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**CRACK GROWTH RATE TESTING OF BOLT-LOAD COMPACT TENSION SPECIMENS  
UNDER CHLORIDE-INDUCED STRESS CORROSION CRACKING CONDITIONS IN SPENT  
NUCLEAR FUEL CANISTERS**

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**ABSTRACT**

Stress corrosion cracking (SCC) may occur when chloride-bearing salts and/or dust deliquesce on the external surface of the spent nuclear fuel (SNF) canister at weld residual stress regions. An SCC growth rate test is developed using instrumented bolt-load compact tension specimens (ASTM E1681) with experimental apparatus that allows an initially dried salt to deliquesce and infuse naturally to the crack front under temperature and humidity parameters relevant to the canister storage environmental conditions. Characterization of initial shakedown tests was performed to determine a more extensive matrix of testing to provide bounding conditions in which cracking will occur. The test specimen and apparatus designs were modified to enhance the interaction between the deliquescing salt and the crack front for more accurate crack growth rate measurement as a function of stress intensity factor, temperature and relative humidity which is an essential input to the determination of in service inspection frequency of SNF canisters. Testing was conducted over a range of relative humidity controlled by the guidance in ASTM E104 from ambient temperature to 50 °C with salt assemblages of ASTM simulated sea salt. After three months exposure in prototypic dried sea salt, the specimens will be examined for evidence of chloride-induced stress corrosion cracking (CISCC) and observations are reported for a range of relative humidity and temperature conditions. The above testing attempts to provide a technical basis for the boiler pressure vessel (BPV) Section XI code case N-860.

**INTRODUCTION**

Dry cask storage systems are deployed throughout the world for storage of spent nuclear fuels. Dry shielded canister (DSC) designs include welded, austenitic stainless steel multipurpose canisters (MPC) that are placed within concrete overpacks and stored on a concrete pad exposed to the elements. In order to maintain an acceptable thermal profile, overpacks are vented to the environment to allow for cooling of the canister by convective air flow. Experimental studies have established the susceptibility of stainless steels to stress corrosion cracking in environments containing chloride salts [1-4]. Salts are transported in aerosol droplets from sea water and continental sources on air currents or atmospheric convection and entrapped or deposited in dust layers on an MPC, over time [5]. Moist chloride-rich salt deposits are aggressive to stainless steel, particularly at weld heat-affected-zone (HAZ) regions with sensitized microstructures and welding residual stresses (WRS). Because heat treatment for stress relief is not required for the construction of an MPC, the canister is expected to retain WRS from its fabrication and closure welds. These stresses have been directly measured [6] and estimated [7] using recommendations from API-579 [8]. These stresses were used to calculate the stress intensity factor and determine the bounding surface flaw configurations for canisters sections adjacent to weldments in MPC [9]. The data show that the canisters sections adjacent to weldments can retain WRS in excess of the yield strength of the base metal and a stress intensity factor in excess of the canister material's

postulated threshold stress intensity factor ( $K_{ISCC}$ ). Hence, the potential exists for canisters to undergo CISCC in weld or HAZ regions under long-term storage conditions. The scenario that would be required in order for this occur has been identified by Enos *et al.* [5]. Heat from radioactive decay typically provides an MPC with sufficient thermal mass to maintain the surface temperatures above temperatures that allow deliquescence of atmospheric salts to occur (~85 °C), thereby limiting the surface of brine (the substance essential for the onset CISCC). However, thermal gradients at the surface and variances in initial loading of the canisters could enable the potential for surface temperatures to drop below this threshold in the future. If the local relative humidity at the canister surface is high enough, salt deposits may deliquesce and form aggressive brine that may undermine the integrity of canisters [3].

To mitigate the potential of SCC in canisters, the U.S. Nuclear Regulatory Commission has proposed an Aging Management Program (AMP) for licensees to institute to ensure that the structural integrity and confinement function of the canisters is maintained for extended storage conditions [10]. Consensus rules for in-service inspection of the canisters are being developed, and these rules can serve as the foundation for the canister AMP and a Task Group was constituted in 2015 to provide a new code case (N-860) for the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code under Section XI [11]. No evidence of pitting or CISCC on canisters due to corrosion has been observed to date. However, probabilistic approaches that consider the likelihood of through-wall penetration as a result of CISCC in austenitic stainless steel canisters [12] used to identify selection criteria for inspections suggest the potential does exist.

Flaw acceptance criteria for the disposition of the inspection results will be needed if relevant indications are found during the examination. Disposition of flaws that would be identified as stress corrosion cracks, and characterized or sized using visual, surface, and volumetric examination methods. Calculation the stress intensity factors (K) of these flaws under design basis loading cases and bounding residual stress levels will be conducted for identified flaws [9]. A prediction of flaw stability will be determined with allowance for flaw growth between inspection intervals.

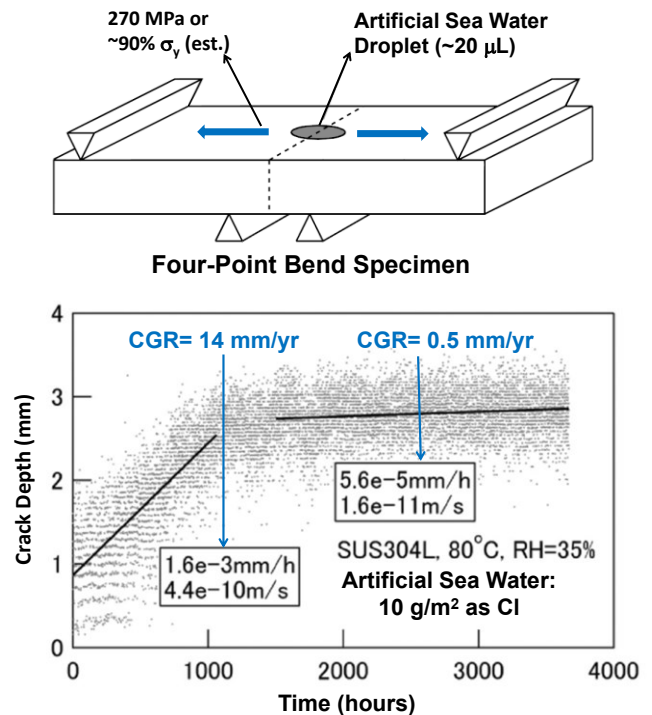
For stainless steels, it is commonly concluded that canisters are highly tolerant of flaws. However, when residual stress is included as part of the loading in the analysis and if consideration is given to elastic-plastic fracture mechanics through the Failure Assessment Diagram (FAD) formulation [8], the instability crack sizes are significantly reduced from those based on the net section yielding limit load failure criterion and more conventional code based analyses [7, 12]. Hence, accurate and reliable crack growth velocities become of increasing importance for reliable flaw disposition.

The challenge with generating data relevant and directly applicable to the DSC is the environmental loading and testing for what is called “dry salt” conditions. That is, with initial fully dried deposits on the canister surface, and under falling

canister temperatures with extended times, the deposits with deliquescing salts will eventually create the aggressive environments but at an uncertain rate and severity.

### CRACK GROWTH RATE TESTING

Crack Growth Rate (CGR) testing aimed at conditions for a DSC in natural exposure settings and with salt assemblages loaded onto the specimens has been performed over the past several years. Researchers at Central Research Institute of Electric Power Industry (CRIEPI) in Japan have generated the largest compendium of data [5, 14-17]. Several specimen designs, included 4-point bend [1, 3], 3-point bend [14, 15], and compact tension (CT) [14, 17] were used. Figures 1 and 2 summarize the crack growth rates measured for the various specimen designs, mechanical loading and environments. The wide range of CGR (0.5-110 mm/year) is evident from these figures. The impact of the range of crack growth rates on the disposition of flaws and proposed inspection frequency would be difficult to implement without further understanding the role of environmental factors on CGR

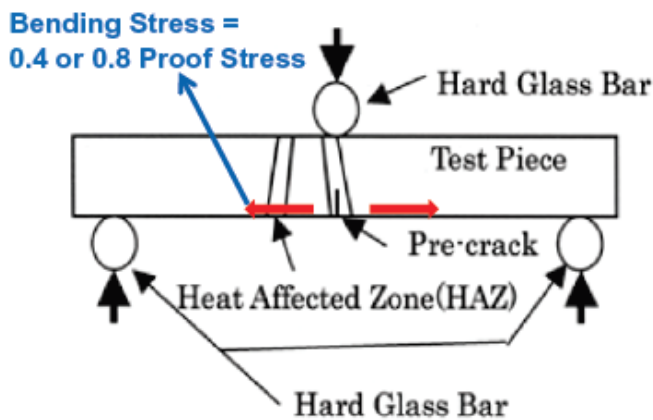


**FIGURE 1: 4-POINT BEND TEST SPECIMEN CONFIGURATION AND RESULTS [4] (specimen does not appear to have been pre-cracked)**

A phenomenological understanding of the role of initiation, environment, stress intensity factor and test temperature on CGR is difficult to formulate from the previous data due to several contributing factors. Crack initiation was not reported in some studies [3], hence, it is unknown whether or not initial flaws were uniformly pre-cracked or if cracks were initiated *in situ*. Crack geometry could complicate estimating stress intensity factors (K) at the crack tip. Fatigue pre-cracking in air

was used in the testing with 3-point bend and Compact Tension (CT) design specimens [14-17]. The aggressive environmental conditions used to load the crack growth test specimens was chloride solutions or artificial seawater. Natural exposure testing was also used in the 3-point bend specimen testing.

CGR typically has a variable dependency to the applied K. A threshold stress intensity,  $K_{ISCC}$ , can be observed, below which SCC does not propagate. At higher K, a region of K dependency exists. Finally, rapid increase in CGR is observed until fast fracture occurs at K levels approaching the materials fracture toughness. The relationship between K and crack growth rate is not well understood from review of the data in Figures 1 and 2.



Test	Test Type	Environmental Conditions	Minimum CGR (mm/yr)	Maximum CGR (mm/yr)
1	Natural Exposure	Ambient Temperatures	0.04	0.55
2	Accelerated Testing	60 °C 95% RH NaCl Steam Mist	3	110

**FIGURE 2: 3-POINT BEND TEST SPECIMEN CONFIGURATION AND RESULTS FOR 304 and 304L SS [15, 16].**

Results vary widely among the available literature on CGR testing under conditions similar to the deliquescent salt conditions on the canister. This appears to be due, in part, to the difficulty in crack nucleation, or propagation in a uniform crack front. This could be related to insufficient electrolyte or loss of chloride from the salt brine composition. Previously, a new “dry salt” SCC specimen test configuration was designed to overcome the stochastic nature of crack nucleation and the irregular crack front during propagation [18-20]. Some cracking was observed in 304 SS base metal, but the cracking conditions tested were shown to be brine limited. Uncertainty was identified as to the role that inert dust played in limited availability of liquid. Cracking was also slow to initiate from

pre-cracks, was non-uniform and deviated from a planar crack front. Propagation was localized to high stress locations suggesting a threshold could be identified under the ideal test conditions.

In order to simplify the test method, further testing was proposed for conditions of higher relative humidity to address the limited brine. Lower temperatures were proposed to maintain the same absolute humidity that is bounding in environmental conditions [5]. Furthermore, since the role of artificial dust in brine availability is uncertain [19], pure salt would be used in the next tests matrix. Previously, specimens were selected from 304 SS plate in base metal condition [19]. To elucidate the role of thermal history on CISC susceptibility, samples were harvested from a mock-up dry storage canister [6]. Results from specimens near heat affected zones (HAZ) adjacent to welds are compared to results from the base metal of the same plate. The testing has been initiated and is discussed in the current study.

## EXPERIMENTAL DRY SALT CRACK GROWTH TESTS

### Specimen

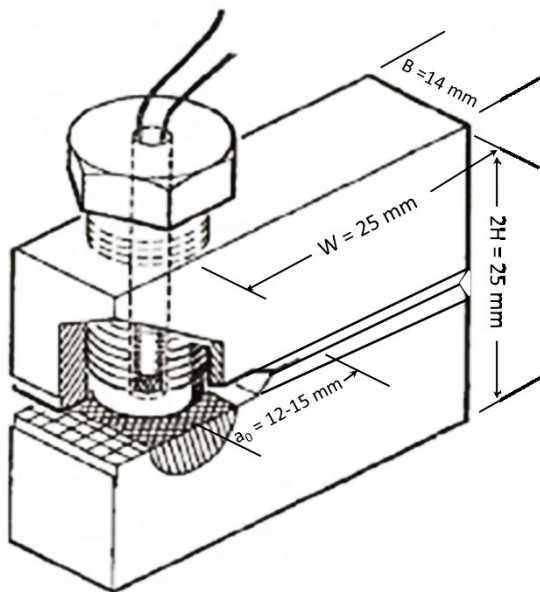
Using a bolt-load compact tension CT specimen (BLCT, Figure 3) as defined in accordance ASTM E1681 [21], samples were cut from plates harvested from a type 304L canister mockup [6]. Details of composition and fabrication strategies are discussed in reference 6. The material was 16 mm thick, dual certified (304, 304L) stainless steel that was cold formed into cylinders and welded using a multi-pass, shielded arc weld (SAW) for joining. The cylinders were joined together with a circumferential weld by the same method. Figure 4 shows the specimens as they were removed from the plates by electric discharge machining (EDM). Individual welds were oriented orthogonally with respect to each other so the specimens were harvested such that they would all be the same orientation. Table 1 shows the matrix of test conditions. Pre-cracks ( $a_0$ ) were grown in fatigue (in air) to lengths of 0.5-0.6 ratio of length over width ( $a/w$  ratio). Crack lengths were examined using an optical microscope on both sides of the specimen surfaces.

The modulus (or compliance) of the cracked specimen was measured on an Instron load frame outfitted with a crack opening displacement (COD) clip gauge prior to bolt loading. The specimens were loaded using instrumented and conventional bolts to bear the load. The type 304L SS test specimen was pre-cracked in fatigue with side grooves to ensure planar crack fronts.

### Environment

During the test, a porous glass frit holder maintains intimate contact between the specimen, the dry salt and environment in order to simulate chloride bearing deposits (see Figures 5 and 6). The dry salt is from dehydrated artificial sea water [22] that is dehydrated at 95 °C and ground with a mortar and pestle. Constituents of the salt are permitted to naturally

deliquesce to provide brine to induce crack growth if the material is susceptible. In order to ensure CISCC favorable conditions, a cellulose wick was inserted at the front of the machined notch in order to ensure adequate brine will be transported evenly along the cross-section to the fatigue precrack front of the specimen. The instrumented bolt and loading configuration afford the real time measurement of load vs time by which crack growth can be established. The falling K configuration allows for the identification of a  $K_{ISCC}$ . As a compliment to the specimen, teardrop specimens are inserted to the same environment to help establish conditions are favorable for crack growth. Initial test conditions were selected to be 50 °C and 50% relative humidity (RH), which bounds storage configuration [5]. Current test conditions were specified to be more prototypic of storage for the DSC and were maintained at RT or 37 °C at 75 ± 5% RH (i.e., 15 and 33 g/m<sup>3</sup> AH, respectively). Saturated brine at the bottom of glass vessel is used to control the RH in accordance with ASTM E104 [23]. Temperature is controlled in a convection oven. Atmospheric conditions are monitored using a Vaisala HUMICAP® Hand-Held Humidity and Temperature Meter HM70 equipped with a high temperature probe tip. A Data Acquisition System (DAS) to acquire load drop with time for a fixed COD. The specimens are held for approximately 3 months at constant conditions.



**FIGURE 3: SCHEMATIC CONFIGURATION SHOWING BLCT WITH AN INSTRUMENTED BOLT**

### Analysis and Characterization

The final load on the specimen will be determined by unloading the specimen and reloading in the Instron while recording load and COD. These values were compared to initial results. Specimens will be cleaned in accordance to ASTM G1 [24]. Final crack lengths will be established independent of load by heat tinting at 350°C for 30 minutes and fatigued until the crack grows to the back face of the specimen.

The crack lengths are measured under an optical microscope in accordance ASTM E1681 at the transition from heat tint and un-oxidized fatigue surface. The fracture surface will be characterized using an Scanning Electron Microscope (SEM) equipped with an Energy Dispersive Spectrometer (EDS) for elemental analysis.

## RESULTS AND DISCUSSION

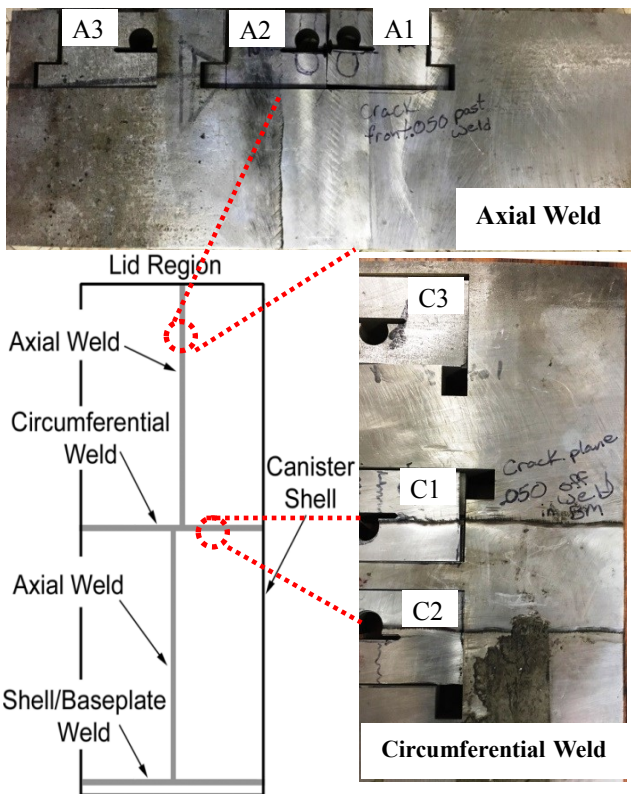
At the exposures in this study (RT and 37 °C at 75% RH), simulated salt mixture does deliquesce to provide adequate electrolyte to induce CISCC. Specimens were loaded in March, 2018. If the material is susceptible crack growth, it will be observed through the instrumented bolt signal. Figure 7 is a typical representation of load vs time for a bolt load specimen over time. Load relaxation is expected during the initial heat up phase but should not the signal over long term tests. Further load drop over a period of time will be assumed to due to crack growth. At the appropriate opportunity during testing the specimens will be pulled and crack growth measured. The results will be fully discussed in a supplemental paper.

Teardrop specimens provide a rapid assessment of the conditions during testing [25]. These teardrop specimens have been utilized in several studies to date in order to provide a real time indicator of environmental conditions. In sea salt tests that used artificial sea salt/dust mixtures [19], the corrosion or cracking was not present after 5 months indicating that an environment conducive to crack growth may not exist. Its follow on testing at 50 °C/50%RH did establish the aggressive nature of the environment; the teardrop specimens did crack after an additional 3 months at these conditions. Figures 8-10 show results from these tests and illustrate the presence of CISCC in samples. Specimens are shown in Figure 8 a-c for 100% salt, 25% salt/75% CeO<sub>2</sub> and 25% salt/75% artificial dust conditions, respectively. Figure 9 a and b shows a cross section the same teardrop conditions as Figure 8a. Cracking in several locations of this specimen is clearly evident and appears to originate in multiple locations and grow through wall quickly. Previous work by Metzger *et al.* [20] showed that the crack depth in teardrop specimens is not necessarily related to pit depth (see Figure 10).

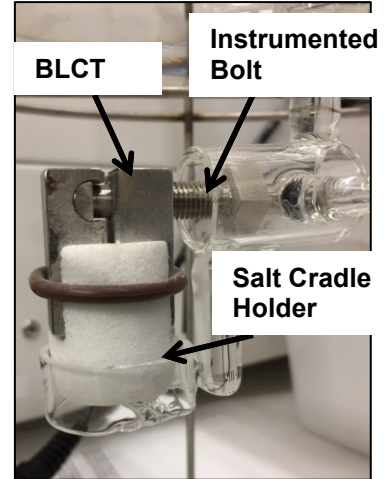
Although these cracks are a robust indicator of environmental conditions, their usefulness as a predictor of CGR is extremely limited for the following reasons. First, the flaws and stresses in teardrop specimens are not uniform so determination of stress intensity factors is difficult. Secondly, there is inadequate control of where the crack nucleate. Hence, bolt load test specimens offer a favorable method of CGR determination.

## SUMMARY AND PATH FORWARD

Corrosion and stress corrosion cracking resistance was examined for base metal and HAZs of stainless steels in dry salt environments. At the exposures in this study (room temperature to 37 °C at ~75% RH), simulated sea salt residues deliquesce to provide adequate electrolyte which leads to pitting corrosion and cracking in 304/304L stainless steel. The nature of the cracking is still under investigation and CGR are being measured. The effects of temperature, thermal history and relative humidity are being varied to determine the rate limiting factors. Approximately 3 months of exposure at these conditions is expected to initiate both pitting and cracking in BLCT and teardrop specimens. The ability to accurately measure the  $K_{ISCC}$  will depend on the planar nature of the crack and uniformity of the crack front.



**FIGURE 4: SPECIMENS AS THEY CUT FROM MOCKUP CANISTER SHOWING ORIENTATION WITH RESPECT TO WELDS** (Samples 1 & 2 cut from HAZ, Sample 3 cut from Base Metal of plate)

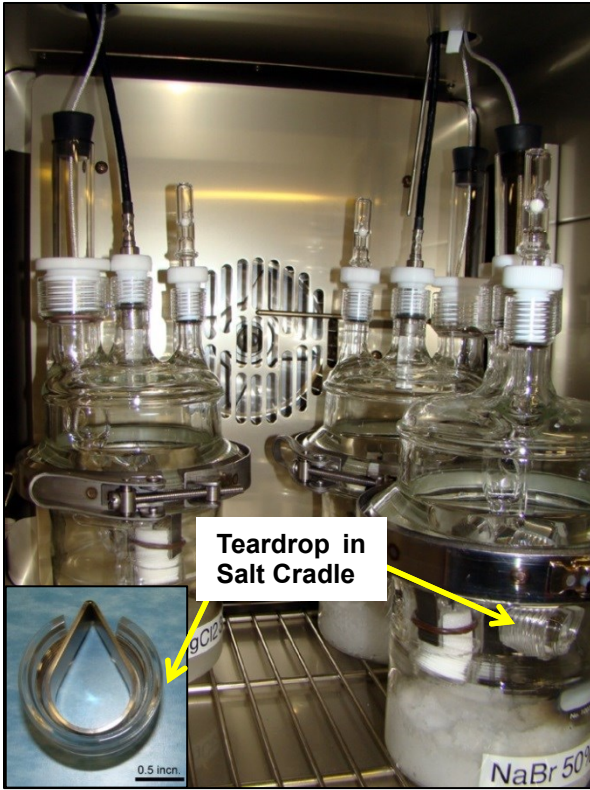


**FIGURE 5: ASTM E1681 BOLT-LOAD COMPACT TENSION SPECIMEN DESIGN, 20% SIDE-GROOVED, IN A SALT CRADLE & AN INSTRUMENTED BOLT**

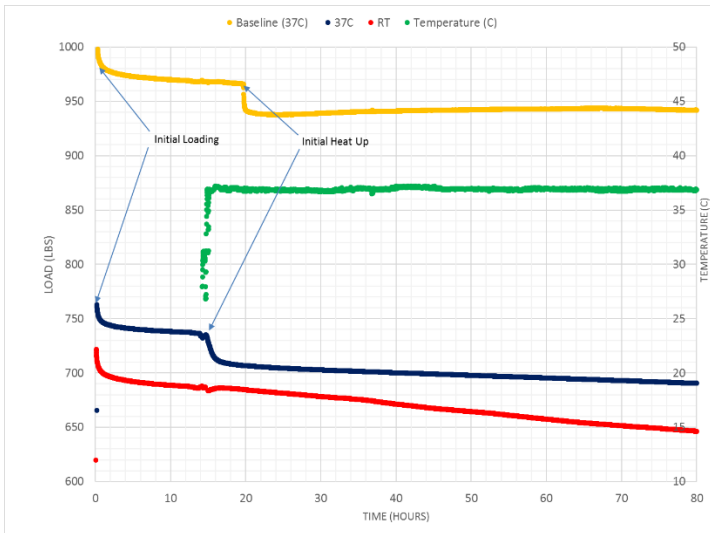
**TABLE 1: TEST MATRIX OF CONDITIONS**

Sample	Temperature	RH	Thermal History	Orientation
Axial 1	20 °C	75%	HAZ	TL
Circ. 1			HAZ	
Axial 3			BM	
Axial 2	37 °C	75%	HAZ	TL
Circ. 2			HAZ	
Circ. 3			BM	

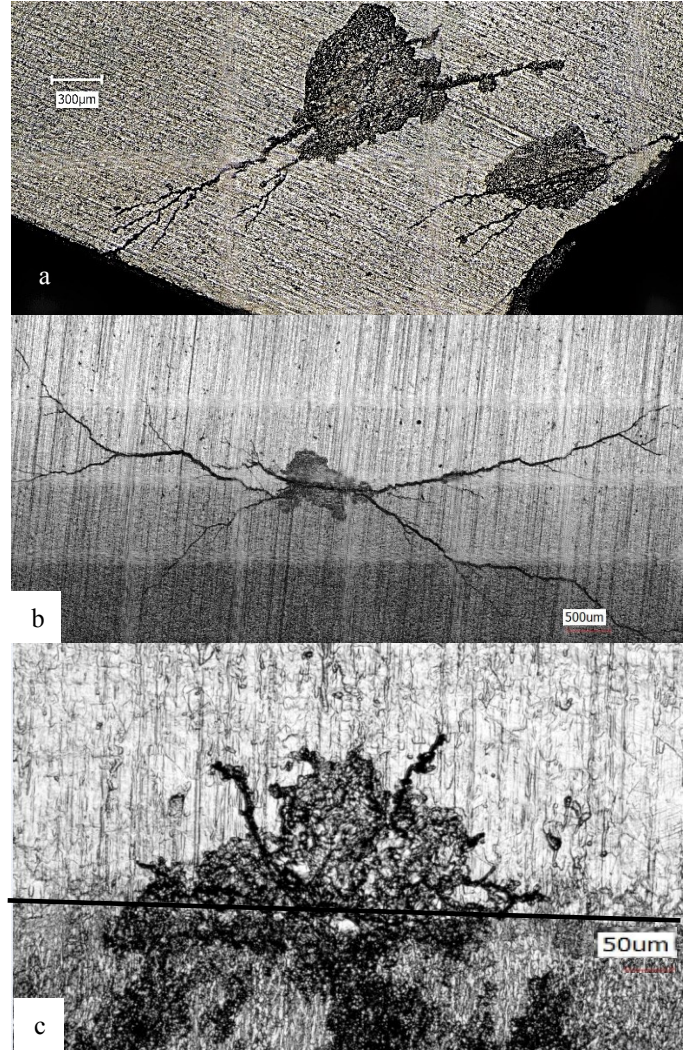
BM: Base Metal



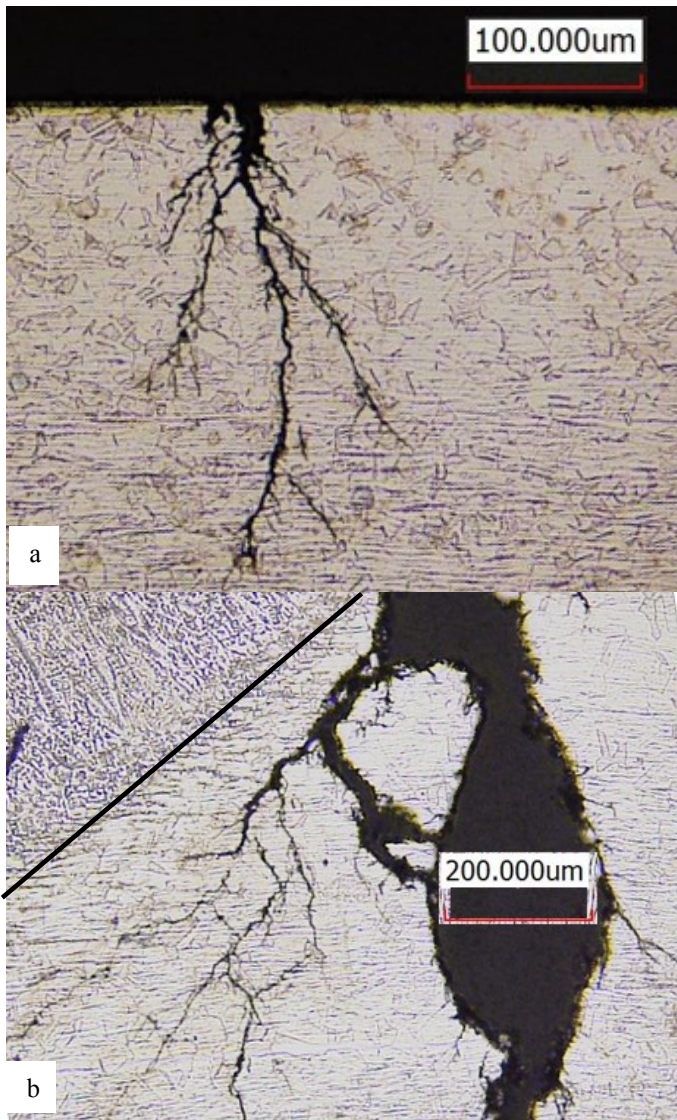
**FIGURE 6: SCC TEST CONFIGURATION SHOWING THE CRADLED BLCT WITH AN INSTRUMENTED BOLT AND A COMPANION TEARDROP COUPON IN A CONSTANT HUMIDITY VESSEL**



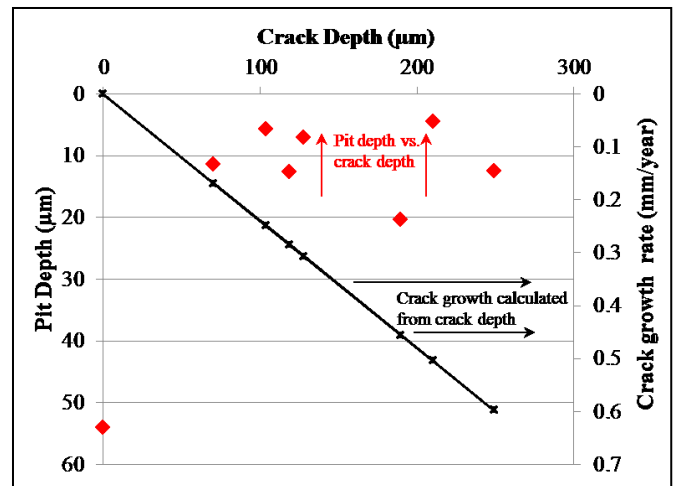
**FIGURE 7: TYPICAL LOAD VS. TIME OF INSTRUMENTED TEST BOLT FOR BOLT LOAD SPECIMEN OVER TIME.**



**FIGURE 8: LASER CONFOCAL MICROSCOPE (LCM) IMAGES OF CRACKING INITIATED AT SURFACE OF TEARDROP SPECIMENS AT 50 °C AND 50% RH [20] FOR 100% SEA SALT (a), 25% SEA SALT + CeO<sub>2</sub> (b) AND AT AUTOGENOUS WELD INTERFACE ON SURFACE (SHOWN BY LINE) IN 25% SEA SALT + ARTIFICIAL DUST (c).**



**FIGURE 9: OPTICAL IMAGE OF PITTING AND CRACKING INITIATED AT SURFACE OF TEARDROP SPECIMEN (a) AND AT AUTOGENOUS WELD INTERFACE (SHOWN BY LINE) ON SURFACE OF TEARDROP SPECIMEN (b) AT 50 C AND 50% RH. (100 % SEA SALT)**



**FIGURE 10: PIT DEPTH VS. DEPTH OF ASSOCIATED CRACK [20].**

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