“Overview and Progress on the Laboratory Experiments of Canister Life Prediction from Pitting to Cracking”

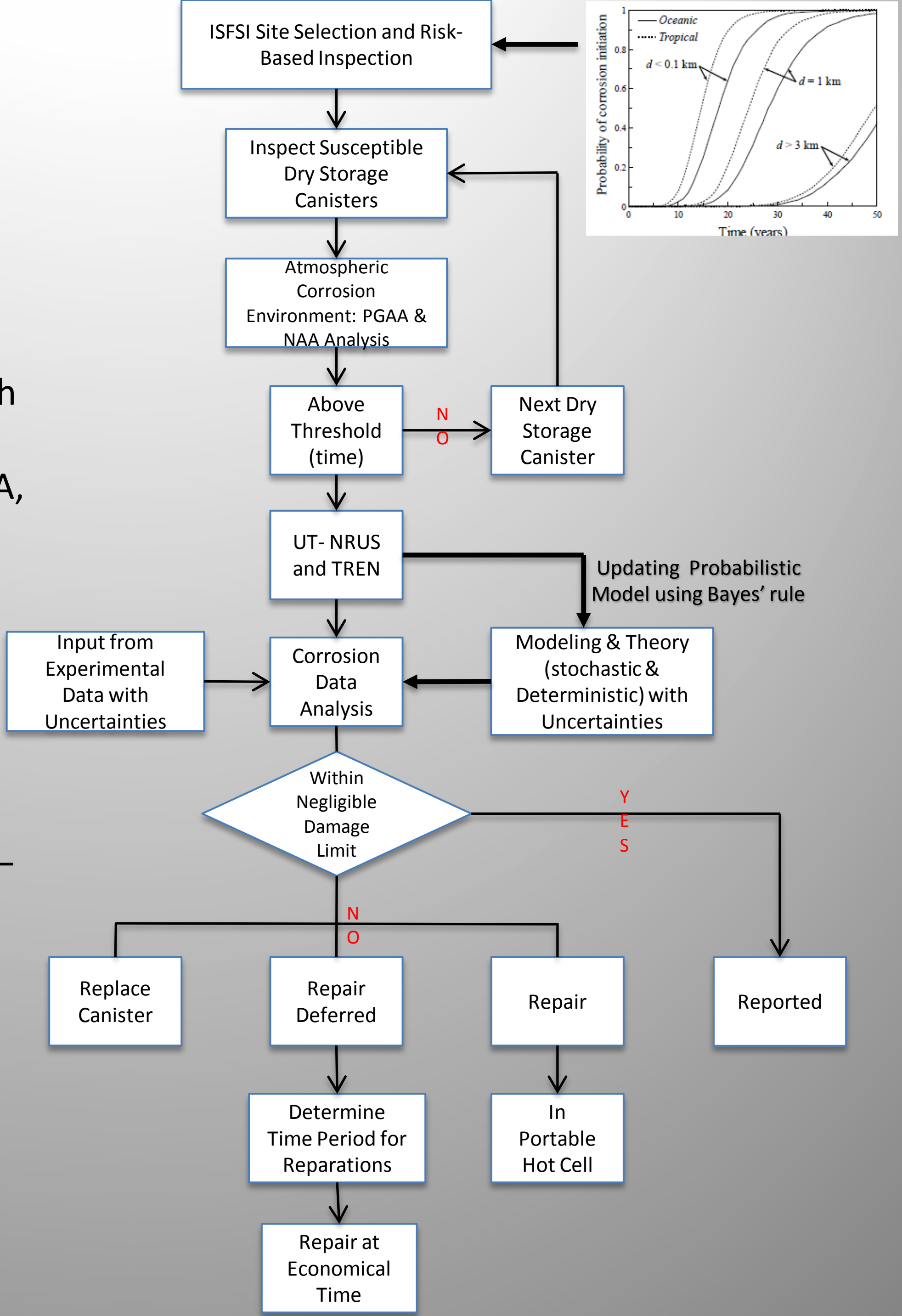
Research Team

Colorado School of Mines
North Carolina State University
University of South Carolina
Sandia National laboratory
Los Alamos National Laboratory
Argonne National Laboratory
Westinghouse (Formerly CB&I)

FY16 Used Fuel Disposition Annual Working Group Meeting
University of Nevada, Las Vegas, June 6-9, 2016

Program start: November 2015

- Corrosion behavior with marine environment – Published and On site data
- Deterministic and stochastic modeling with uncertainties – Risk based Inspection plan
- Detection and detection limit – PGAA, NAA, NRUS, TREN
- Susceptible microstructure simulation (for use by the different collaborators)
- Stresses and pit experimentation and modeling
- Pit/crack characterization
- Crack growth rate – K_{ISCC}/J_{ISCC}
- Crack front environment characterization – Synchrotron in situ (corrosion chamber) XRT
- Data generation/transmittal/sharing /analysis/ storage



PGAA – Prompt Gamma Ray Activation Analysis
NAA – Neutron Activation Analysis
NRUS – Non Linear Resonance UltraSound
TREN – Time Reveral Non Elastic Wave

CISCC Detection and Inspection

Physical simulation of accelerated environment for incubation time and crack initiation experiments:

Microstructure simulation

- Heat treatment
- Thermal model

Stress simulation

- Load stress in Mock up canister
- Weld region
- 3D stress measurement on the mock up (Sandia).

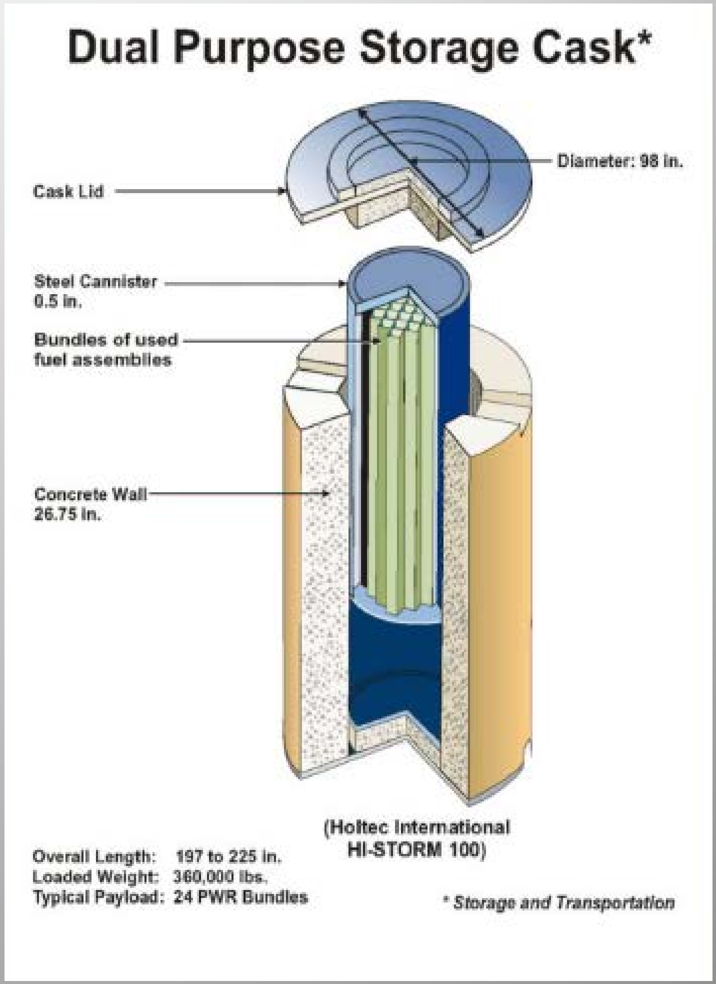
Accelerated corrosion test

- Simulated crack susceptible microstructure
- Four point bending sample with fine notch at simulated stress in mock up
- Elevated temperatures
- Salt mist environment
- K_{ISCC}/J_{ISCC} measurements
- Crack tip characterization

Novel approach to minimize the uncertainty in CISCC prediction.

Sources of Stresses

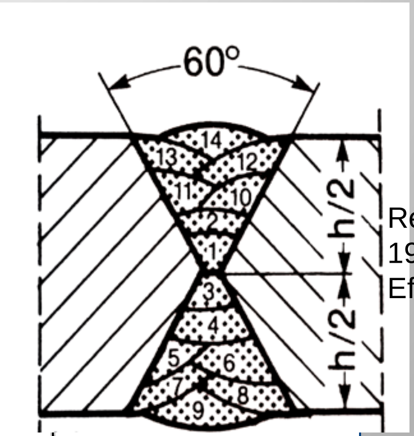
- Manufacturing stages:
  - Machining
  - Rolling
  - Bending
- Fabrication: e.g., Welding
  - Thermal property
  - Phase transformation, e.g., quasi martensite, and anode tip compound formation
  - Constraint
  - Weld procedure design
- In-service condition: air flow, temperature, pressure, and marine environment (salt load, relative humidity, …)



Welding Procedure Information

Detailed information about the weld procedure is needed for an accurate simulation of weld residual stress.

Double-V groove welds



Ref: D. Radaj, 1992. Heat Effects of Welding

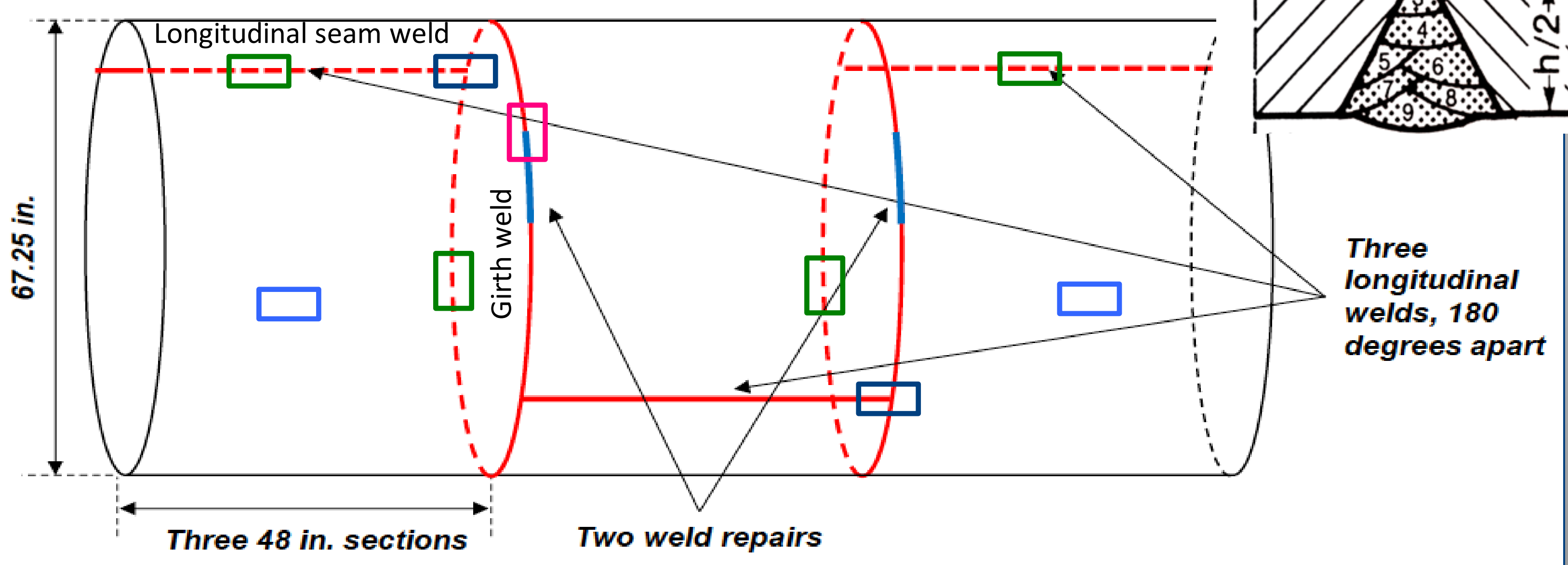
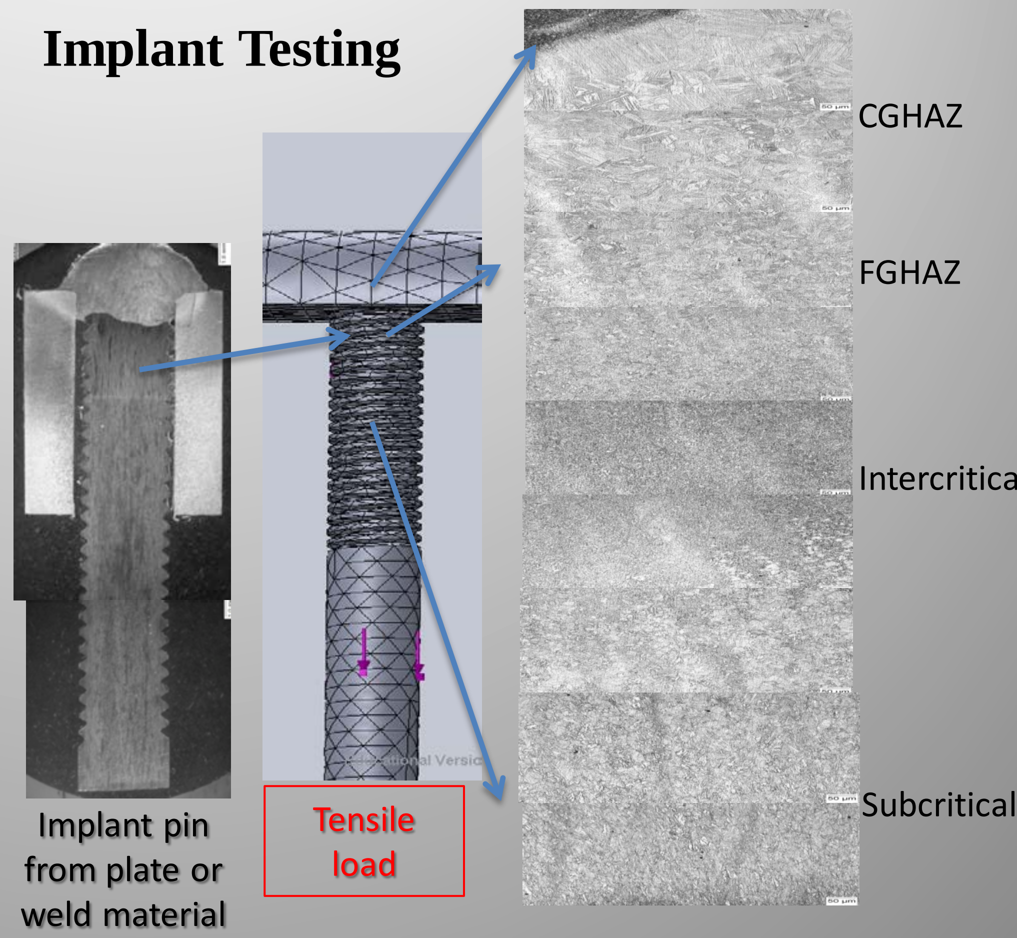


Figure 1: Schematic representation of the full scale mock storage container manufactured at Ranor

Location of Microstructure Most Susceptible to Cracking

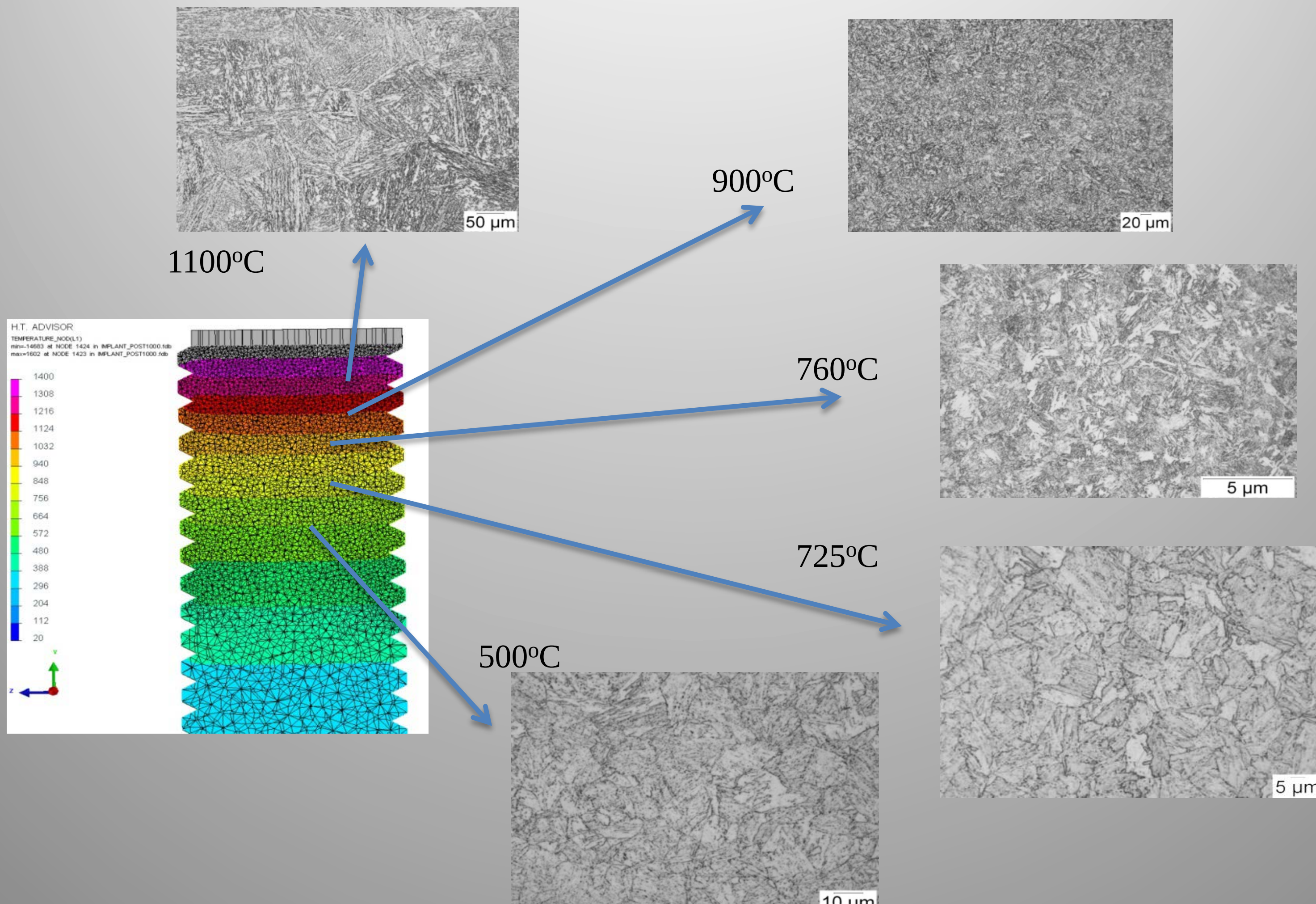
- Weld thermal cycles replicated using Implant testing
- Identify microstructure most susceptible to cracking (as a function of location and temperature) to be reproduced for subsequent fracture mechanics testing and corrosion testing

Implant Testing



Structural steel weld application

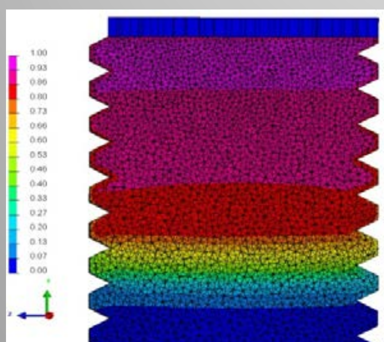
Simulating HAZ and RHZ – From both Furnace and Gleeble System



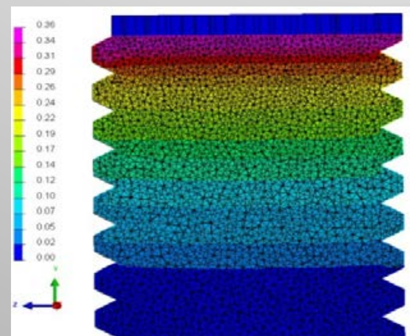
Sysweld Modeling

- FEA simulation to analyze through-thickness stresses and residual stresses developed as a function of plate material, filler material, weld thermal cycle, part and weld geometry, pre- and post-heating condition

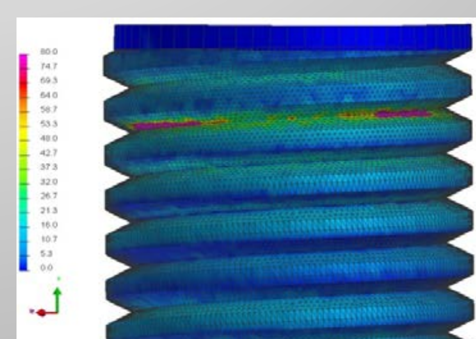
- Sysweld modeling to determine stress and residual stress development in the different HAZ subzones

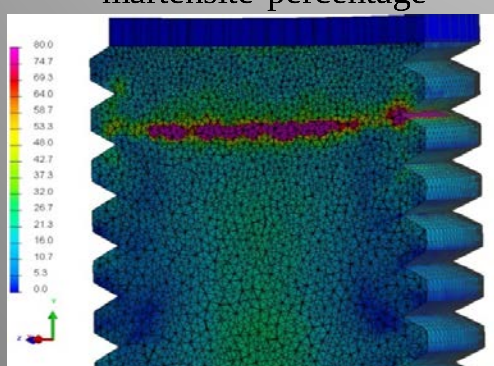


Bainite + Tempered martensite percentage

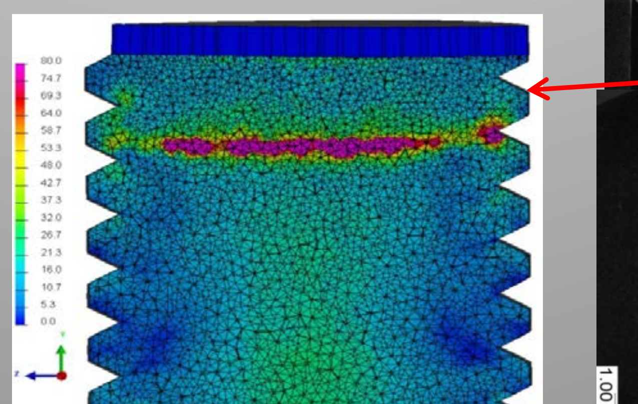


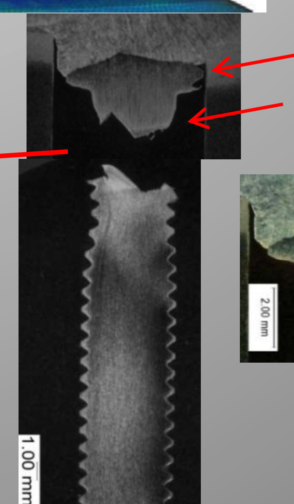
Martensite percentage





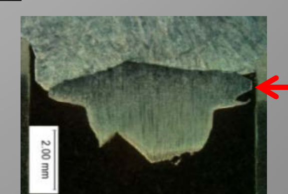
Sub-Surface Crack



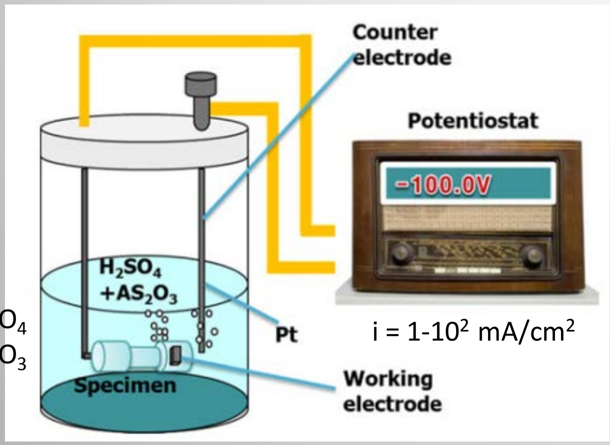


1st thread

2nd thread

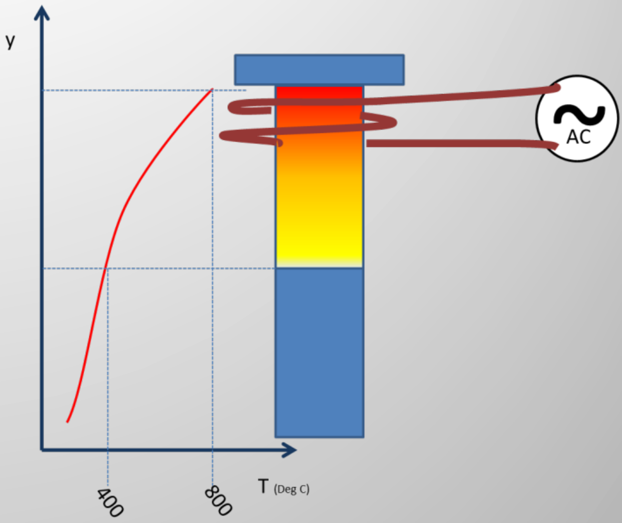


Modified Implant Testing for This Work

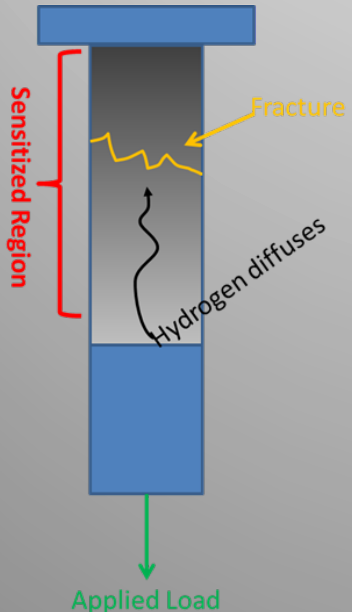


5-6 vol% H_2SO_4
0.5 g/L As_2O_3

a) Schematic Drawing showing electrochemical hydrogen charging of implant specimen. (Bae et al., 2016)



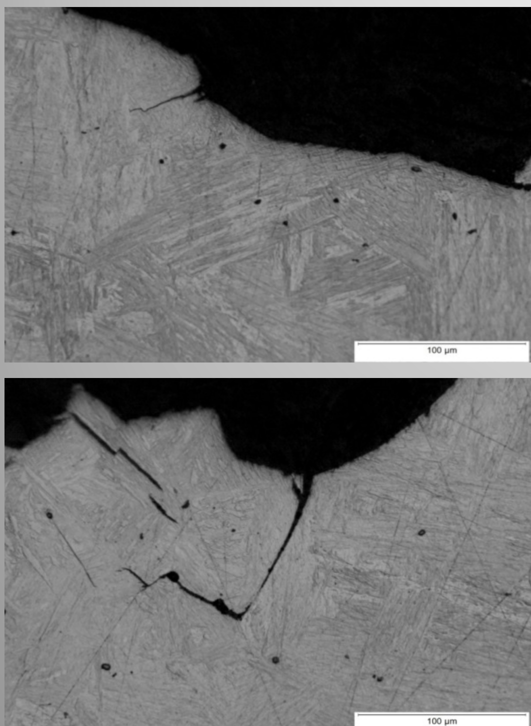
b) Schematic Drawing showing induction heating of implant specimen. By not melting the test specimen no coarse grain heat affected zone is created



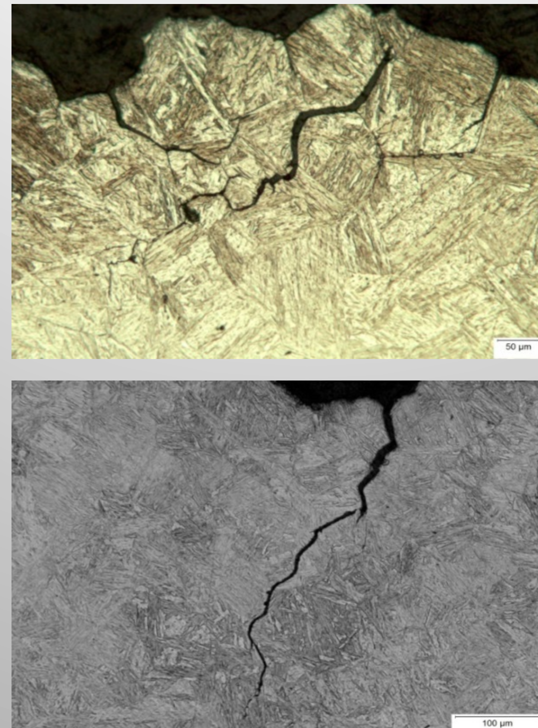
c) Schematic Drawing showing testing of implant specimen.

d) Photograph showing implant testing machine at CSM

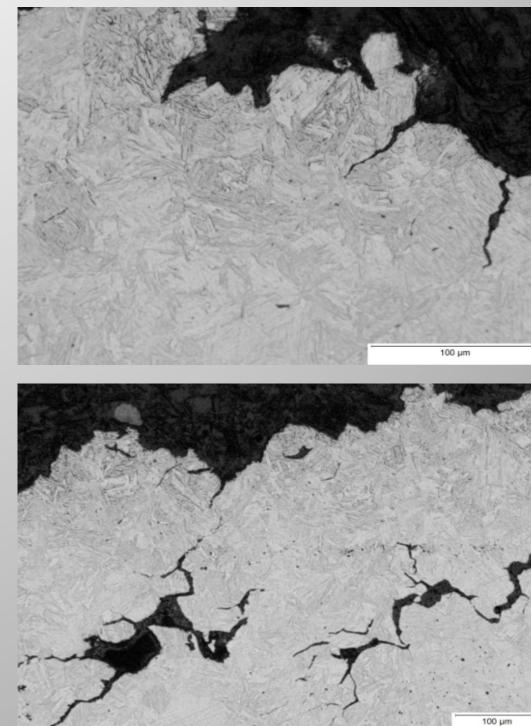
Selected Metallography for Hydrogen Pre-charged & Fractured Samples



Gleeble-simulated
1200°C - 5ppm H_2



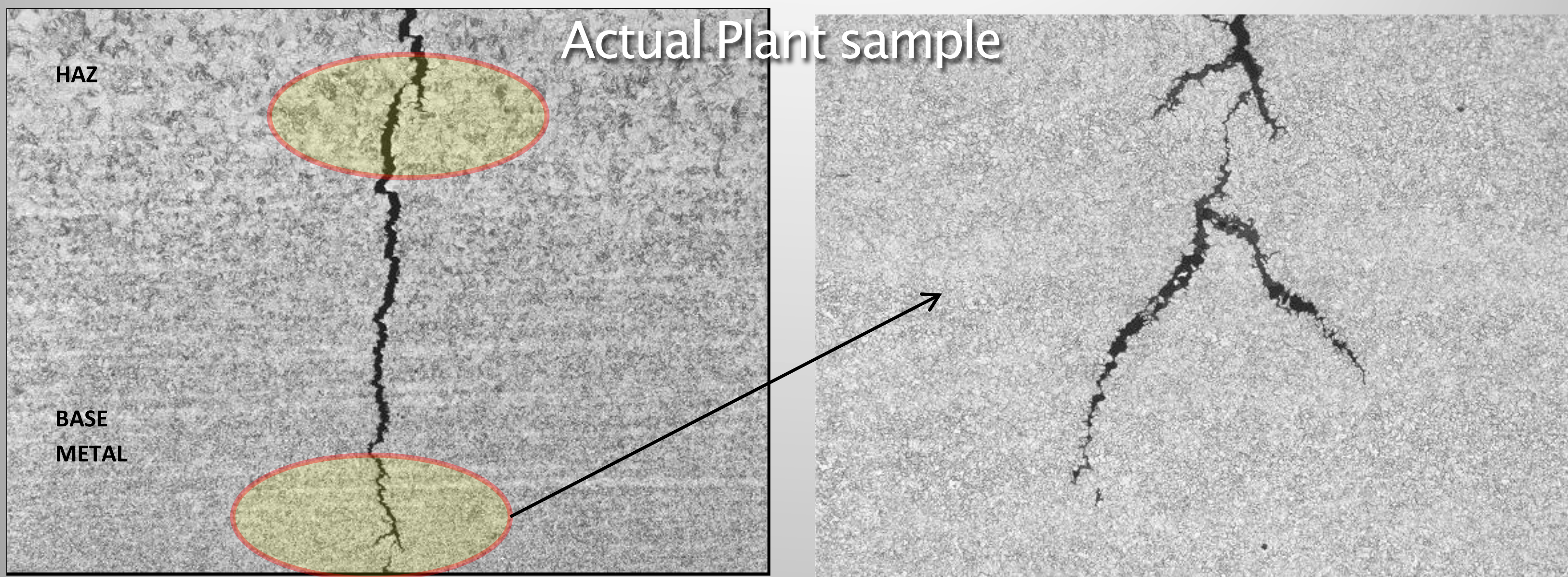
Gleeble-simulated
1100°C - 5ppm H_2



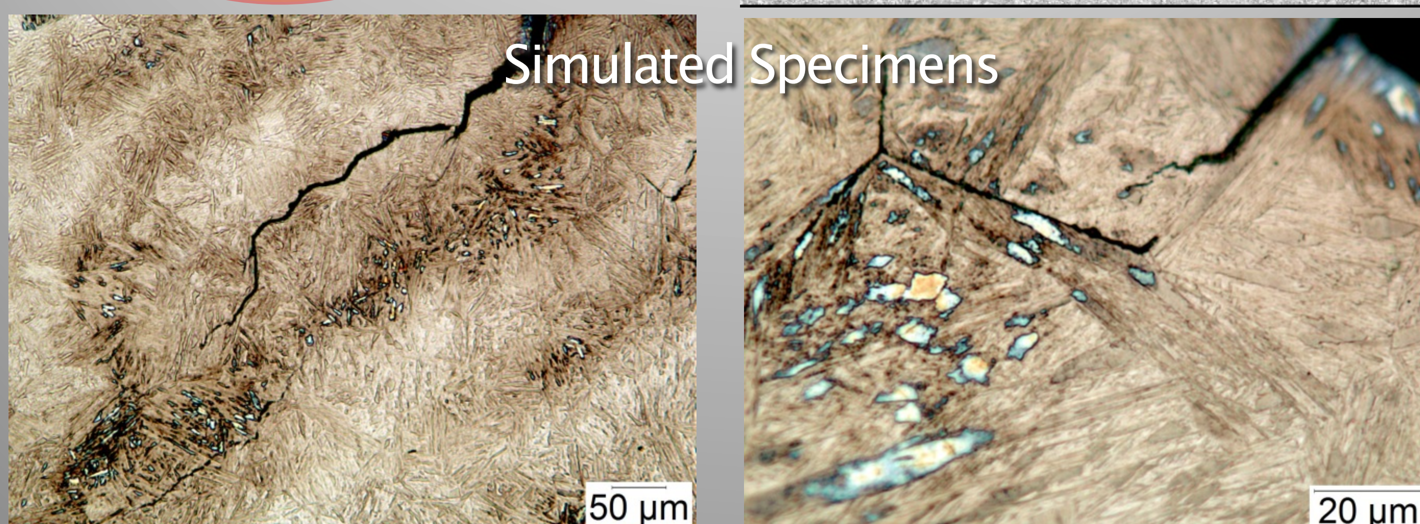
Gleeble-simulated
1000°C - 5ppm H_2

Comparison between Microstructures – Plant and Pre-charged & Fractured Samples

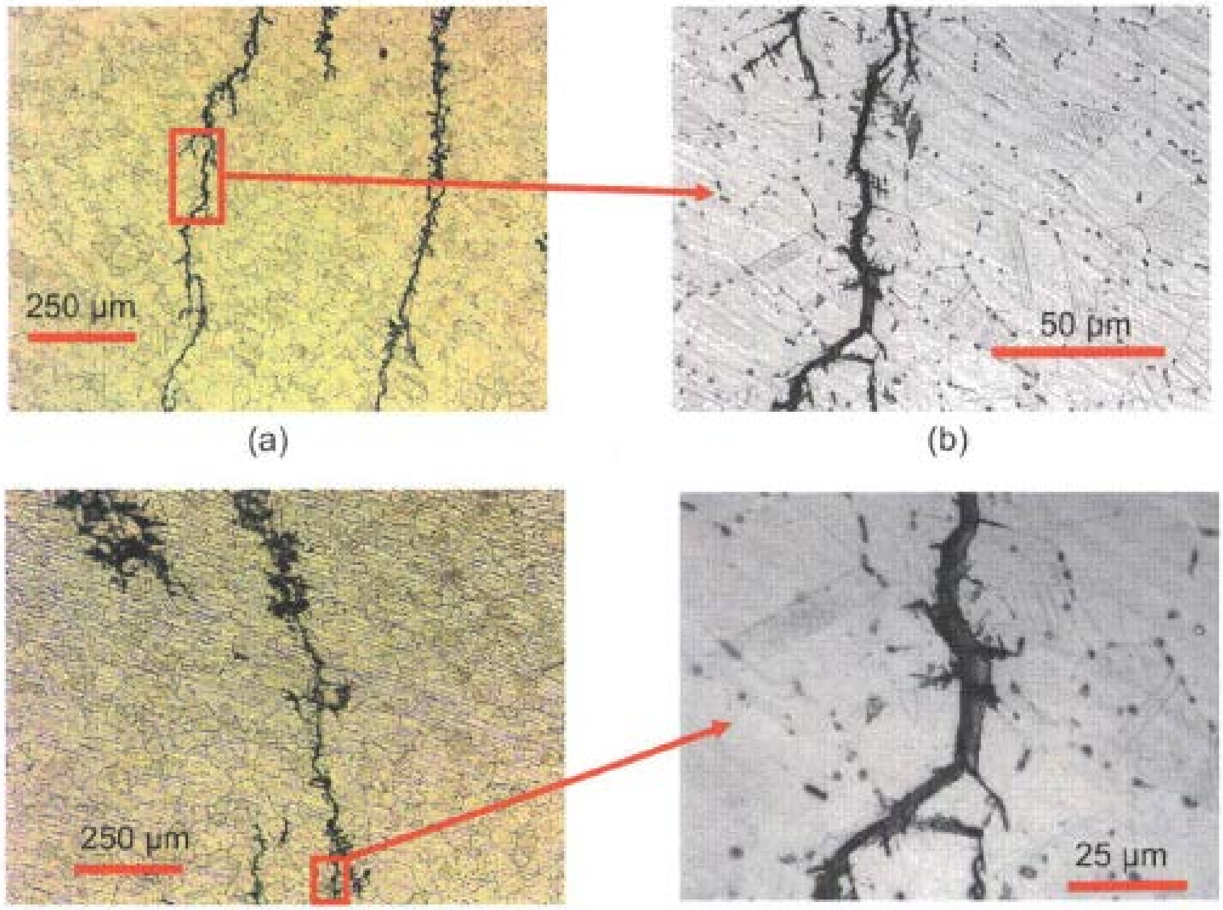
Actual Plant sample



Simulated Specimens



CISCC Crack Morphology



(a) (b) (c) (d)

304 Single U-Bend specimen

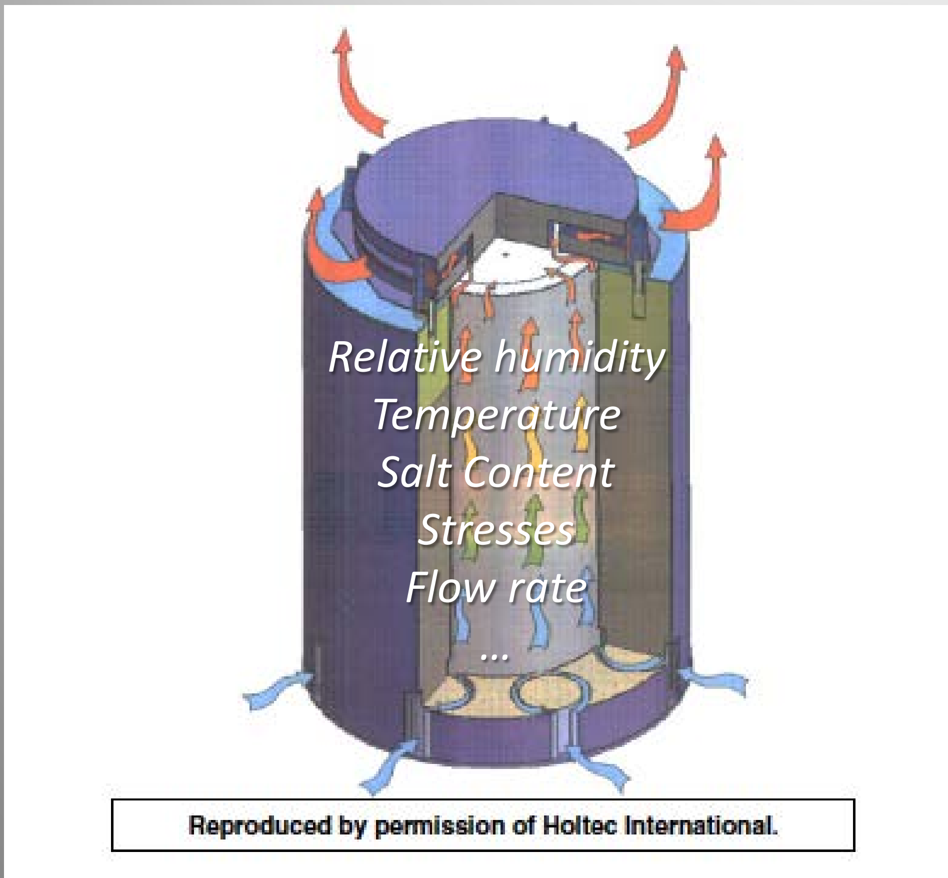
16 weeks of exposure at 43°C in marine environment

304L Single U-Bend specimen

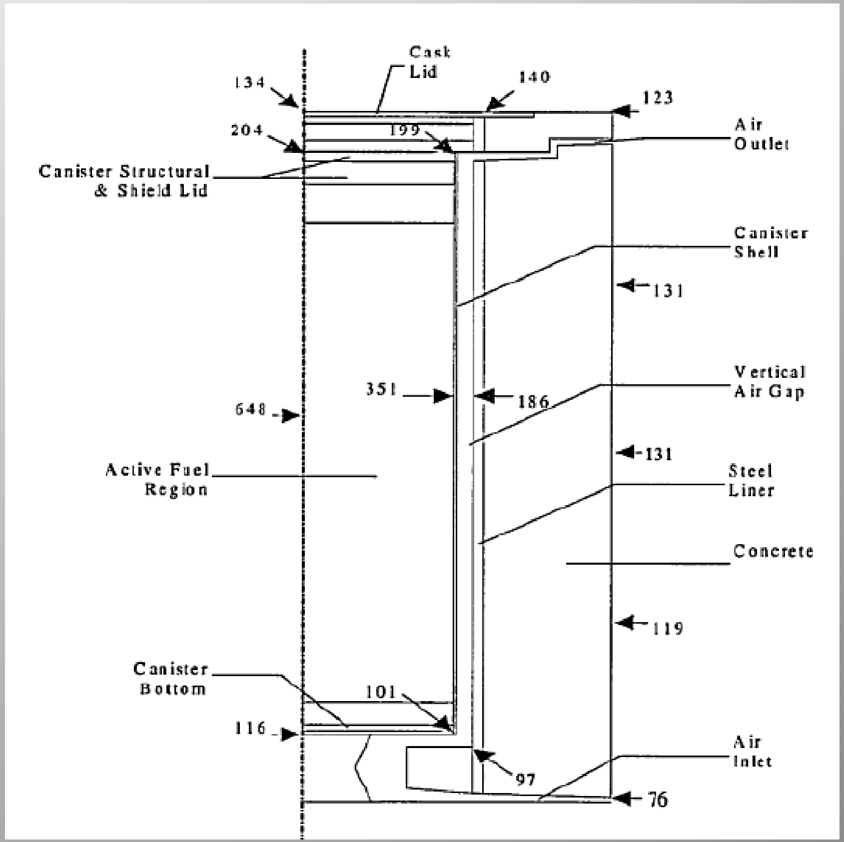
Ref: US NRC 2010 NUREG/CR-7030

In-Service and Experimental Environment Condition

Air flow for a typical vertical canister



UMS canister temperatures (°F) for normal operation at design heat loading (23 kW) [23]



EPRI, 2013, Failure Modes and Effects Analysis (FMEA) of Welded Stainless Steel Canisters for Dry Cask storage Systems

Need for simulated samples to be located on site for in situ exposure.

Incubation time and Crack Initiation – Accelerated Corrosion Experiments

- Four-point bending specimens at various stress levels
- Two surface temperatures, 40ºC and 60ºC, respectively, first coated with “sea salt load” (0-4 g/m³ $MgCl_2$*) in a salt spray chamber and then kept in a relative humidity chambers
- Relative humidity (over a range from 15-60% for 40°C, and over a range of 15-25% at 60°C) controls the chloride content of the deliquesced brine
- Periodic examination of samples to check for pit/crack initiation, e.g. evolution of pit depths over time – data critical for the pit growth model

Relative humidity chamber

Z-Plus Chamber Specifications

	ZP(H) - 8
Workspace Volume	8 Cubic Ft (230 L)
Exterior Dimensions	36"W x 57"D x 76"H (91cm x 145cm x 193cm)
Workspace Dimensions	24"W x 24"D x 24"H (61cm x 61cm x 61cm)
Temperature Ranges	Single Stage: -34°C to +190°C (-30°F to 375°F)
*Temperature Control Tolerance	±0.5°C at steady state condition after stabilization
Humidity Range Optional Range	10% to 98% RH 5% to 98% RH
*Humidity Control Tolerance	±3% RH at steady state conditions after stabilization
Distributed Shelf Load Capacity	110 lbs.

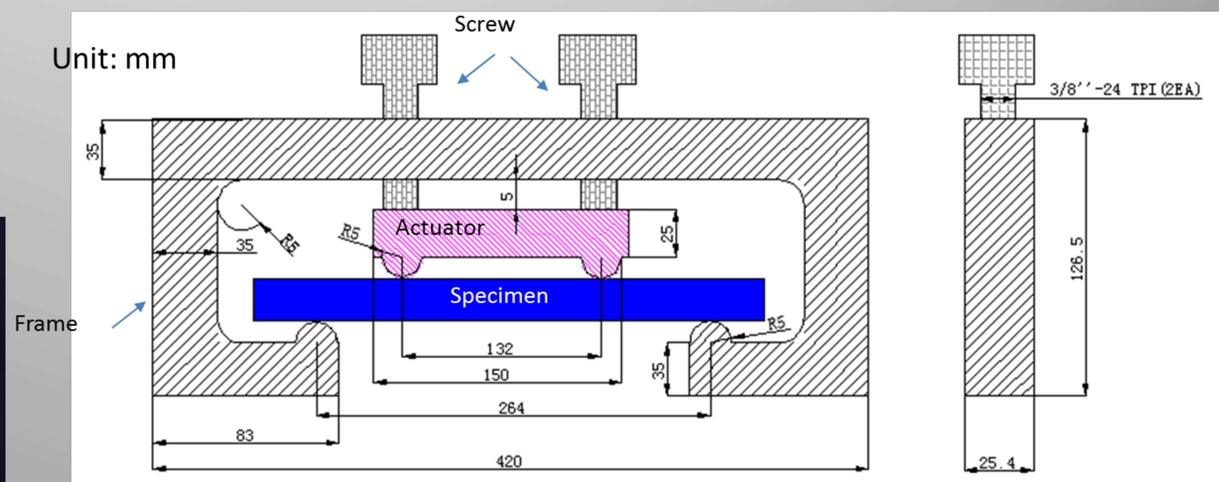
Salt spray chamber

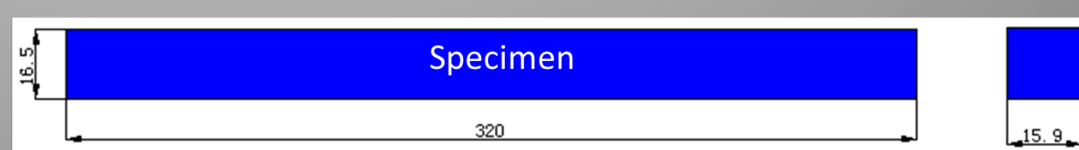
* Concentration to be discussed with SNL scientists

Incubation time and Crack Initiation Experiment in Accelerated Environment

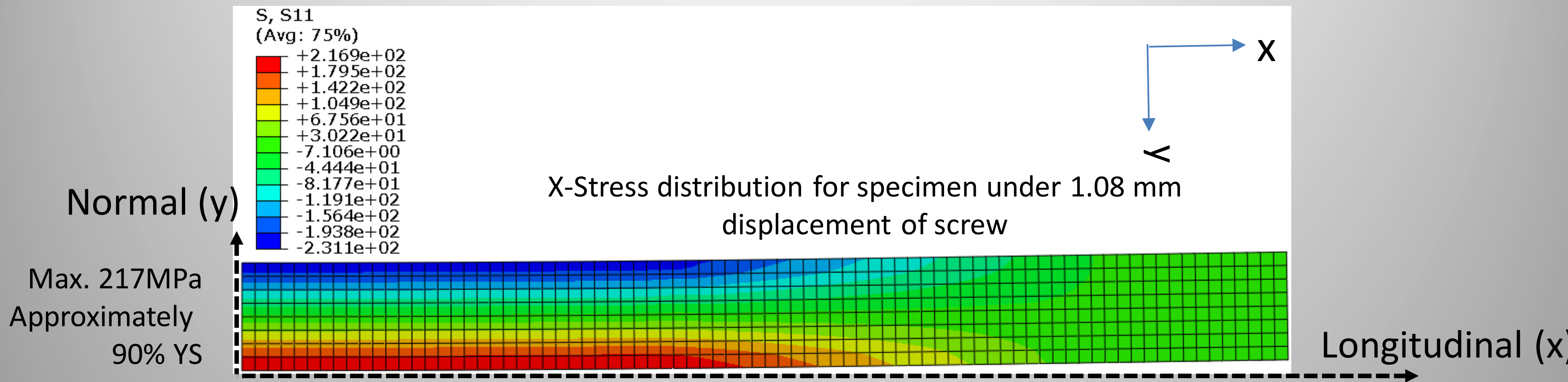
Physical simulation of accelerated environment:

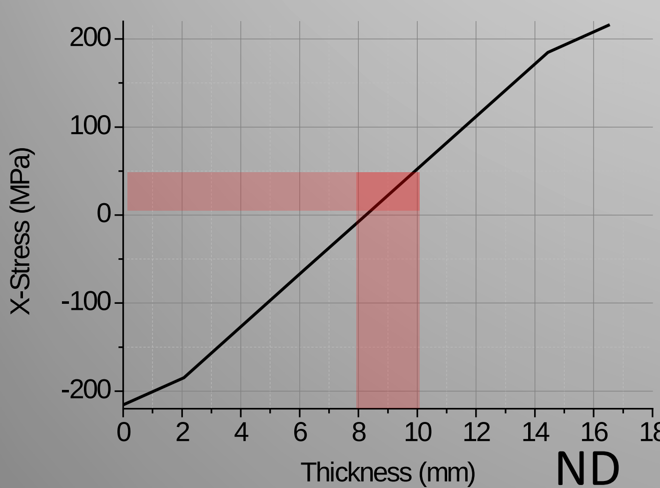
- **Four point bending** specimens
  - Various stress levels can be simulated by a single specimen.
- Hot stage for simulation of elevated temperature environment.

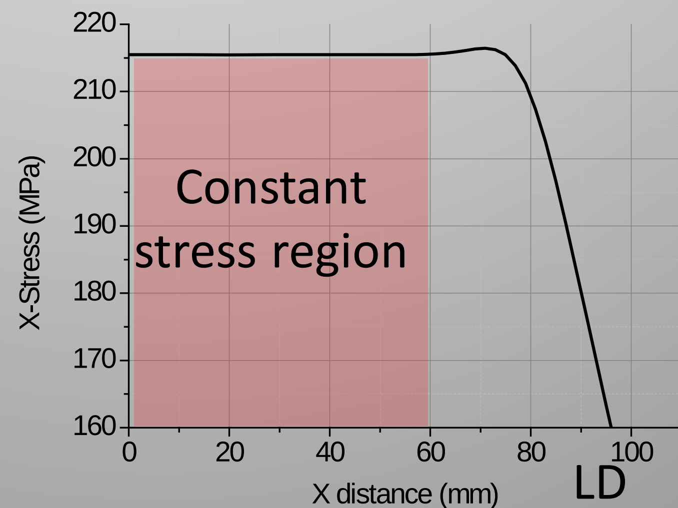




Four-Point Bending Specimen Stress Map: Abacus Simulation





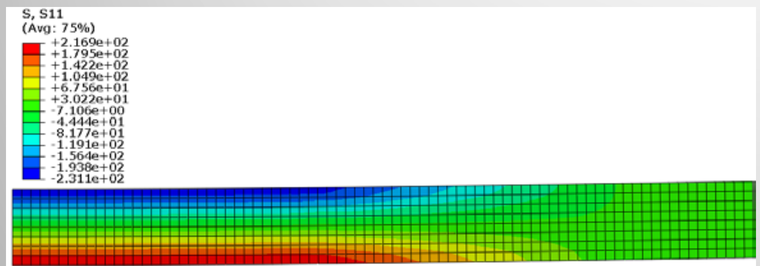


Sampling Area (±25MPa):
2 x 120 mm^2

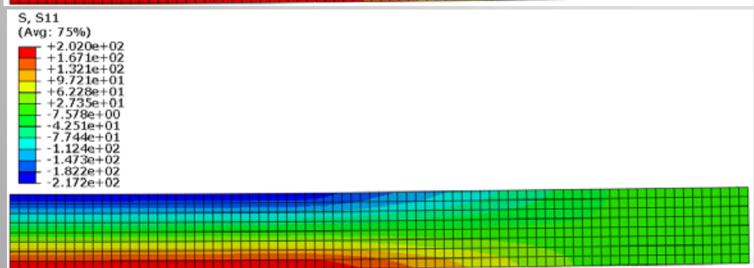
With the tension-compression gradient, a pit density gradient is also expected to exist through plate thickness.

Adjustment of Load due to Thermal Expansion

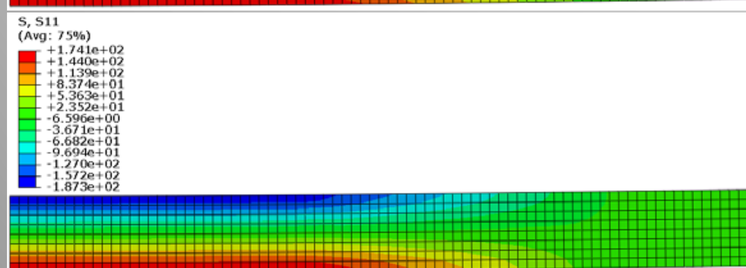
20°C



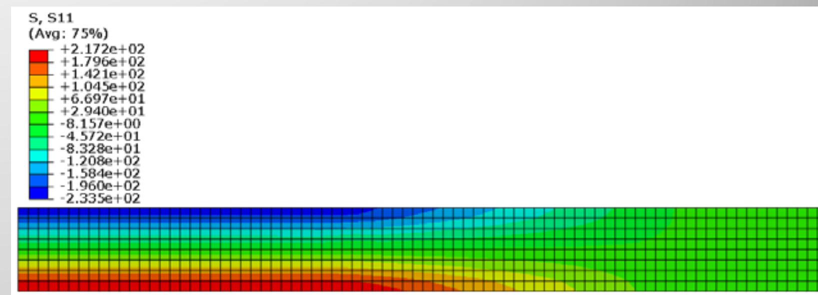
40°C



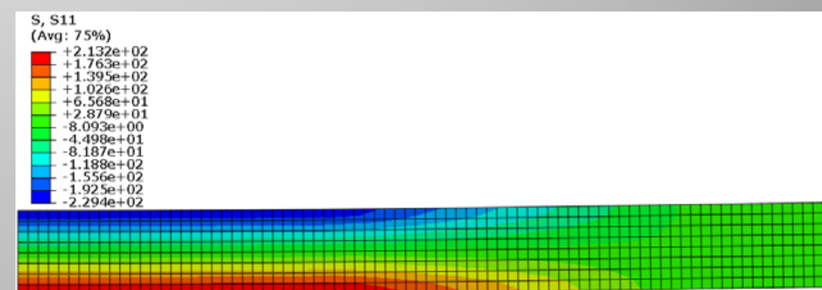
80°C



Displacement load = 1.08 mm



Displacement load = 1.15 mm (40 ℃)

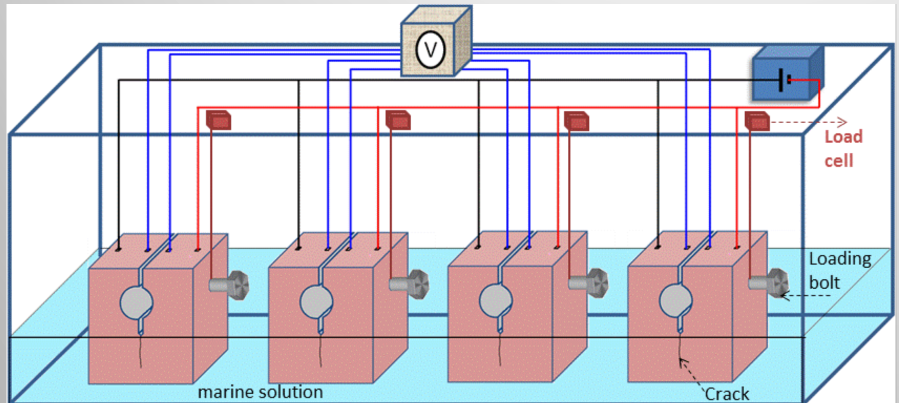


Displacement load = 1.26 mm (80 ℃)

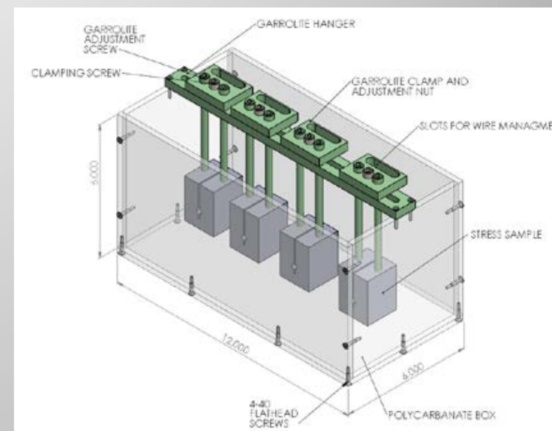
- Under elevated temperature (e.g., 80˚C), the stress on the specimen will be partly relieved due to thermal expansion.
- Load needs to be increased for corrosion test under higher temperatures.

K_{ISCC} Evaluation using Wedge Opening Loading (WOL) Sample

- Stress corrosion cracking in a chamber capable of conducting four (4) wedge opening loading (WOL) samples with crack growth monitoring using DCPD



Corrosion chamber for measurement of K_{ISCC}



- **Purpose**: Determination of K_{ISCC}
- **Determination of K_{ISCC}**

$$K = \frac{(2+a)(0.886+4.64a-13.32a^2+14.72a^3-5.6a^4)P}{B\sqrt{W}(1-a)^{3/2}}$$

$$K = \frac{(30.96-195.8a-730.6a^2+1186.3a^3-754.6a^4)P}{B\sqrt{a}}$$

where $a=a_o/W$; a_o=initial crack length, B=thickness, W=width

- **Step 1**: K_{IC} measured in the absence of corrosive medium.
- **Step 2**: For a given load and crack size such that $K_I < K_{IC}$, testing will be done in marine solution for different time to determine K_{ISCC}.

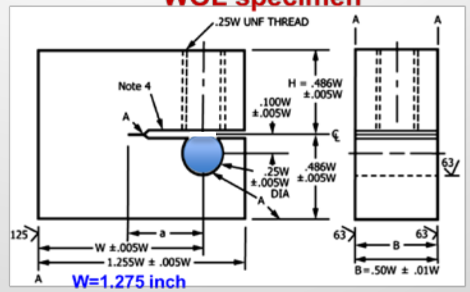
DCPD – Direct Current Potential Drop

K_{ISCC} Evaluation using WOL Sample

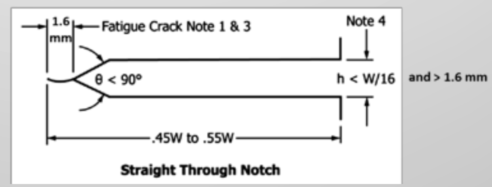
- Stress corrosion cracking experimental set-up at NCSU

- Details of the WOL sample

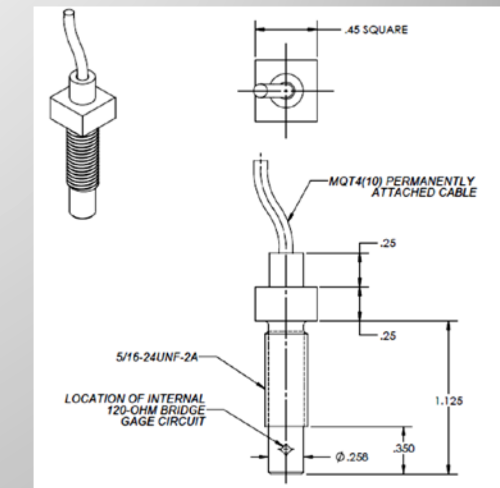
WOL specimen



ASTM E1681 – 03 (2013)

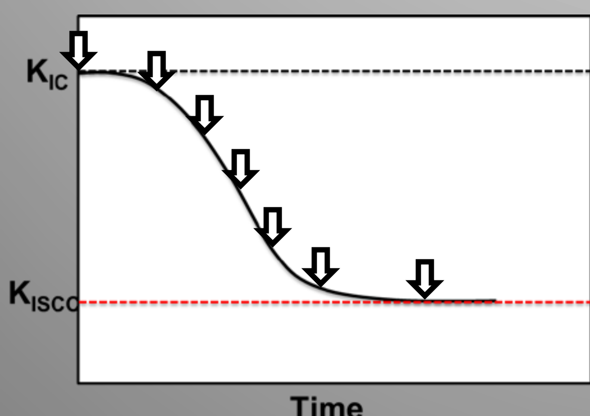


- Instrumented bolt for loading WOL sample and measuring load during test



- Nanovoltmeter for crack-size measurement

- Testing strategy



Expected outcome

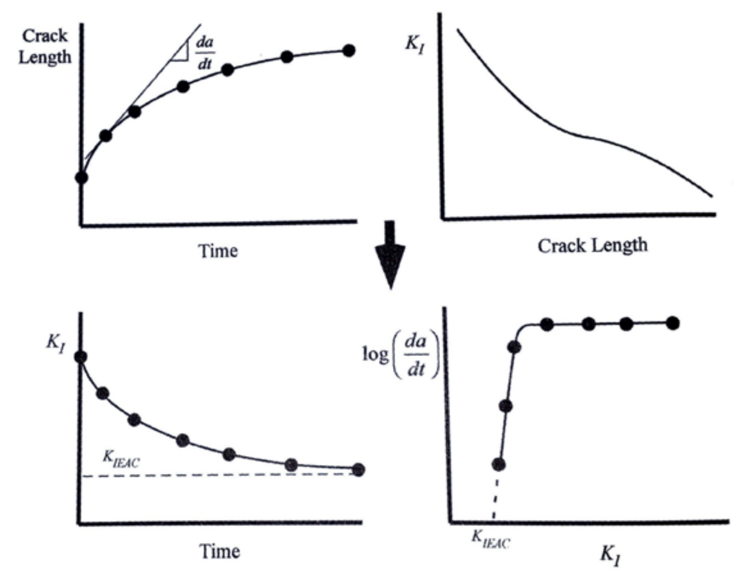


Figure 11.42, Fracture Mechanics: Fundamentals and Applications by T L Anderson

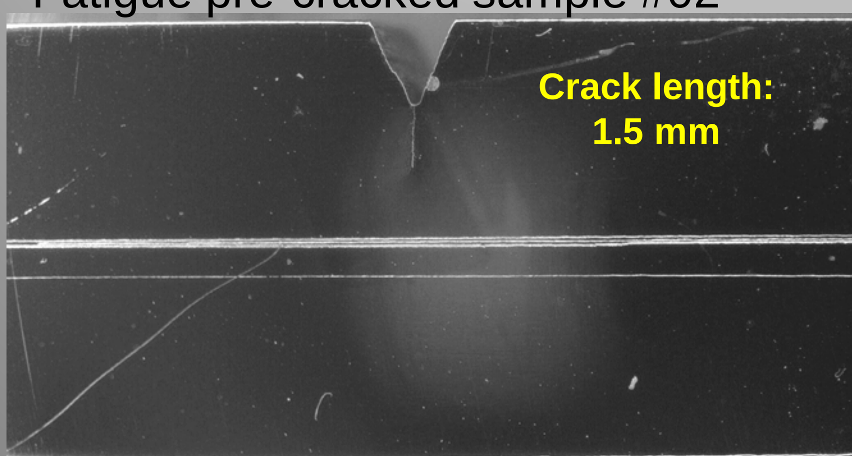
Fatigue Pre-Cracking followed by 3-Point Bending

- Fatigue pre-cracking conditions (ASTM standard followed - ASTM E1820-15, E399-12, E1681-03 (2013)): unit of K_Q: $MPa.m^{1/2}$

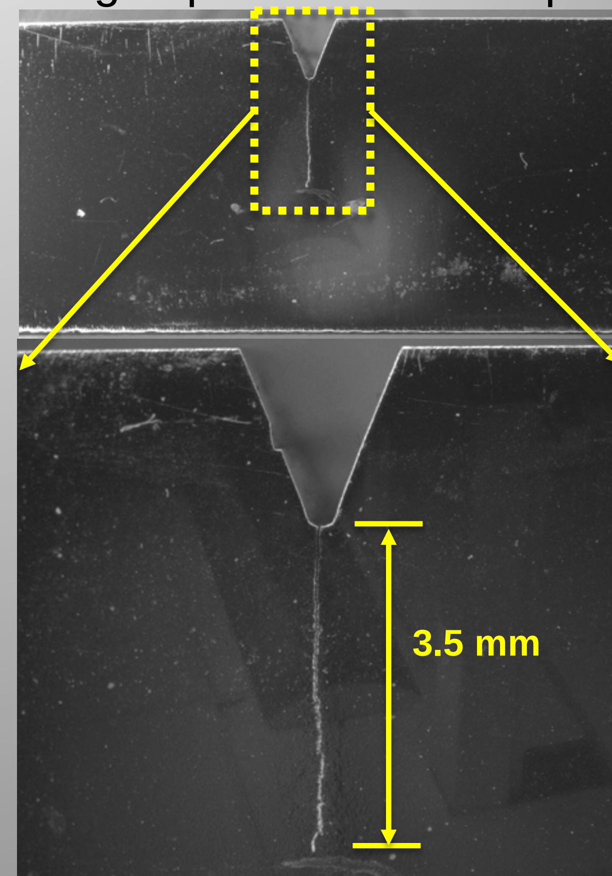
Sample1		Load, lb (start of the test)	Load, lb (end of the test)	K_Q (start)	K_Q (end)	Sample2	Load, lb (start of the test)	Load, lb (end of the test)	K_Q (start)	K_Q (end)
	min	150	14	3.13	0.78		150	48	3.13	1.47
	max	950	490	19.8	27.3		850	740	17.7	22.7

- Fatigue pre-cracking unit

- Fatigue pre-cracked sample #02

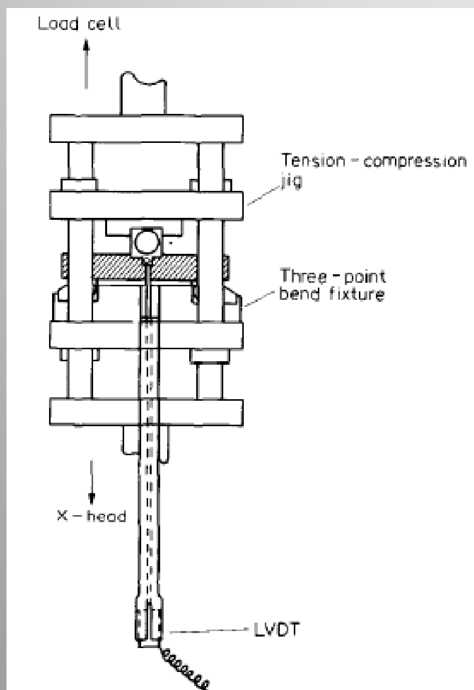


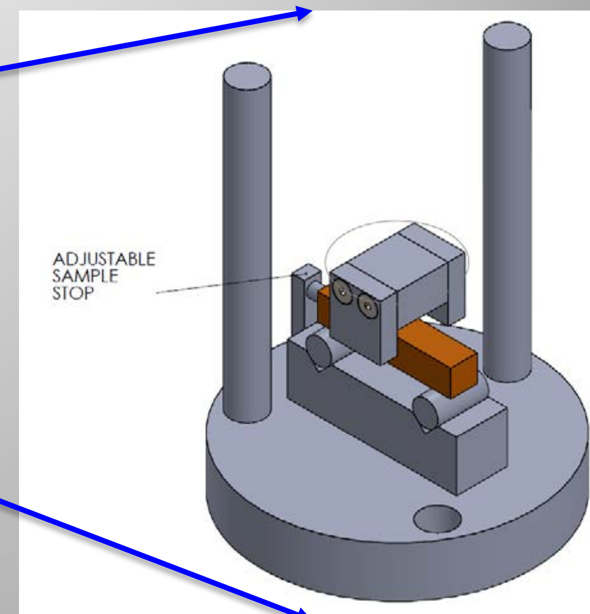
- Fatigue pre-cracked sample #01



Details of design and development of the experimental set-up and determination of J_{ISCC}

- Fixture development for using sub-size Charpy specimens to evaluate fracture toughness (J_{IC}) using unloading compliance technique under 3-point bend loading





- Measurement of crack length using elastic compliance calibration equation

$$\delta = \frac{C\left(\frac{a}{W}\right)}{EB}P$$

$$B_{eff} = B\left\{1 + 0.67\frac{B_n}{B}\left(1 - \frac{B_n}{B}\right)\right\}$$

For side grooved specimen, B is replaced by B_{eff}. B_n is the effective thickness of the side-grooved specimen.

$$J = 2\frac{A_i}{B_{eff}\,b}$$

A_i = the area under the load –line displacement curve up to the i^{th} step
b = the initial remaining ligament

J-integral: experimental evaluation procedure – unloading compliance approach

$$v = \frac{P}{EB} \times C(a/w)$$

(V = δ)

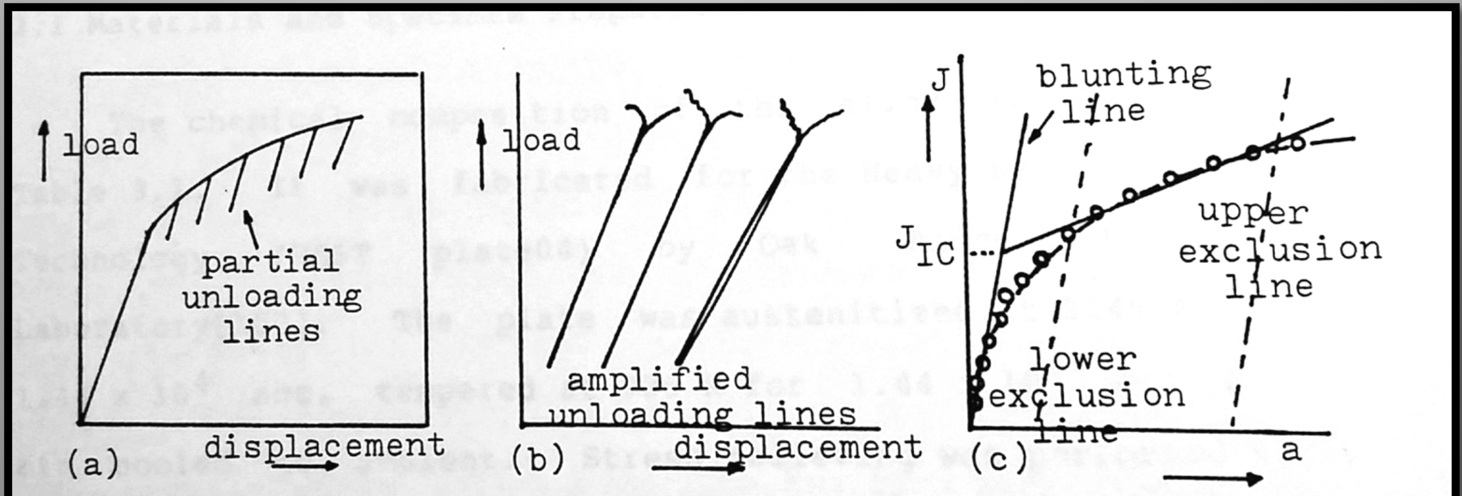
P = load
E = Young's modulus
B = nominal thickness

$$C(a/w) = 0.24(s/w)\left\{1.04 + 3.28(w/s)\left(1+v^2\right)\right\} + 2\left(1-v^2\right).(a/w).(s/w)\{4.21(a/w) - 8.89(a/w)^2 + 36.9(a/w)^3 - 83.6(a/w)^4 + 174.3(a/w)^5 - 284.8(a/w)^6 + 387.6(a/w)^7 - 322.8(a/w)^8 + 149.8(a/w)^9\}$$

- For side-grooved specimens, equivalent thickness is given as follows

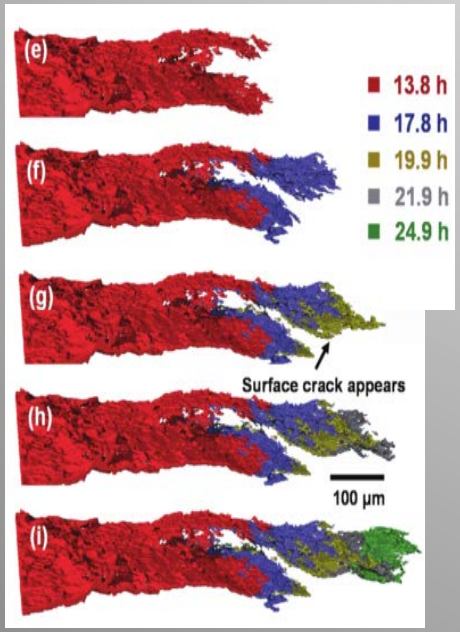
$$B_e = B\left\{1 + 0.67\frac{B_n}{B}\left(1 - \frac{B_n}{B}\right)\right\}$$

B_n = net thickness
s/w = span to width ratio (= 4 for three point bend specimens

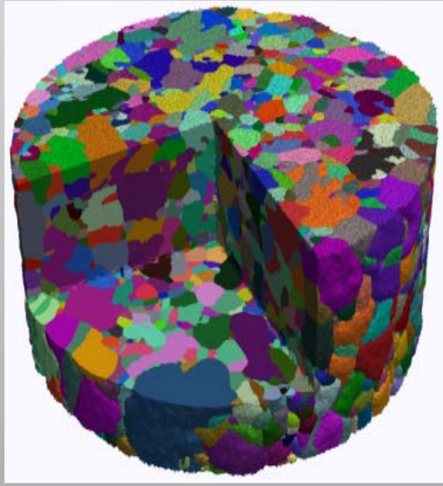


Use of In-situ X-Ray tomography for corrosion studies

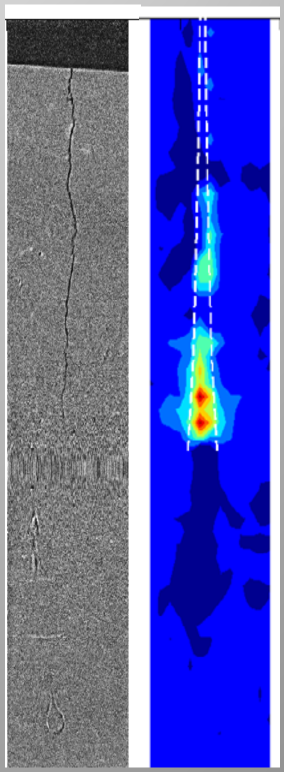
- **XRD:** determination of the phase fraction of martensite
- **Absorption tomography:** imaging the whole crack at different times: determination of crack morphology and accurate measurement of crack propagation
- **Near-field High Energy Diffraction Microscopy:** mapping the polycrystalline structure of the materials (including grain orientations): transgranular or intergranular crack ?
- **Far-field High Energy Diffraction Microscopy:** mapping the stress/strain field within grains: deformation-induced martensite ?



Crack propagation during in-situ stress corrosion cracking of Al 7075 im 95% humidity (*Singh 2015, PhD Thesis, Arizona State University*)



Reconstruction of microstructure and grain orientation of a polycrystalline material (*Picture courtesy of ANL)*



Reconstruction of a crack and corresponding plane strain measurement (*Picture courtesy of ANL)*

Experiments for In-situ X-Ray Tomography

- **Analyses of crack propagation only (no initation: use of pre-cracked specimen)**
- **Experimental conditions (in-situ) chosen:**

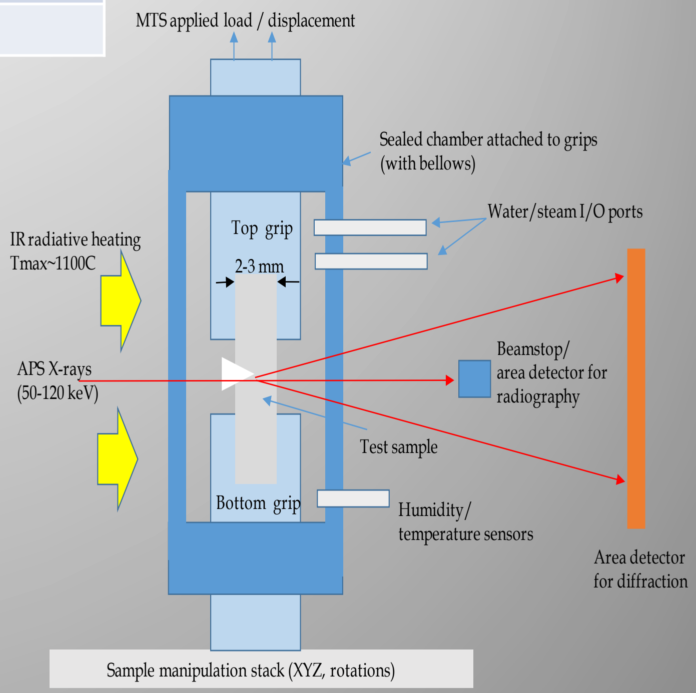
	Temperature	RH (%)	Salt	Stress
Accelerated	80ºC	30-35	$MgCl_2$	YS
Realistic	<55ºC	25	Synthetic sea-water	YS

  - Accelerated conditions: To ensure the observation of crack propagation ($MgCl_2$ more aggressive) (Based on the report "*Summary of relevant crack growth rate*" by Bryan and Enos (results from Cook 2011 and Hayabashi et al 2008))

    Expected growth rate ~2.10^{-9} $m.s^{-1}$
  - Realistic conditions: Assess the crack growth rate for conditions representative of storage conditions. Based on the presentation "*Environment presentation*" by Bryan and Enos (Represents the most aggressive conditions possible at the surface of a canister)

Tests should be started at USC (crack initiation) then processed for Synchrotron experiment; Building a small cell in which a crack can be induced by fatigue on a larger Single Edge Notch Tension (SENT) specimen

- **Ex-situ analyses of interrupted tests from NCSU:**
  - Grain mapping around the crack
  - Plastic strain at crack tip
  - Characterization of corrosion phases



Schematic representation of in-situ tomography device at ANL

Specimen and In-Situ Frame Design

Trip to Argonne in April 2016 with discussions =>

- **Specimen**

  Single edge notched tension specimen (SENT)

  Full length: 35 mm, distance between grips: 17.22 mm

  Stress intensity factor given by *Zhu 2016, Fatigue Fract Engng Mater Struct, 39, 120-131* (correct for 0<*a*/*W*<0.98)

  If 1-ID experiments:

  - Cross-section 1.2 x 1.2 mm (max. dimension 1.8 mm)
  - Pre-crack induced by fatigue cycling on a larger SENT specimen (cross-section 1.2 x 6 mm) with further machining to the desired dimensions

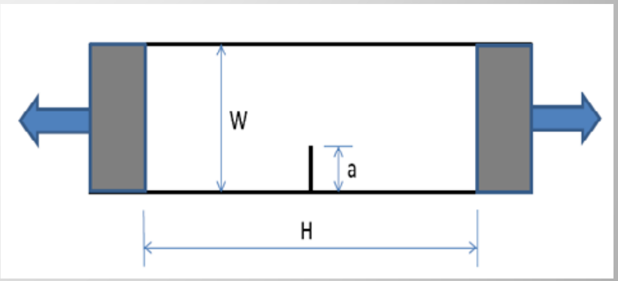
  If 2-BM experiments:

  - Larger sample dimension allowed (max. dimension 3 mm)
  - Cross-section: 2.8 x 0.6 mm (*Singh 2015*)
  - Pre-crack induced by fatigue cycling on the specimen

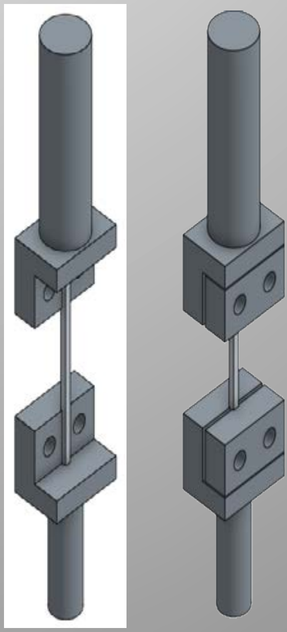
- **Frame:**

  Based on the small loading frame at Argonne National Laboratory

  Heating induced by a heat gun directed toward one end of the specimen



SENT specimen (*Zhu 2016*)



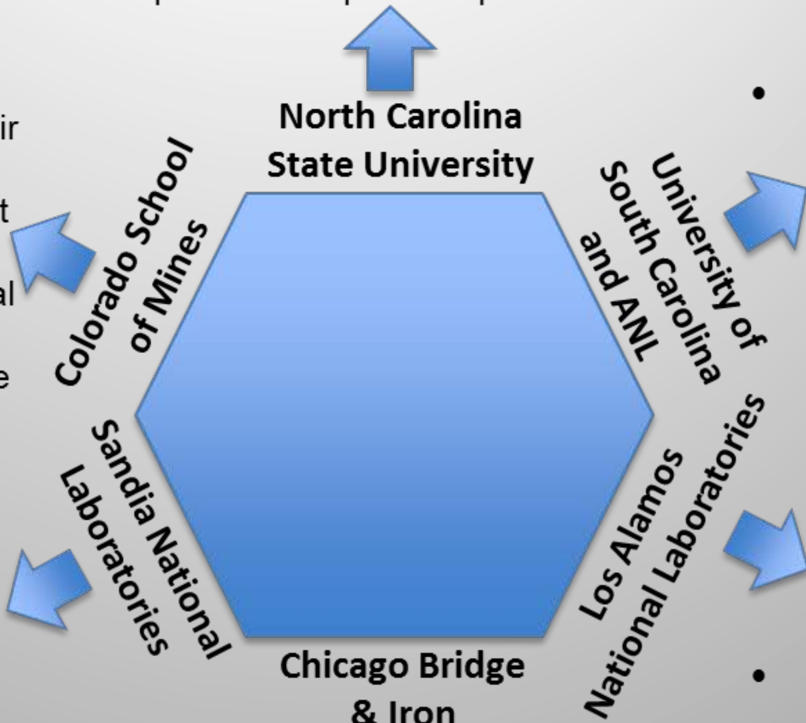
New friction grips for the SENT Specimens

Prototype of corrosion chamber – made of PMMA

- Fracture mechanics based analysis of stress corrosion cracking and determination of K/J_{ISCC}
- Crack growth study using DC potential drop technique

- Full size mock-up simulation of stress distribution by finite element method
- Design of environmental chamber for corrosion
- CFD modeling of flow rate of humid air and salt aerosols
- Downsize mockup design for field test using ABAQUS
- Simulation of downsize mockup in real marine environment
- Determination of the most susceptible HAZ microstructure for SCC
- Incubation time experiment
- Feasibility study of prompt gamma activation method

- Crack growth rate under different environment, using in-situ TEM
- Crack growth rate under different environment using in-situ 3D synchrotron X-ray tomography at ANL



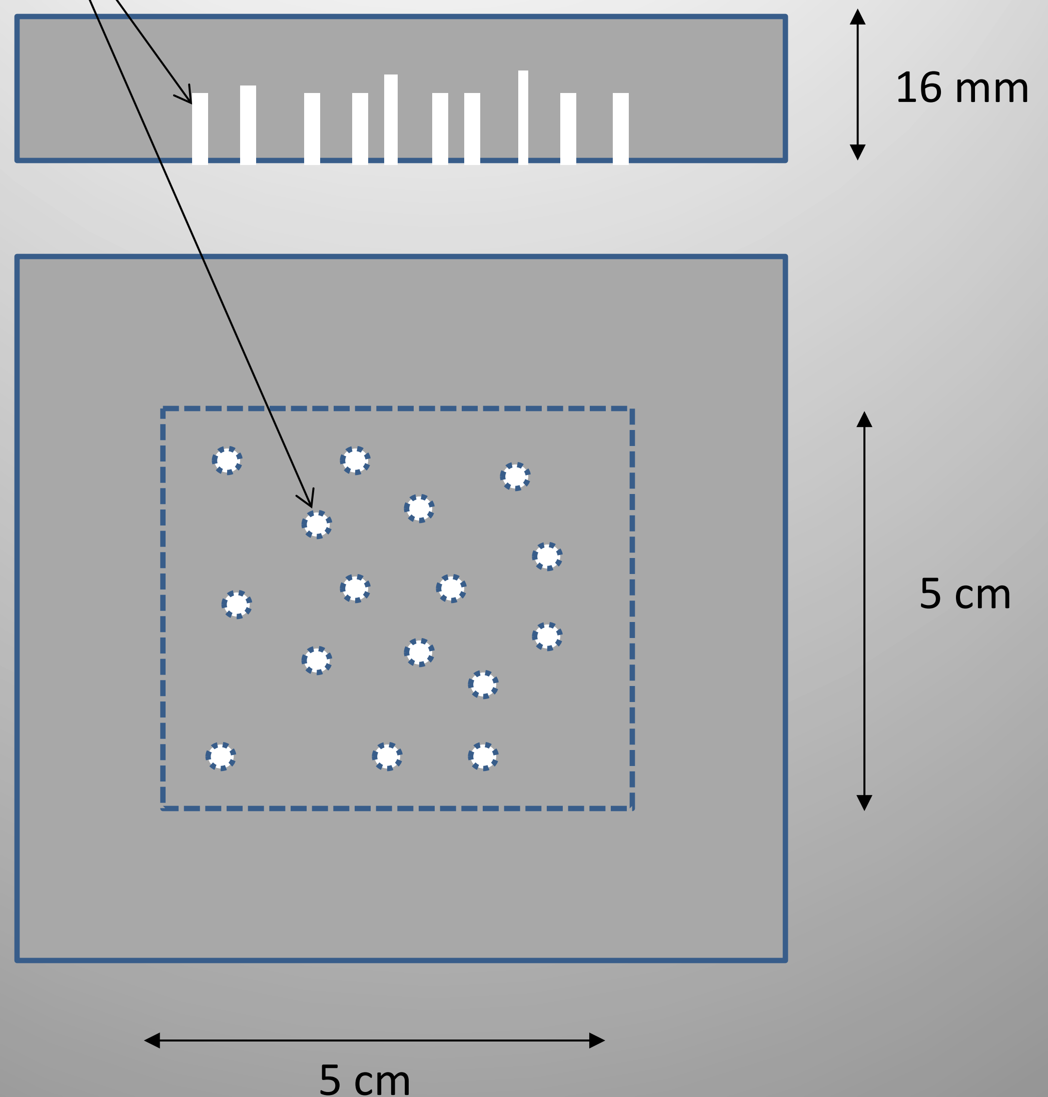
- Help design the downsize mockup with CSM
- Support field testing of the downsize mockup and canister inspection and monitoring system
- Provide expertise and guidance in corrosion simulation

- Evaluating the impact of project results on the ISFSI licensing process, primarily with regards to the ISFSI Aging Management Program
- Interface with utilities that operate ISFSIs, and with spent fuel storage system vendors as necessary, to help make arrangements for testing of NDE instrumentation in the field
- Assistance regarding QA of welded samples for the experiments

- Non-linear ultrasonic NDE at LANL of partially crack grown samples

NDT Detection Limit?

Fifteen to ninety "micron-sized holes" randomly spaced and simulating pits between 100-500 microns in diameter, 0.1-12.5 mm deep, i.e. different aspect ratios.



How can one prepare such micro-sized features? What methods are available?

Typical smallest drill bit (#80) diameter: 0.3429mm