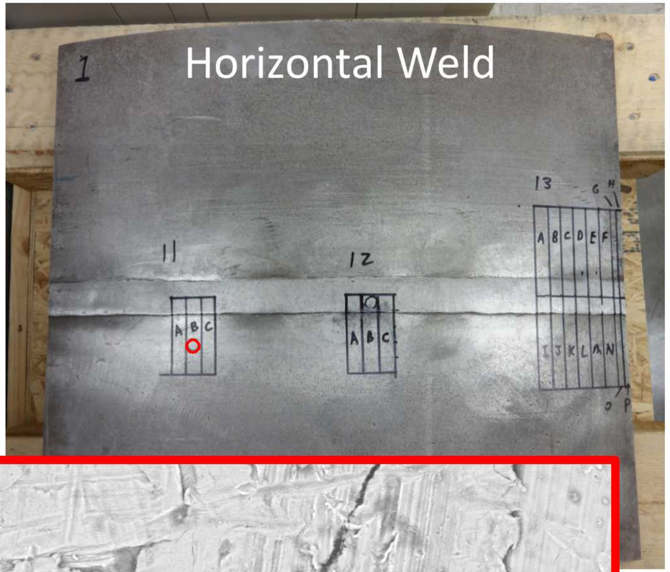

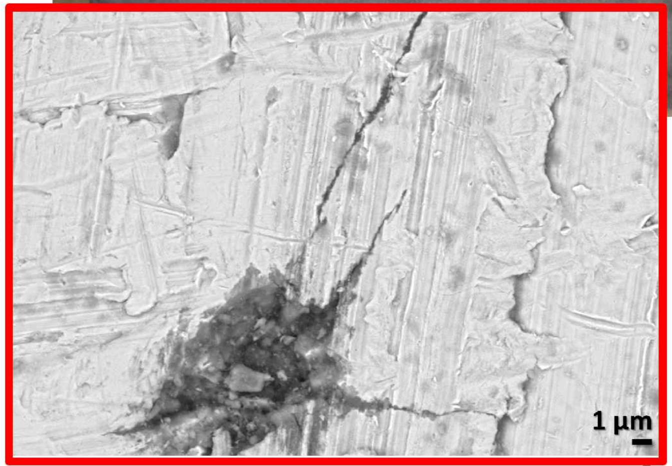

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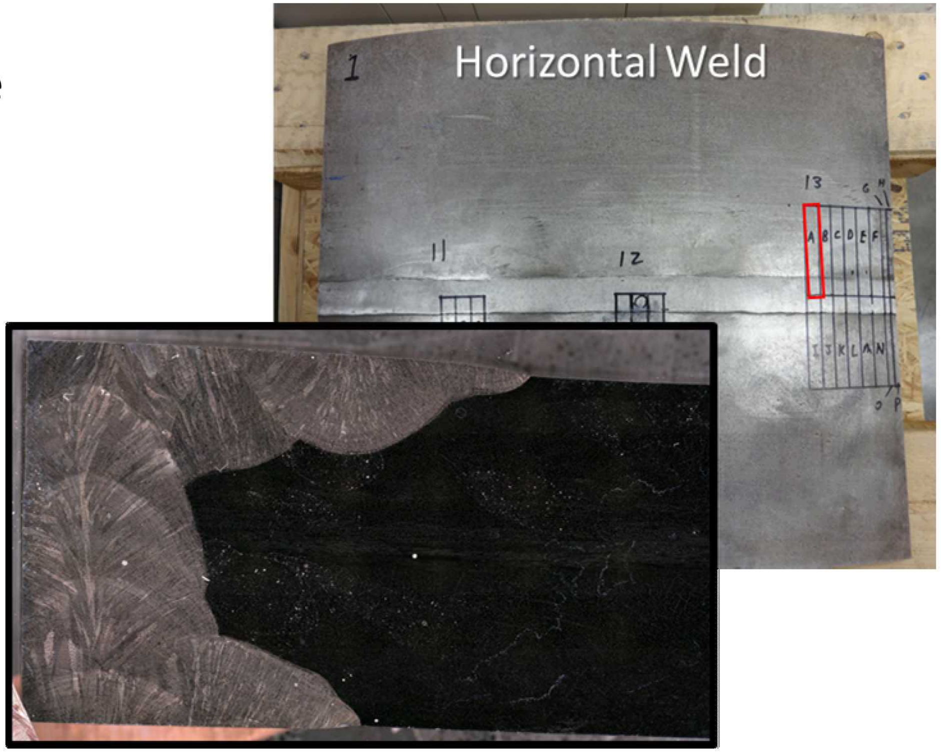

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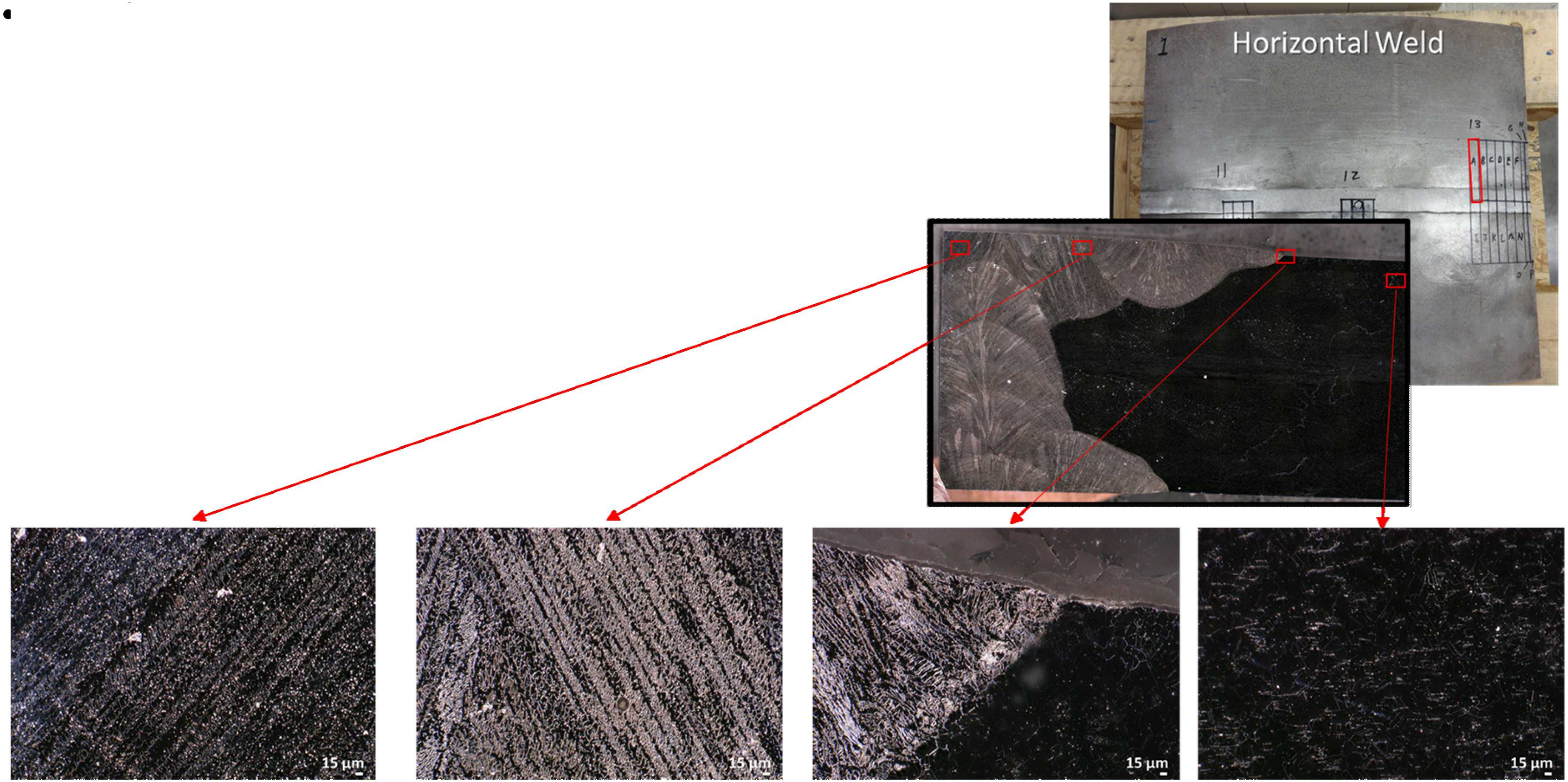

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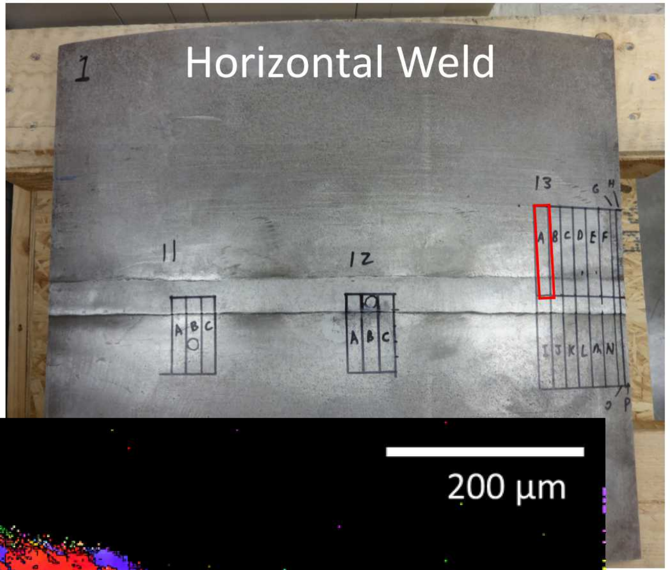

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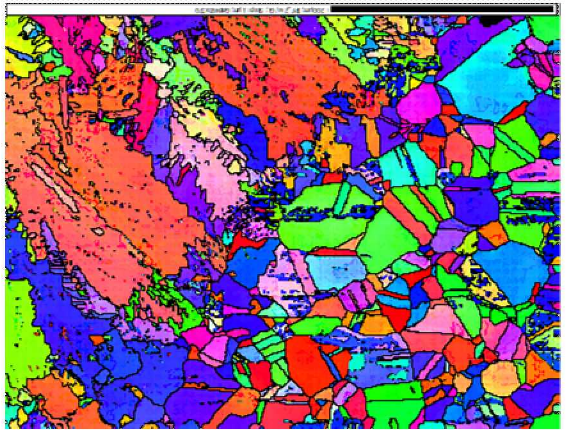

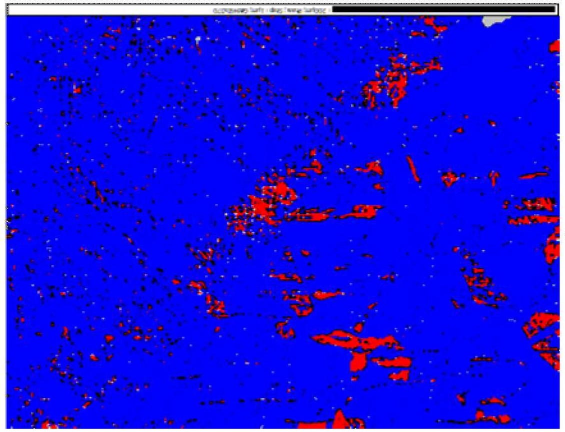

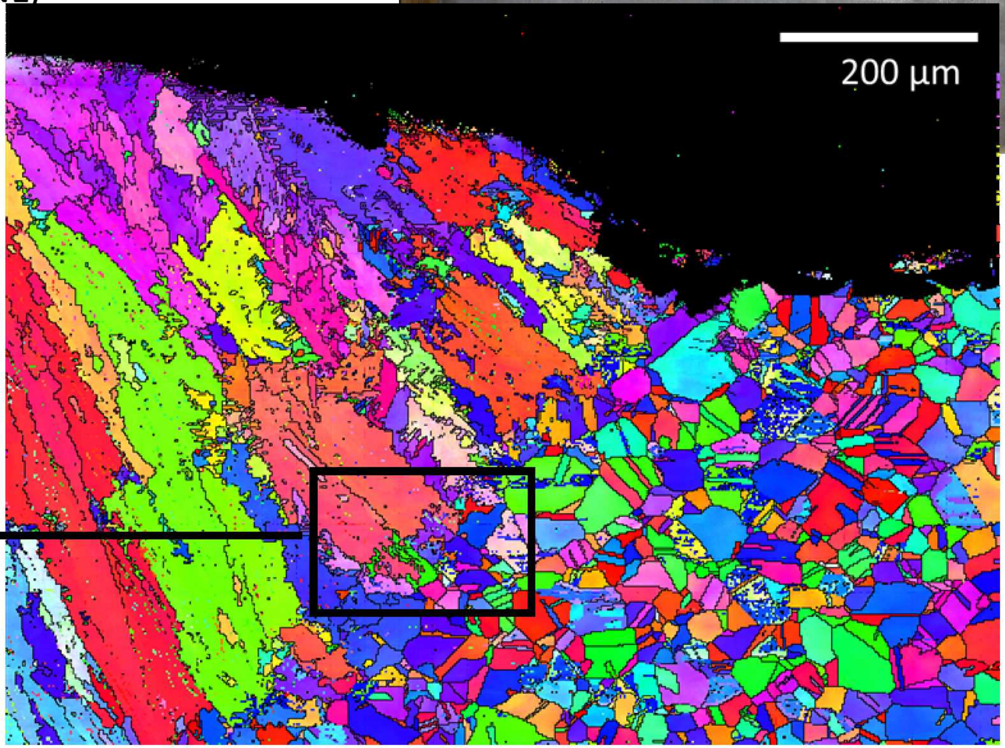

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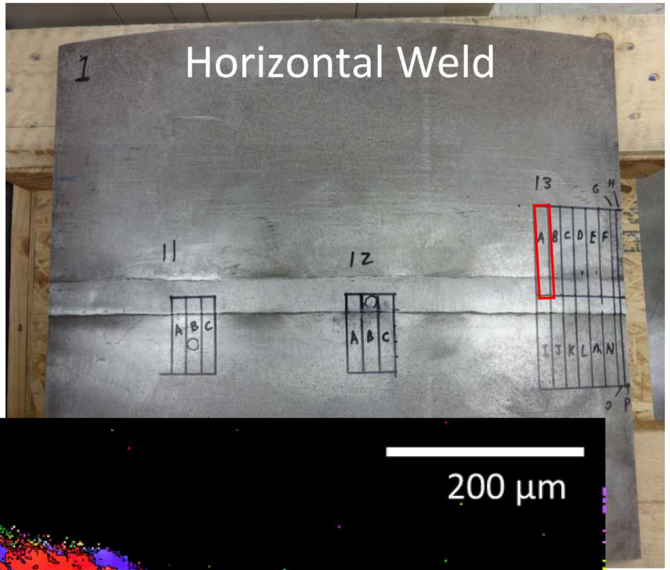

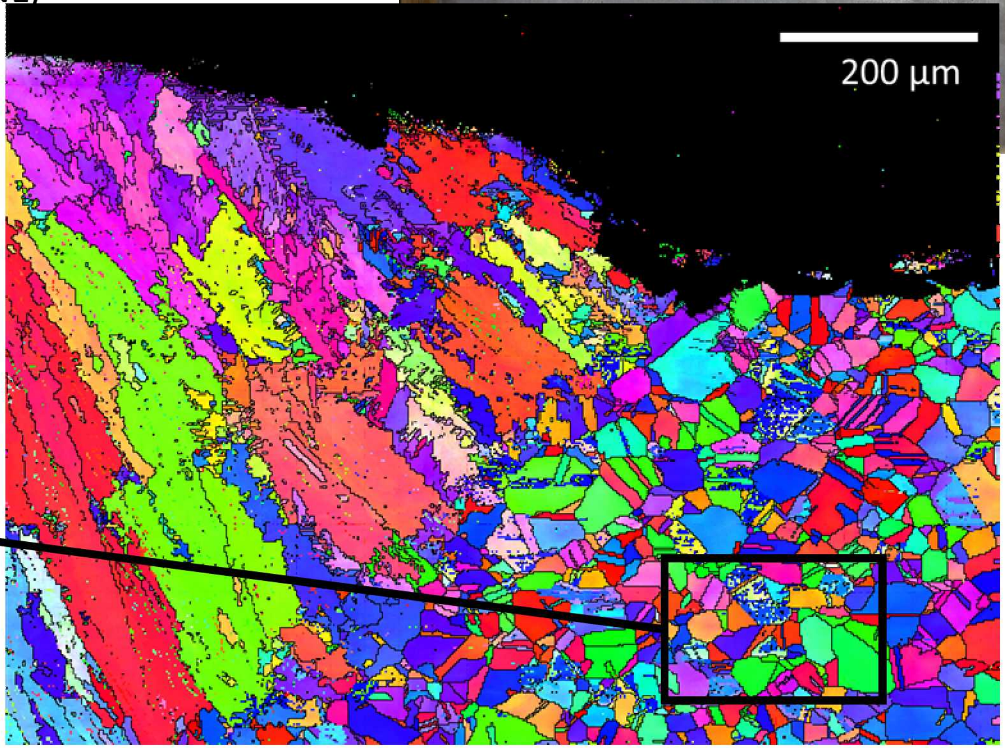

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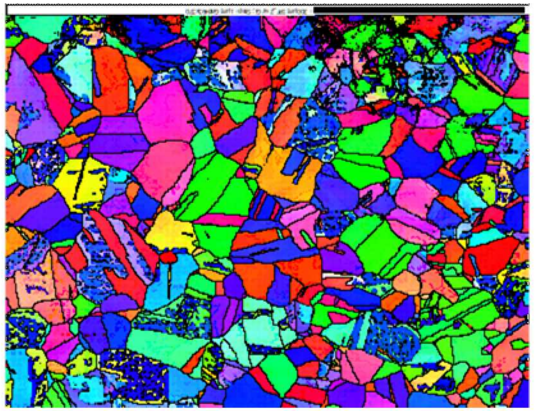

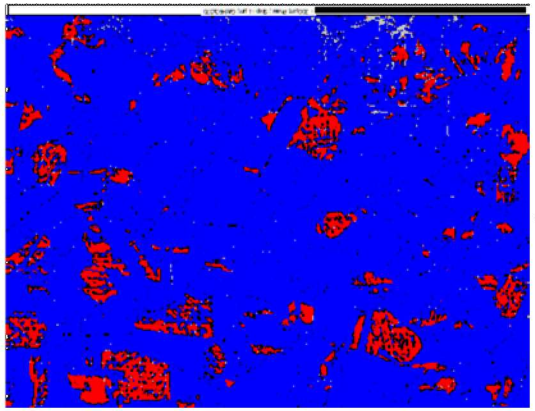

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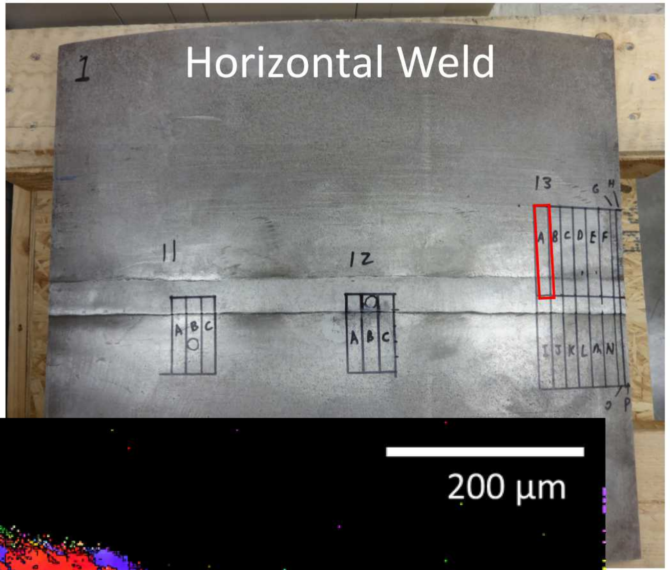

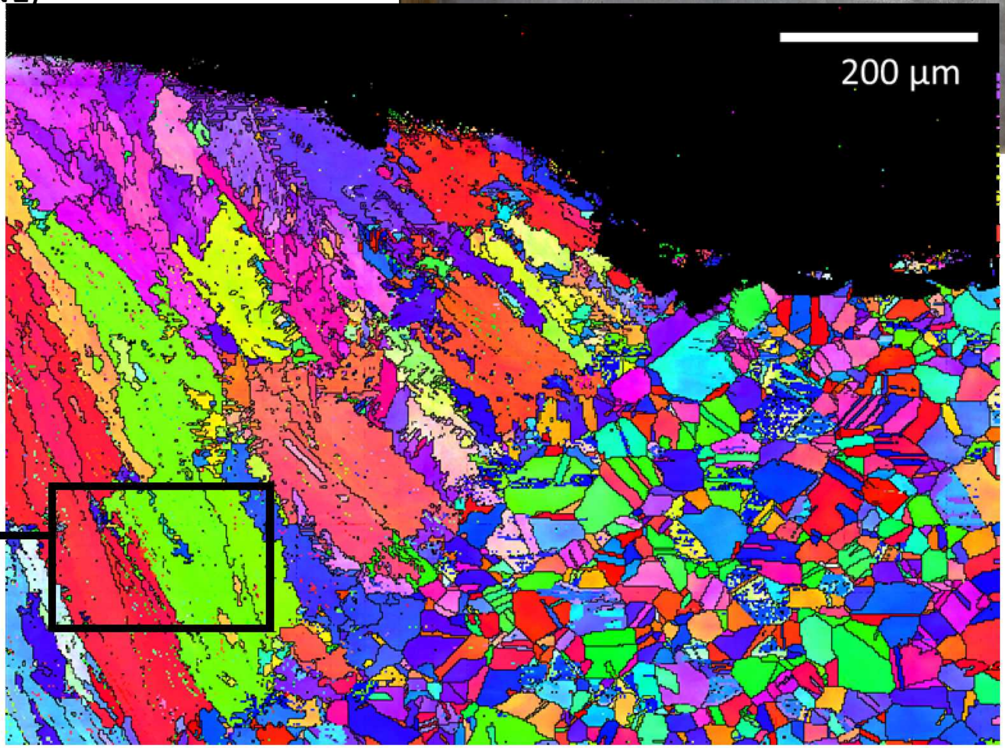

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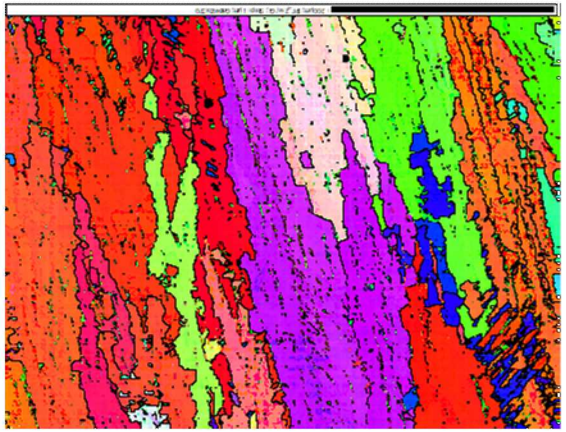

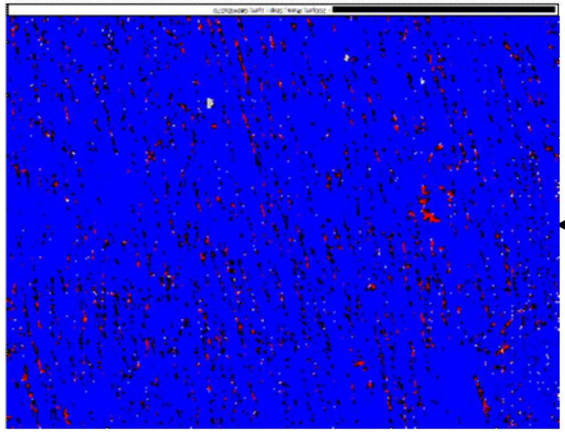

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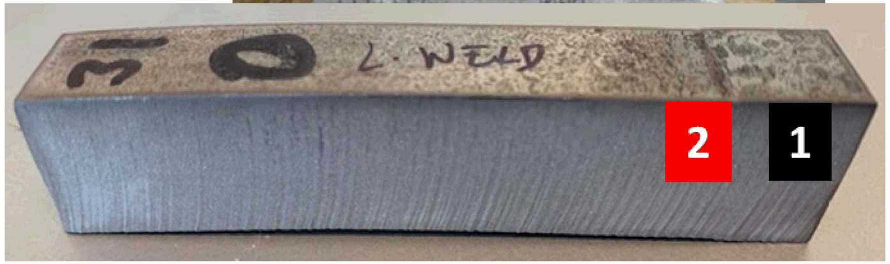

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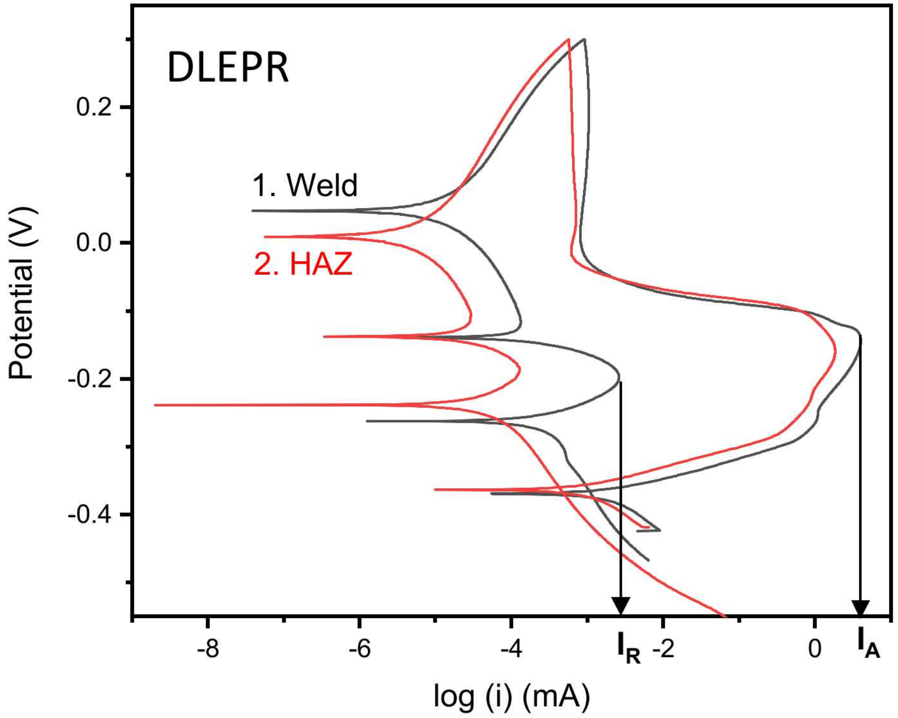

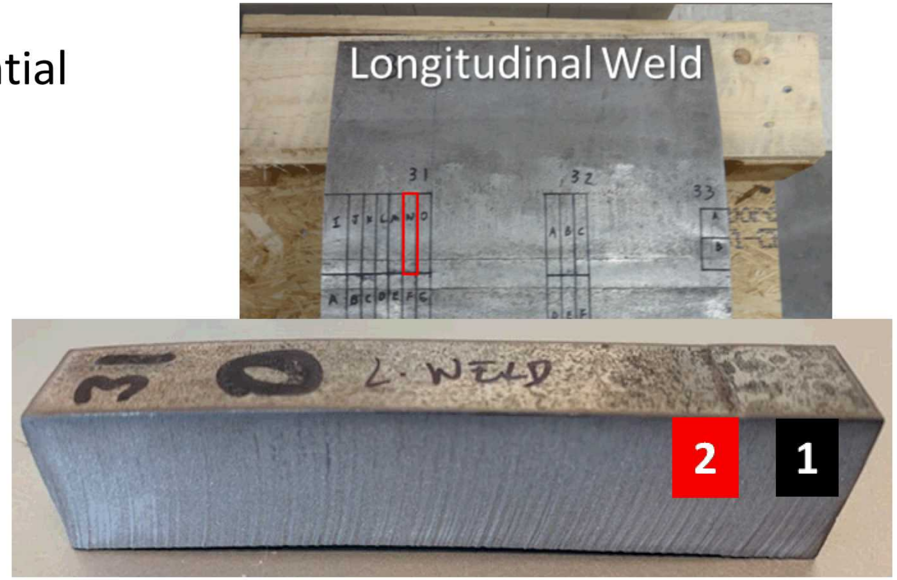

SNL/ Montoya

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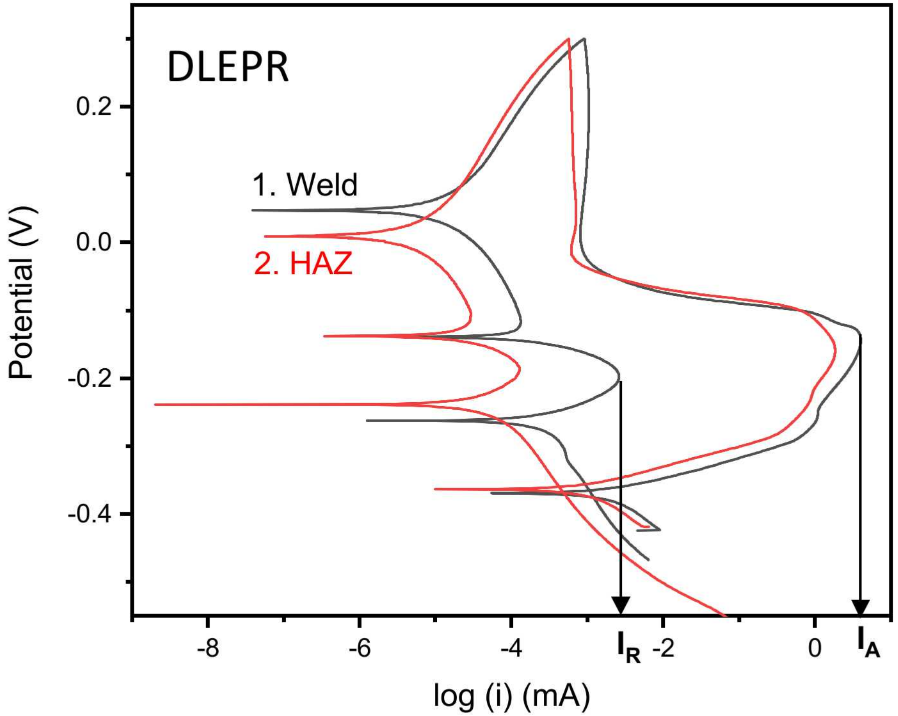

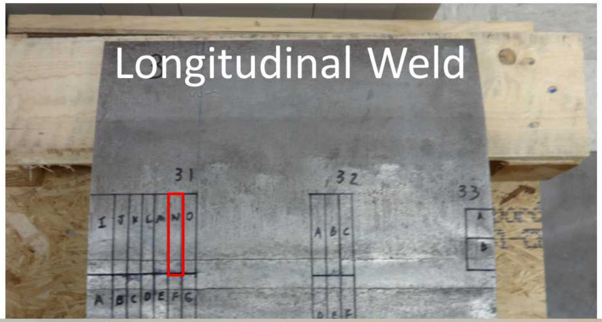

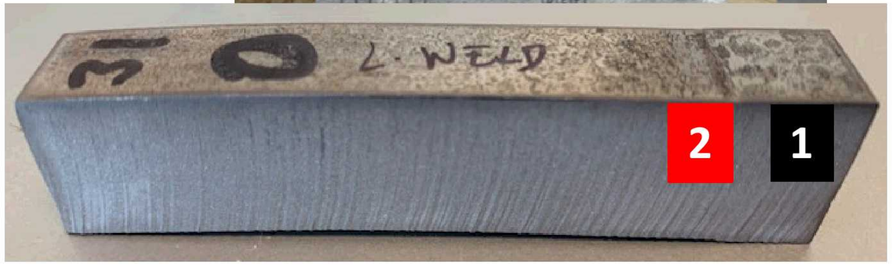

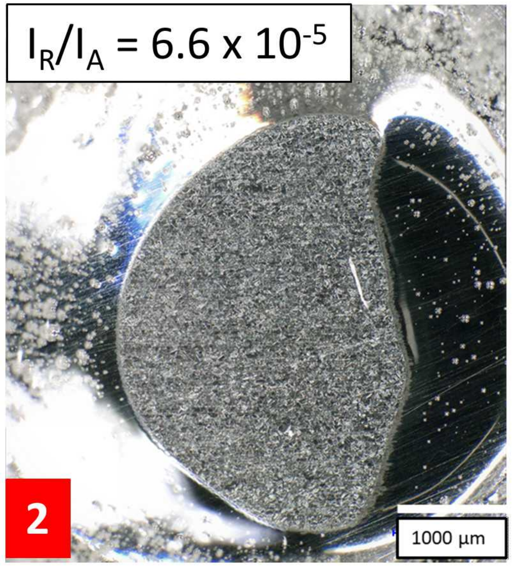

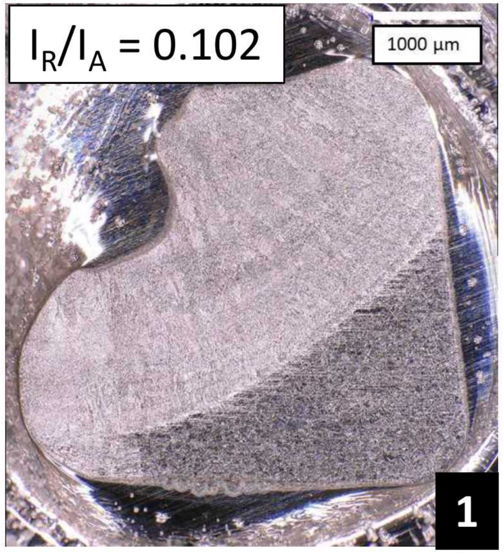

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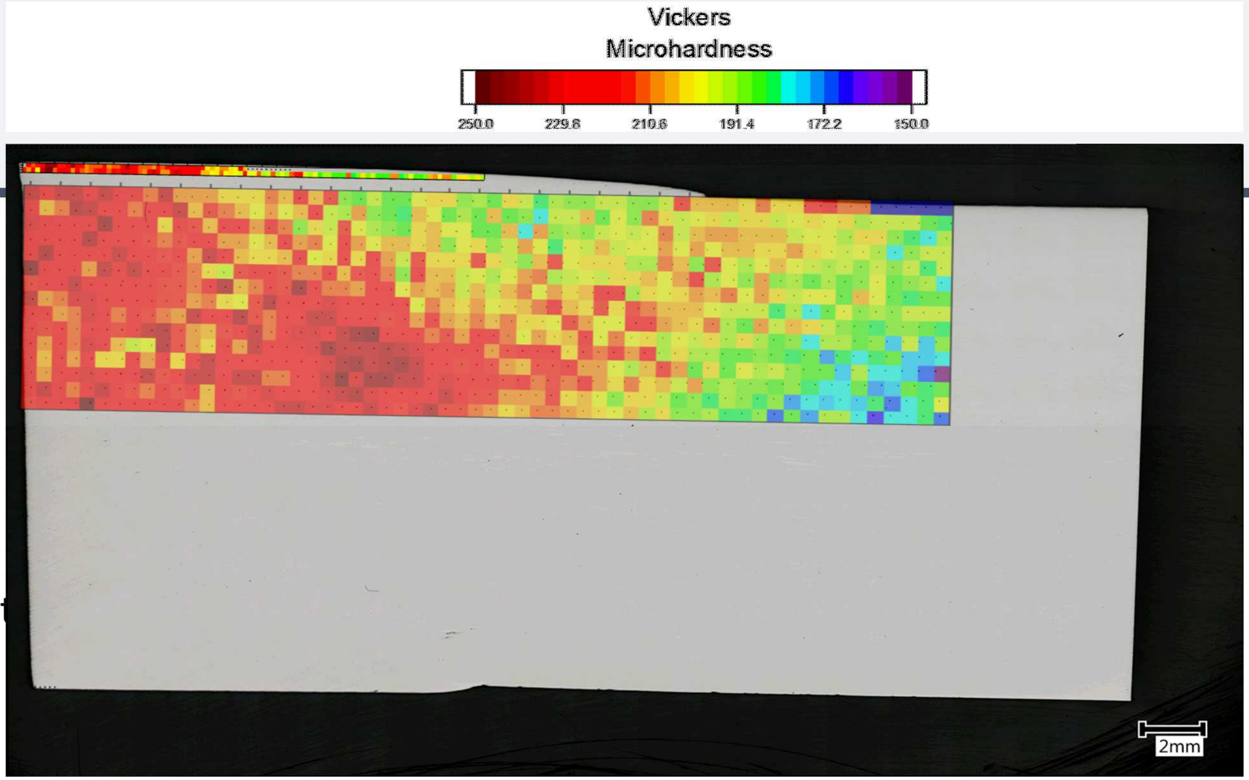

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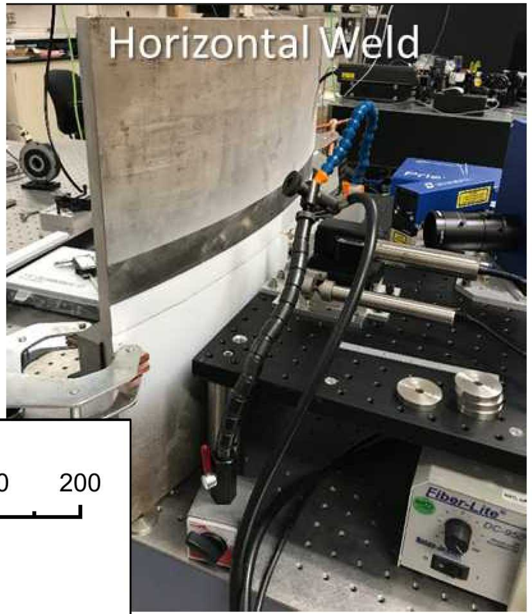
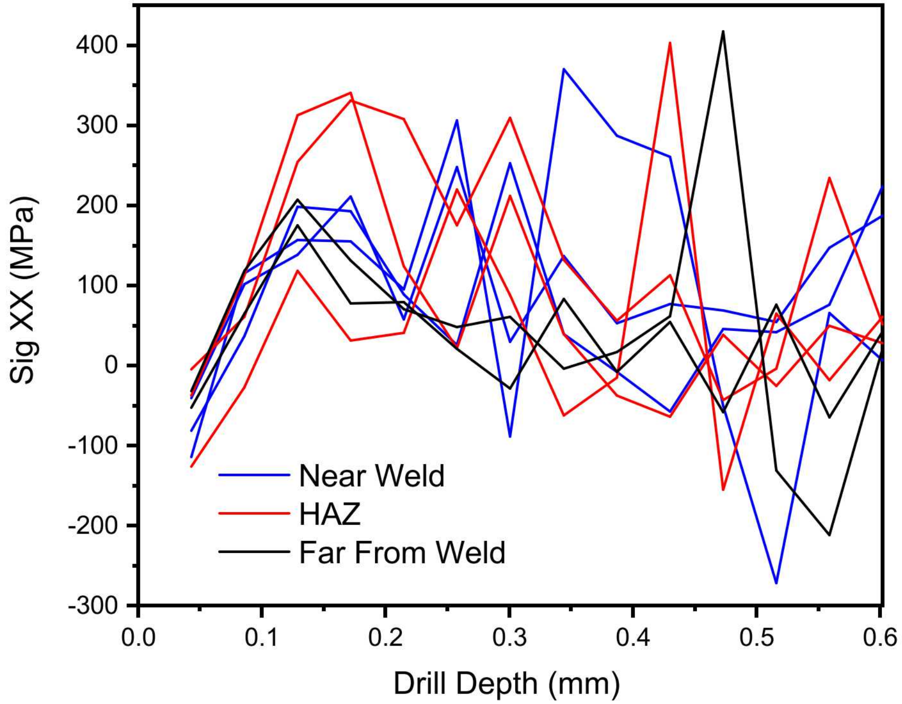
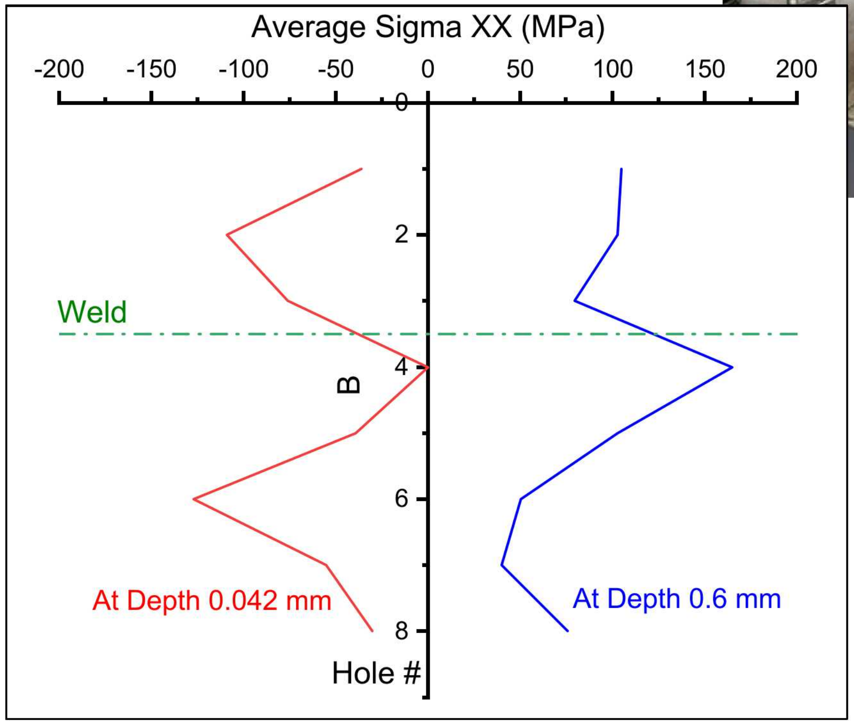


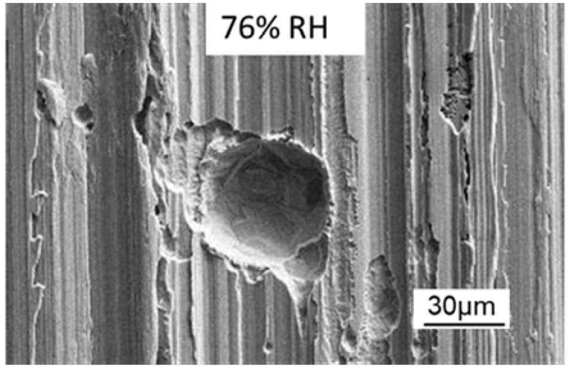




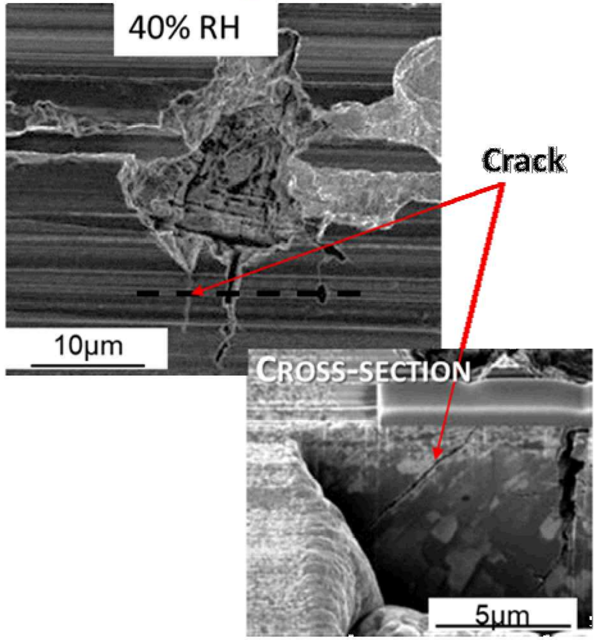
Surface Environment:
Concentrated Brines ($MgCl_2$): Cathode Size

- Experiments:
  1. Droplet exposures
     - Varying size/ brine droplet exposure with in-situ imaging and post-mortem pit analysis
  2. Evaluate possible change in mechanism of cathodic reaction (ORR to HER) and possible H uptake in concentrated $MgCl_2$ brines
     - Full immersion
     - Under a droplet/ thin film while pitting
  3. In-Situ Raman detection of cathodic precipitates in concentrated $MgCl_2$ brines
- Why:
  - Establish relationship between max pit size and cathodic coverage (*further parameterize model*).
  - Investigate additional factors influencing pit morphology and subsequent pit-to-crack transition.
    - Understand micro-crack formation under highly concentrated brines, is this due to HER, H uptake, and embrittlement?
  - Establish influence of cathodic precipitates on the pitting process
- Who/What else it feeds:
  - Determine important environments and significance of cathodic control for pitting and crack initiation/ growth studies at SNL and PNNL.
- What is needed
  - Data on canister-relevant salt loads, compositions, and distributions to better determine relevant surface environments for testing/ modeling.

High RH: NaCl rich brine



Low RH: $MgCl_2$ rich brine



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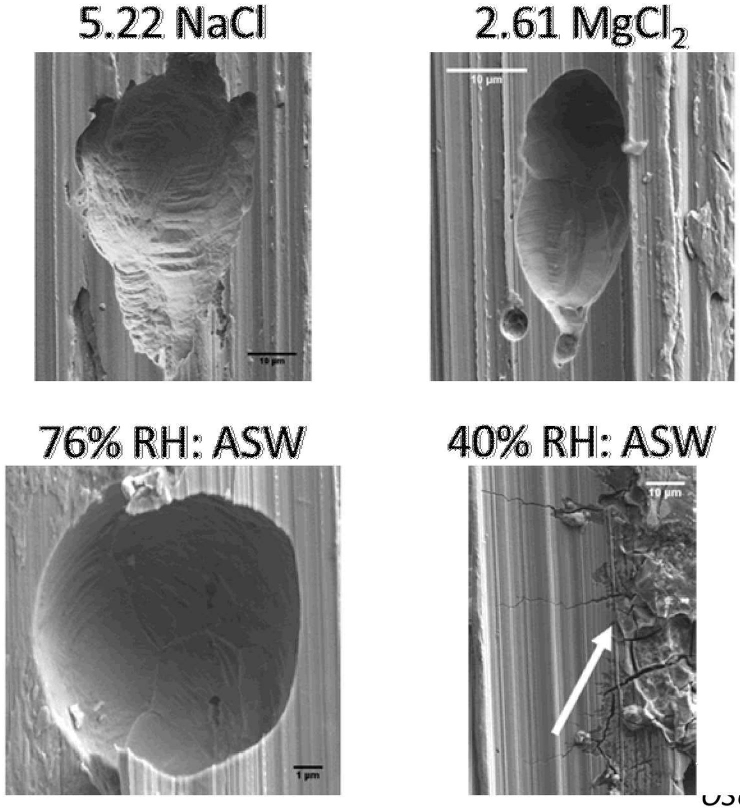
Initial comparison: Various full immersion brines



OSU/ Furey

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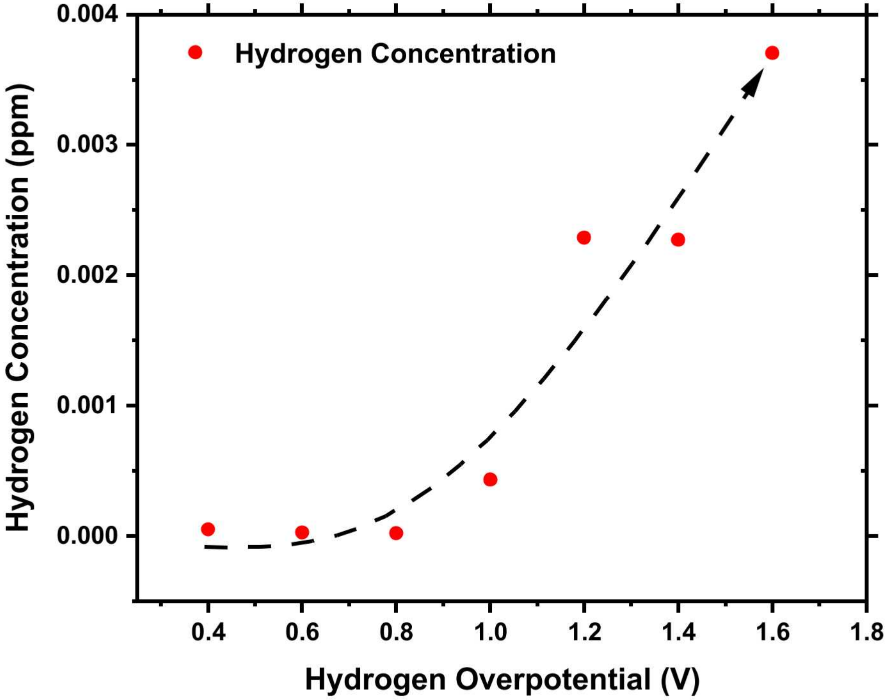
Full Immersion Charging Calibration
48 h, 0.6M NaCl Charge



Preliminary Results

SNL/ Montoya

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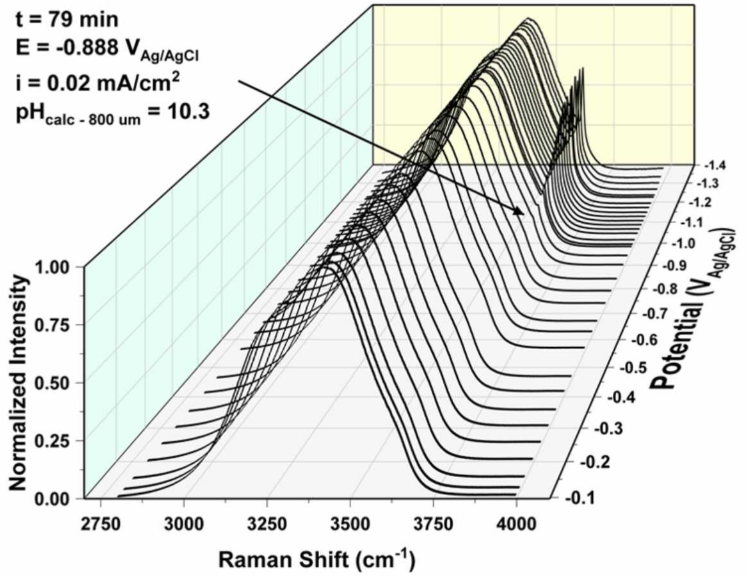
In-situ Raman of SS under cathodic polarization in $MgCl_{2(aq)}$.



UVA/ Katona

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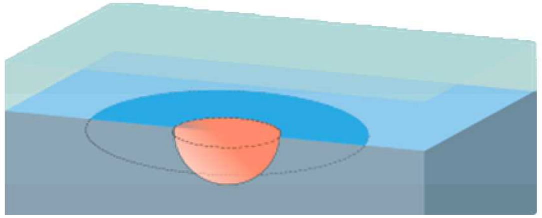
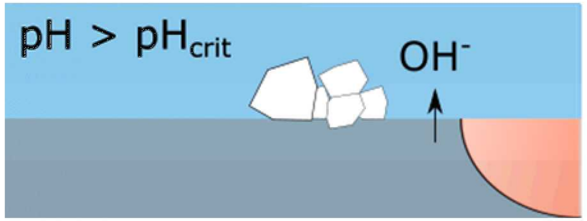
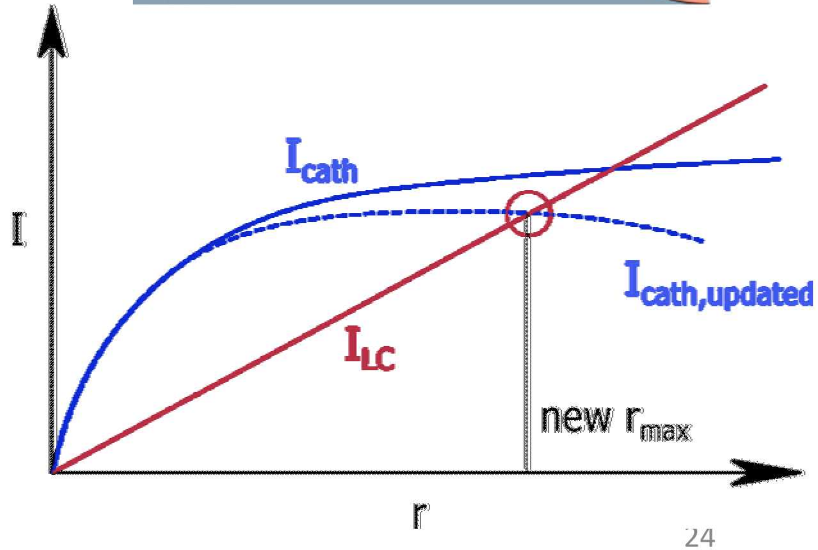
Input effect of cathodic precipitate into max pit size model





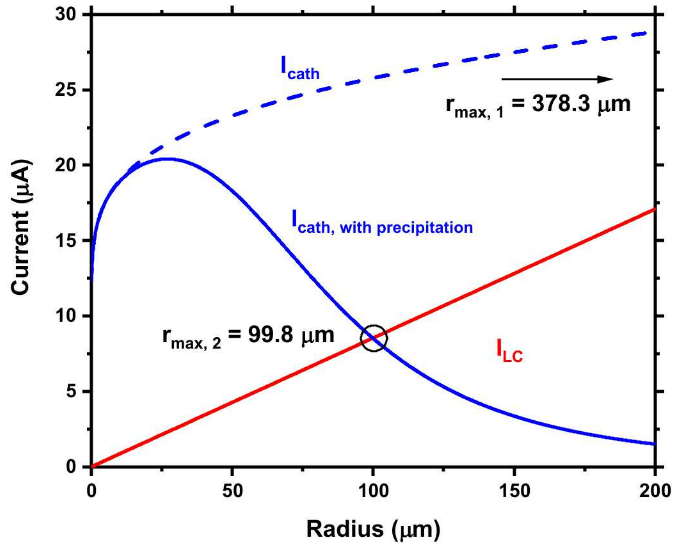


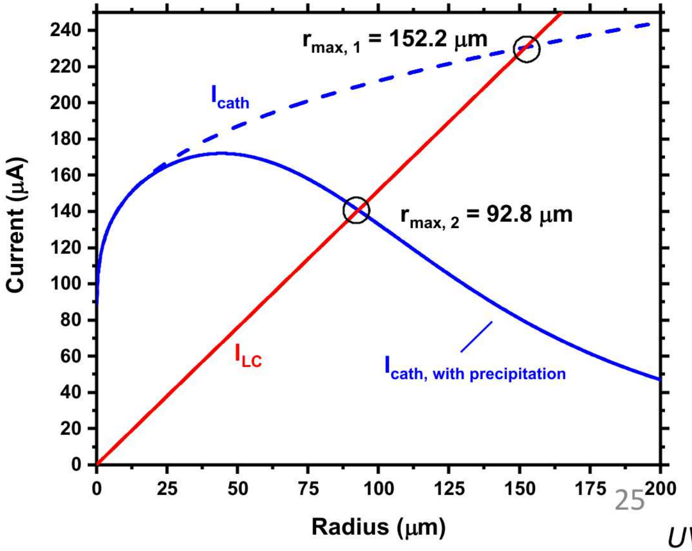
UVA/ Katona

SURFACE ENVIRONMENT:
CONCENTRATED BRINES ($MgCl_2$): CATHODE SIZE

- Experiments:
  1. Droplet exposures
     - Varying size/ brine droplet exposure with in-situ imaging and post-mortem pit analysis
  2. Evaluate possible change in mechanism of cathodic reaction (ORR to HER) and possible H uptake in concentrated $MgCl_2$ brines
     - Full immersion
     - Under a droplet/ thin film while pitting
  3. **In-Situ Raman detection of cathodic precipitates in concentrated $MgCl_2$ brines**
- Why:
  - Establish relationship between max pit size and cathodic coverage (*further parameterize model*).
  - Investigate additional factors influencing pit morphology and subsequent pit-to-crack transition.
    - Understand micro-crack formation under highly concentrated brines, is this due to HER, H uptake, and embrittlement?
  - Establish influence of cathodic precipitates on the pitting process
- Who/What else it feeds:
  - Determine important environments and significance of cathodic control for pitting and crack initiation/ growth studies at SNL and PNNL.
- What is needed
  - Data on canister-relevant salt loads, compositions, and distributions to better determine relevant surface environments for testing/ modeling.

Greater effect in more concentrated brines





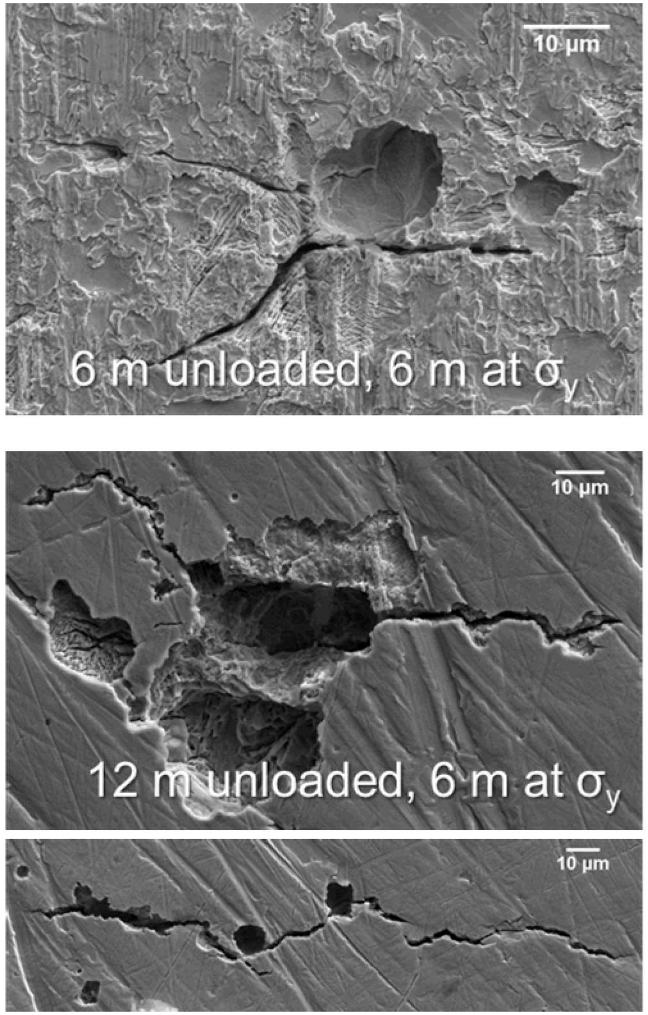
UVA/ Katona

Damage Distribution and Rates:
Pit-to-Crack and CGR

- Experiments:
  - **Factors controlling pit to crack transition**
    - Pit morphology, **environment dependence,** material properties – residual stress
  - CGR experiments:
    - Strain rate dependence
    - Environment dependence f(T, RH, Cl^-, etc.)
    - Material properties
  - Acoustic monitoring of CG in SS
    - Effect of cyclic weather conditions, evolving brine properties
  - Large Plates: Why don't these crack?
    - What are the surface stresses? Center hole drilling stress measurements
    - Metallurgy; microhardness to determine work hardening
- Why:
  - Provides information on rate determining factors for pit to crack transition and crack growth rates to enhance overall predictions
  - Establish methods for monitoring crack growth under atmospheric exposures (relevant to canister environments)
  - Establishing an understanding of why crack aren't occurring provides further information to enhance lifetime predictions
- Who/What else it feeds:
  - CGR as f(environment/material) as inputs into overall SNL model
  - Provides validation/comparison with PNNL
  - Basis for understanding SRNL large plate tests
- What is needed:

Tensile Bar: Atmospheric Exposure

- 300 µg/cm^2 ASW, 35 ºC, 40% RH



OSU/ Parey

Damage Distribution and Rates:
Pit-to-Crack and CGR

- Experiments:
  - **Factors controlling pit to crack transition**
    - Pit morphology, environment dependence**, material properties – residual stress**
  - CGR experiments:
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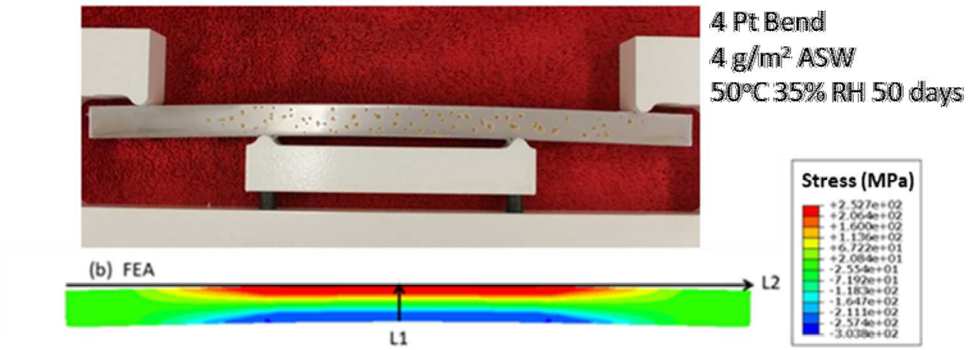
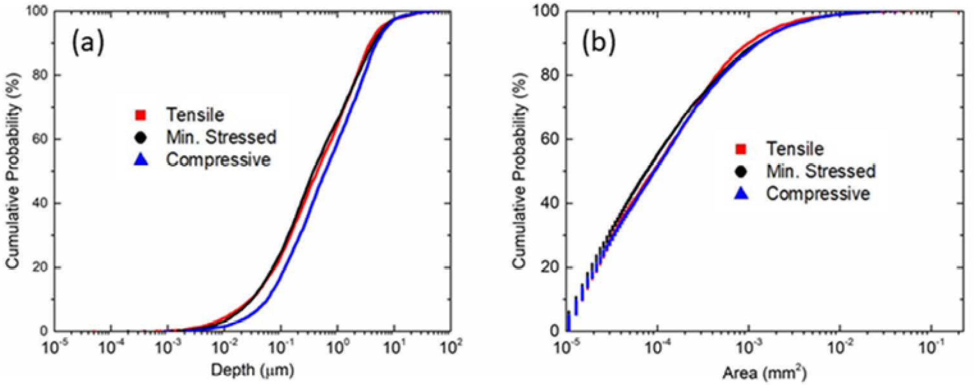
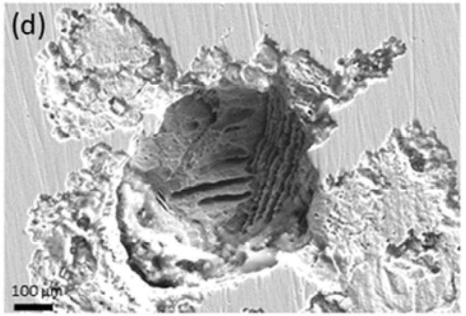
- What is needed:

Brine composition > influence than residual stress



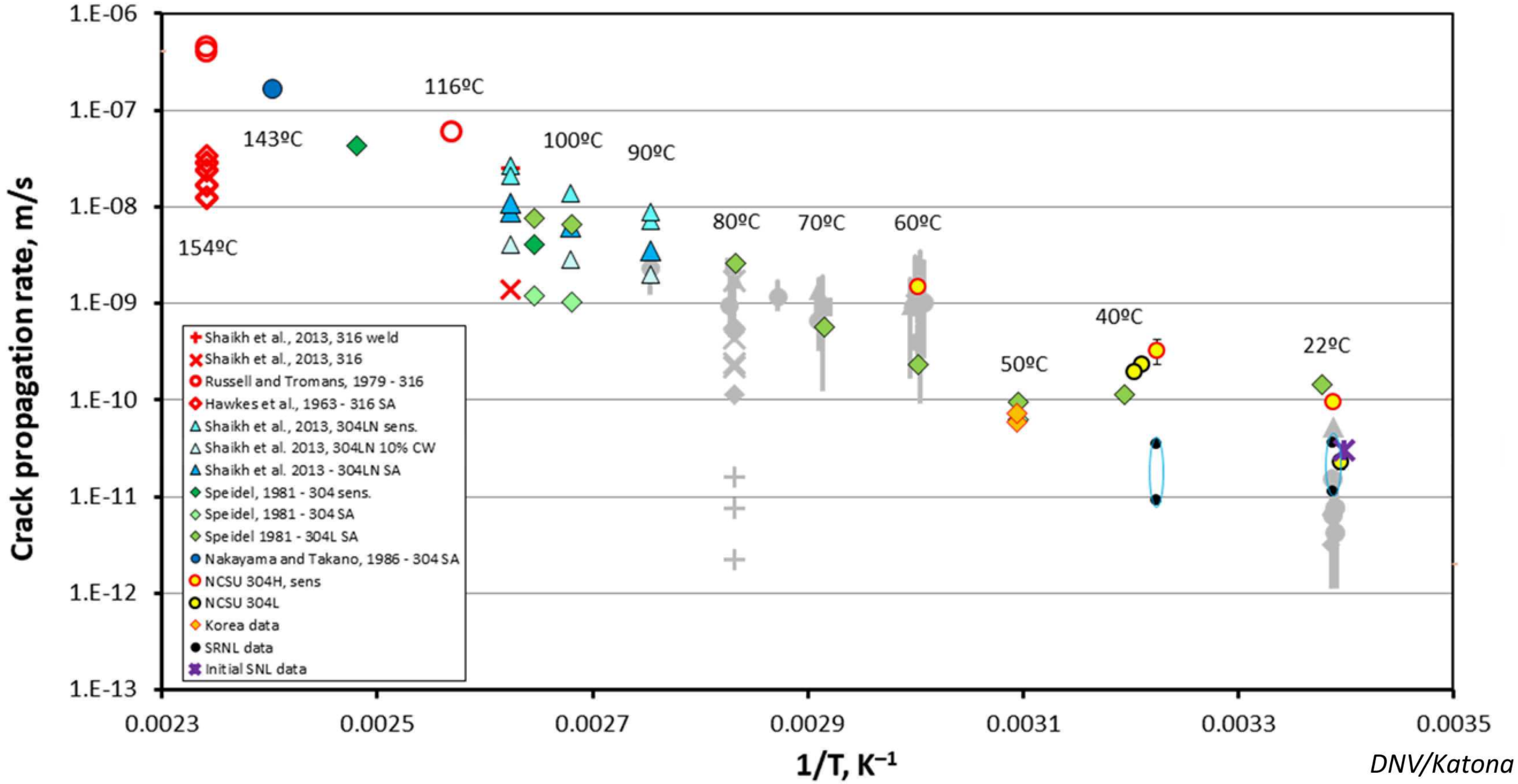




DAMAGE DISTRIBUTION AND RATES:
PIT-TO-CRACK AND CGR

- Experiments:
  - Factors controlling pit to crack transition
    - Pit morphology, environment dependence, material properties – residual stress
  - **CGR experiments:**
    - **Strain rate dependence**
    - **Environment dependence f(T, RH, Cl^-, etc.)**
    - **Material properties**

Preliminary Results

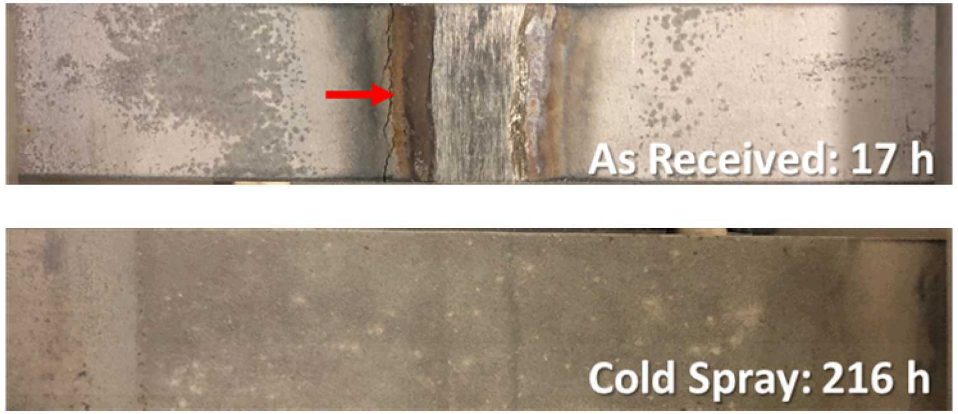


DNV/Katona

MITIGATION STRATEGIES:
FSW AND COLD SPRAY

- Experiments:
  - **Preliminary Evaluation Testing:**
    - **Boiling $MgCl_2$**
    - $FeCl_3$
  - Post corrosion microstructural evaluation
  - Environmental exposure in canister relevant conditions
- Why:
  - Baseline evaluation for susceptibility to SCC
  - Baseline evaluation for susceptibility to pitting
  - Develop a further understanding of mitigation techniques in relevant environment/ exposure conditions
- Who/What else it feeds:
  - Provides evaluation of mitigations repairs from PNNL/ NEUP
  - Establish techniques/ evaluation qualifications for potential corrosion of mitigation for overall program
- What is needed:
  - Test samples – currently provided by PNNL or NEUP

Boiling $MgCl_2$ test



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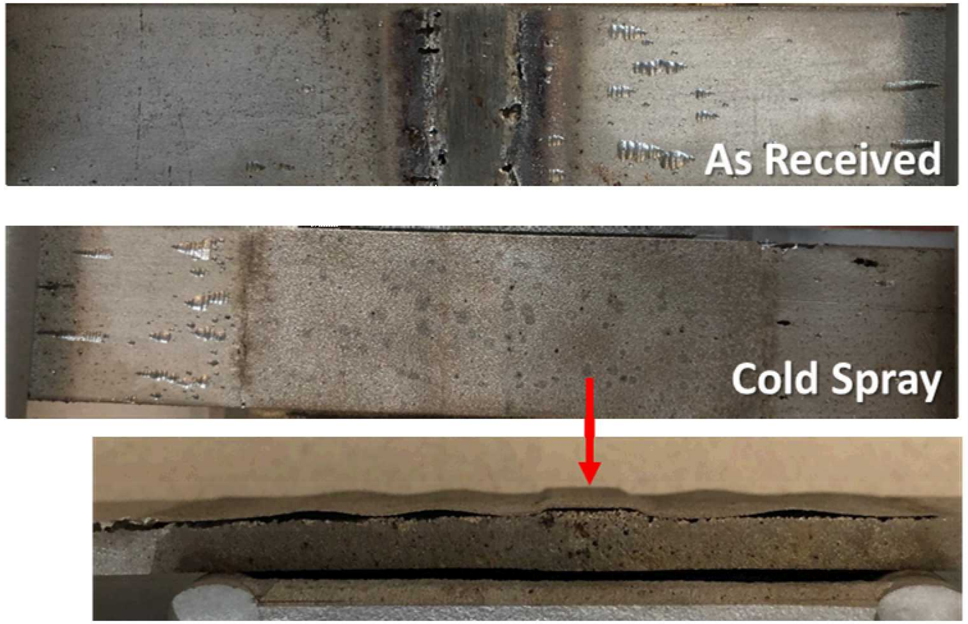
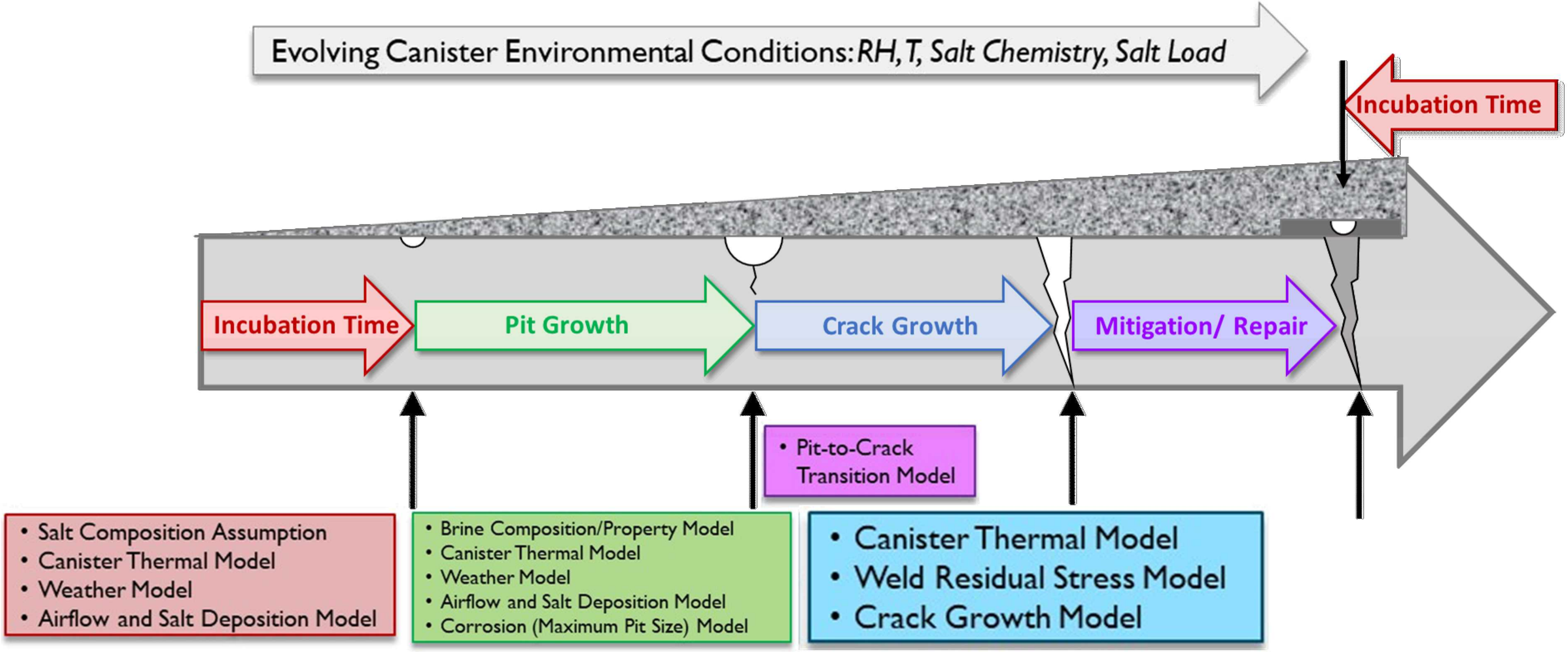
$FeCl_3$ pitting test



Integrated Probabilistic Model for CISCC
Inclusion of Mitigation Strategies/ Repair



Develop and parameterize a mechanistically-based probabilistic SCC model for improved prediction of canister performance.