

US NRC-Sponsored Research on Stress Corrosion Cracking Susceptibility of Dry Storage Canister Materials in Marine Environments – 13344

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ABSTRACT

At a number of locations in the U.S., spent nuclear fuel (SNF) is maintained at independent spent fuel storage installations (ISFSIs). These ISFSIs, which include operating and decommissioned reactor sites, Department of Energy facilities in Idaho, and others, are licensed by the U.S. Nuclear Regulatory Commission (NRC) under Title 10 of the Code of Federal Regulations, Part 72. The SNF is stored in dry cask storage systems, which most commonly consist of a welded austenitic stainless steel canister within a larger concrete vault or overpack vented to the external atmosphere to allow airflow for cooling. Some ISFSIs are located in marine environments where there may be high concentrations of airborne chloride salts. If salts were to deposit on the canisters via the external vents, a chloride-rich brine could form by deliquescence. Austenitic stainless steels are susceptible to chloride-induced stress corrosion cracking (SCC), particularly in the presence of residual tensile stresses from welding or other fabrication processes. SCC could allow helium to leak out of a canister if the wall is breached or otherwise compromise its structural integrity. There is currently limited understanding of the conditions that will affect the SCC susceptibility of austenitic stainless steel exposed to marine salts. NRC previously conducted a scoping study of this phenomenon, reported in NUREG/CR-7030 in 2010. Given apparent conservatism and limitations in this study, NRC has sponsored a follow-on research program to more systematically investigate various factors that may affect SCC including temperature, humidity, salt concentration, and stress level. The activities within this research program include: (1) measurement of relative humidity (RH) for deliquescence of sea salt, (2) SCC testing within the range of natural absolute humidity, (3) SCC testing at elevated temperatures, (4) SCC testing at high humidity conditions, and (5) SCC testing with various applied stresses. Results to date indicate that the deliquescence RH for sea salt is close to that of MgCl₂ pure salt. SCC is observed between 35 and 80°C when the ambient (RH) is close to or higher than this level, even for a low surface salt concentration.

The views expressed herein are those of the authors and not necessarily those of the U.S. Nuclear Regulatory Commission.

INTRODUCTION

Beginning in the 1980s, a number of operating and decommissioned reactor sites in the U.S. have placed spent nuclear fuel (SNF) into dry cask storage. A dry storage cask (DSC) typically involves fuel placed in a welded stainless steel canister within a concrete structure for radiation shielding. DSC configurations include both vertical and horizontal canisters. The U.S. Nuclear Regulatory Commission (NRC) licenses dry storage of SNF under Title 10 of the Code of Federal Regulations (CFR), Part 72, “Licensing Requirements for the Independent Storage of Spent Nuclear Fuel, High-Level Radioactive Waste, and Reactor-Related Greater than Class C Waste.” Among the provisions of 10 CFR, Part 72 are maintenance of confinement systems, protection of the fuel cladding, and the ability to retrieve SNF. Dry cask storage designs are approved for initial licensing terms up to 40 years, after which the licensee may be renewed up to an additional 40 years.

DSC are designed with external vents in the concrete structure to allow airflow around the canisters for cooling. The canister may therefore be exposed to the ambient atmospheric conditions of the site where the ISFSI is located. In the U.S. some ISFSIs are in close proximity to the ocean or other saltwater bodies where atmospheric monitoring indicates a high concentration of airborne chloride salts [1-2]. This raises the possibility that salts could deposit on the canister via the vents in the vault or overpack. Most canisters in service in the U.S. are fabricated from austenitic stainless steels including AISI types 304, 316, and the corresponding L grades. Past experience shows that austenitic stainless steels are susceptible to stress corrosion cracking (SCC) when exposed to chlorides in marine atmospheres [3-4]. Chloride-induced SCC has been reported for stainless steel outdoor piping and tanks at a number of nuclear power plants [5-6]. The moisture required to support the electrochemical reactions associated with SCC could arise from deliquescence, wherein dry salt draws moisture from the air to form a saturated solution. This could occur if the ambient relative humidity (RH) is above the deliquescence relative humidity (DRH) for the salt. Sustained tensile stresses are also needed for SCC. Dry storage canisters are not significantly pressurized, so the main source of stress is likely to arise from fabrication and welding residual stresses. Although the welding residual stresses in canisters are not well characterized, in other systems, welding residual stresses have been shown to be sufficient to cause SCC [7].

SCC of a dry storage canister may affect the safety functions prescribed in 10 CFR, Part 72 by allowing leakage if the wall is breached or otherwise affecting its structural integrity. To investigate potential for chloride-induced SCC of stainless steel, NRC conducted an initial scoping study which was published in 2010 as NUREG/CR-7030, “Atmospheric Stress Corrosion Cracking Susceptibility of Welded and Unwelded 304, 304L, and 316L Austenitic Stainless Steels Commonly Used for Dry Cask Storage Containers Exposed to Marine Environments” [8]. For testing performed using U-bend specimens exposed at 43, 85, and 120°C, SCC was only observed at 43°C. It was postulated that for this one test condition, the RH was sufficiently high for deliquescence to occur. There were, however, a number of limitations and conservatism associated with this scoping study and there is still uncertainty concerning the factors that will affect the SCC susceptibility. Among these are: (1) the temperature and humidity where deliquescence could occur; (2) the quantity or concentration of salt on the surface that will cause SCC; and (3) the magnitude of tensile stress required for SCC.

To address some of these uncertainties, NRC has sponsored a follow-on test program to more systematically evaluate the conditions where austenitic stainless steel may undergo chloride-induced SCC. There are five primary activities within this program. These activities are:

- Determination of DRH of sea salt at various temperatures,
- SCC testing within the natural range of absolute humidity (AH),
- SCC testing at higher temperatures,
- SCC testing in high humidity conditions, and
- SCC testing at various stress levels.

The following section will describe the motivation and experimental approach for the respective activities, as well as provide a brief report of the results obtained to date.

DESCRIPTION OF METHODS AND RESULTS

Determination of DRH of Sea Salt

It is understood that moisture to support electrochemical reactions would be required for chloride-induced SCC of a dry storage canister. Absent the direct introduction of liquid water, moisture would most likely come from deliquescence of solid salt. Deliquescence could occur if the ambient RH is above the DRH for the salt. The DRH will depend on the chemical composition of the salt as well as the temperature. Sea salt is a mixture of pure salts, the most abundant being NaCl, MgCl₂, CaCl₂, and Na₂SO₄. The objective of this activity is to help determine the DRH for simulated sea salt, thereby providing a reference point for SCC testing.

The simulated sea salt for this and other experiments was prepared according to ASTM D1141-98, “Standard Practice for the Preparation of Substitute Ocean Water” [9]. The DRH for sea salt was measured by two methodologies. In the first, simulated sea salt, along with the pure salts NaCl, MgCl₂, CaCl₂, and Na₂SO₄, were placed into glass beakers. The beakers were then put into an environmental chamber in which the temperature and humidity could be controlled. At temperatures of 45, 60, and 80°C, the RH in the chamber was gradually increased and held at different levels while the beakers were observed for evidence of water absorption and eventually the formation of a saturated solution.

The second method for measuring DRH was developed by Yang et al [10] and used a conductivity cell in which sea salt was deposited onto filter paper between two platinum electrodes. In the environmental chamber at 45, 60, and 80°C, the conductivity cell was exposed to gradually increasing RH while measuring impedance of the cell. High impedance would be measured if the salt was dry and there was low conductivity across the cell. Above the DRH, the absorption of water to form a conductive solution on the filter paper would cause a measured impedance drop.

The observations from the glass beaker testing are summarized in Table I, where the approximate DRH corresponding to each salt and temperature is given. CaCl₂ had the lowest DRH of the sea salt constituents, followed by MgCl₂. The DRH for sea salt appeared to be slightly above or close to that of MgCl₂. NaCl and Na₂SO₄ did not deliquesce within the humidity range for this test. Figure 1(a) shows a

picture of the beakers during one of the tests. As measured by the impedance method, the DRH for sea salt was approximately 43% at 45°C, 35% at 60°C, and 25% at 80°C. Figure 1(b) shows an example of the plot at 60°C. These are generally consistent with the results from the beaker tests. The notable observation from these tests is that the sea salt had much lower DRH than NaCl and is closer to that of MgCl₂, though the former is present in sea salt in greater quantity than the latter. The DRH for sea salt, MgCl₂ and CaCl₂ appears to decrease slightly with increasing temperature. The measured DRH is significantly lower than the RH for the test condition at which SCC was observed in the previous NRC testing reported in NUREG/CR-7030 and higher than those conditions where SCC did not occur.

Table I. Observed DRH for salts in beakers

		Temperature (°C)		
		45	60	80
Species	Sea salt	43%	35%	25%
	CaCl ₂	28%	20%	20%
	MgCl ₂	37%	35%	25%
	NaCl	None up to 59%	None up to 60%	None up to 59%
	Na ₂ SO ₄	None up to 59%	None up to 60%	None up to 59%

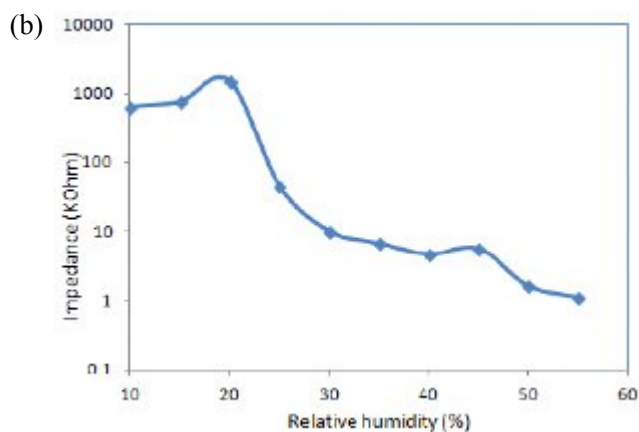


Fig. 1. (a) Photograph of salts in beakers at 60°C and 30%RH. Sea salt, MgCl₂, and CaCl₂ have deliquesced whereas NaCl and Na₂SO₄ remain as dry salts. (b) Plot of measured impedance versus RH for sea salt at 60°C following the methodology of Yang et al [10].

SCC Testing Within the Natural Range of AH

In previous testing by NRC reported in NUREG/CR-7030, it was observed that crack initiation only occurred for a test conducted at 43°C for which the AH was approximately 60 g/m³. Analysis of environmental data, however, indicates that the ambient AH in nature does not generally exceed 30 g/m³ at 30°C [11]. The actual humidity near dry storage canisters is not known, and may be affected by such factors as the local environmental conditions and air flow around the canister. For the purpose of this investigation, however, AH below 30 g/m³ may provide a more reasonable approximation of natural conditions compared to those of NUREG/CR-7030. Thus, the objective of this activity is to determine whether SCC could initiate by deliquescence of deposited salt in this condition.

This activity also evaluated the effect of surface salt concentration on SCC susceptibility. Limited attempts have been made to measure the salt concentration on dry storage canisters [12], but in general the quantity may depend upon the site characteristics, DCSS design, and time in service. In the previous NRC testing, salt deposition on specimens was not well-controlled and a relatively large amount of salt was deposited, about 17 g/m². Reports by Japanese researchers, however, have shown crack initiation for austenitic stainless steel at surface concentration less than 1 g/m² [13]. Therefore, various salt surface concentrations were tested in this activity to help determine if a minimum quantity of salt is needed for SCC initiation.

SCC testing was conducted by depositing simulated sea salt onto AISI type 304 austenitic stainless steel U-bend specimens fabricated according to the specifications of ASTM Standard G30, “Standard Practice for Making and Using U-Bend Stress Corrosion Test Specimens [14], then exposing the specimens to controlled temperature and humidity conditions in an environmental test chamber. Tests were performed at temperatures of 27, 35, 45, 52, and 60°C using cyclic humidity to simulate the diurnal humidity variation generally observed in nature (high RH at night when it is cooler and low RH during warmer daylight hours). The testing protocol consisted of 4 cycles per day between a minimum and maximum AH of approximately 12 and 26 g/m³, respectively. The test conditions can be graphically represented as in Figure 2, showing a plot of RH versus temperature for AH of 15 and 30 g/m³, which are close to the lower and upper AH for these tests. The arrows indicate the span of the humidity cycle for the different test temperatures. Specimens were removed from the test chamber after various periods of exposure for visual and microscopic examination to determine whether cracking had initiated.

Prior to testing, salt was deposited on the U-bends by exposing them to an aerosol salt fog in an environmental chamber. The concentration of salt was determined by measuring the weight gain of a control specimen. Specimens were deposited with 0.1, 1, or 10 g/m² of salt. Deposition of 10 g/m² represents a relatively large quantity, on the same order of magnitude as the previous NRC tests. Deposition of 1 g/m² represents a quantity close to what the Japanese have reported for SCC initiation. Deposition of 0.1 g/m² represents the minimum quantity that could be reliably deposited with the technique used.

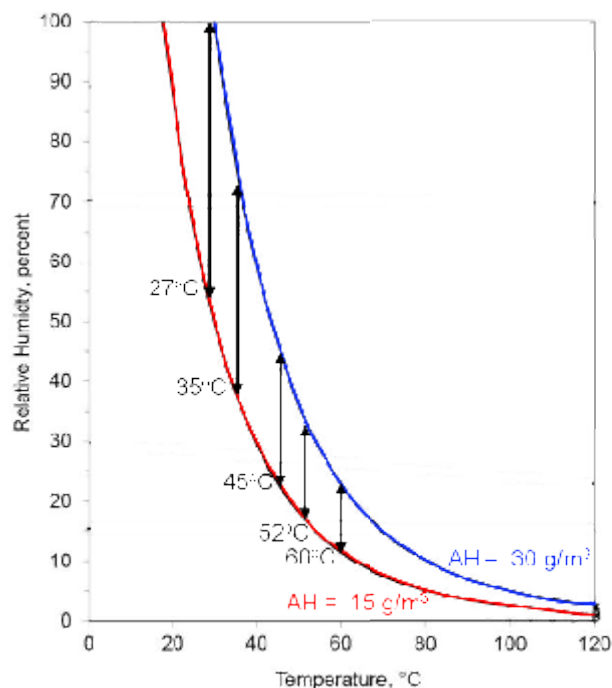


Fig. 2. Plot showing the test conditions for the SCC within the natural range of AH, where arrows indicate the span of the humidity cycle at the respective temperatures (after Fig. 43 in [9]).

For this activity, a number of tests have been completed while others are still in progress. Some of the results were previously reported [15]. The most notable observations to date are as follows:

- At 27°C, salt appeared to go into solution, which then quickly drained off the side of the specimens leaving the surface apparently clean with no indication of SCC.
- At 35 and 45°C, SCC initiated at surface salt concentration as low as 0.1 g/m² within four months of exposure. Sensitized specimens showed higher SCC susceptibility than the as-received or welded. Some specimens with low surface salt concentration are still in testing.
- At 52°C, SCC initiated at surface salt concentration of 10 g/m² within one month of exposure. Specimens with 1 g/m² are still in testing.
- At 60°C, specimens with 10 g/m² are still in testing and have not yet been examined.

An example of the appearance of test specimens after a period of exposure is shown in Figure 3(a), where extensive pitting is visible on the specimens with 10 and 1 g/m² of salt, though the specimen with 0.1 g/m² of salt looks nearly pristine without magnification. Figure 3(b) shows a cross sectional micrograph of cracking in a sensitized specimen tested at 45°C with 0.1 g/m² of salt. The tests at 27, 35, and 45°C likely represent conditions where the RH is above the DRH for sea salt, whereas at 52 and 60°C the RH is likely near or below the DRH for sea salt. These results indicate that SCC initiation could occur within the range of natural AH (i.e., less than 30 g/m³) even with a low surface salt concentration, provided that RH is high enough for some deliquescence to occur.

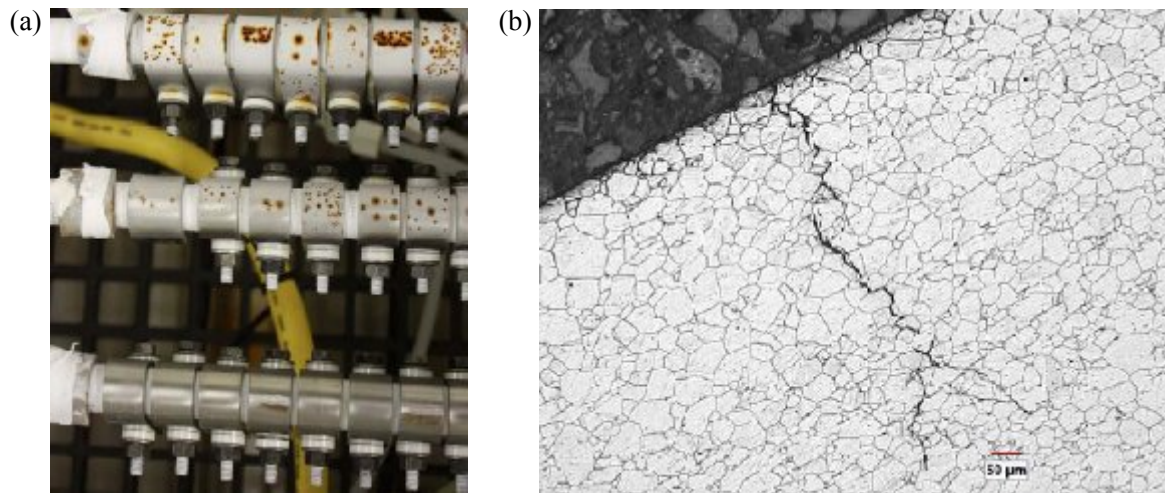


Fig.3. (a) Photograph of U-bend specimens exposed to cyclic humidity conditions. Specimens in top, middle, and bottom row have salt concentrations of 10, 1, and 0.1 g/m², respectively. (b) Cross-section scanning electron micrograph showing initiated crack for sensitized specimen exposed at 45°C for 4 months with 0.1 g/m² of salt.

SCC Testing at Higher Temperatures

As shown in Figure 2, for a given AH, RH tends to decrease with increasing temperature. This suggests that deliquescence, and in turn SCC, is less likely at higher temperatures when AH is limited, as in natural conditions. The cyclic SCC testing described in the previous activity considered relatively low temperatures where, even given the limitations of AH for the test protocol, the RH could be high. Japanese researchers, however, have reported SCC at temperatures up to 80°C where the RH was as low as 15% [16]. This is lower than the DRH for sea salt measured in this test program. Thus, the objective of this activity was to perform testing at higher temperatures than considered in the cyclic tests to evaluate SCC initiation in these conditions.

For this activity, tests were performed at 60 and 80°C using U-bend specimens fabricated in the same manner as for the previously described experiment, though only using specimens with 10 g/m² of salt. The specimens were exposed in environment test chambers using controlled static humidity rather than cyclic humidity because of limited function of the test chamber. The 60°C specimens from this activity may be compared to the 60°C cyclic specimens to determine if there is any difference in the response. At each temperature, specimens were first exposed to RH of 40% at the respective temperatures, which was expected to be high enough for SCC to occur. Specimens were periodically removed from the test chamber for visual and microscopic examination to determine whether cracking had initiated. If cracking had occurred, new specimens were prepared and subsequent tests were performed at progressively lower humidity levels. Following this protocol, tests at 60°C were performed at the RH of 40, 35, 30, 25, and 22%. Tests at 80°C were performed at RH of 40, 35, and 28%.

Some results from this activity were previously published [15]. For the tests at 60 and 80°C, SCC was observed at RH as low as 25 and 28% RH, respectively. These are near the measured DRH for sea salt at these temperatures. Figure 4 shows a photograph of the U-bend specimens exposed at 80°C and 28% RH for 8 weeks. Because of time limitations, the RH for testing at 80°C could not progress as low as 15% to directly compare with the Japanese result. The findings indicate, however, that if RH is sufficiently high for some deliquescence to occur, SCC can initiate at temperature above 43°C, which was reported in NUREG/CR-7030.

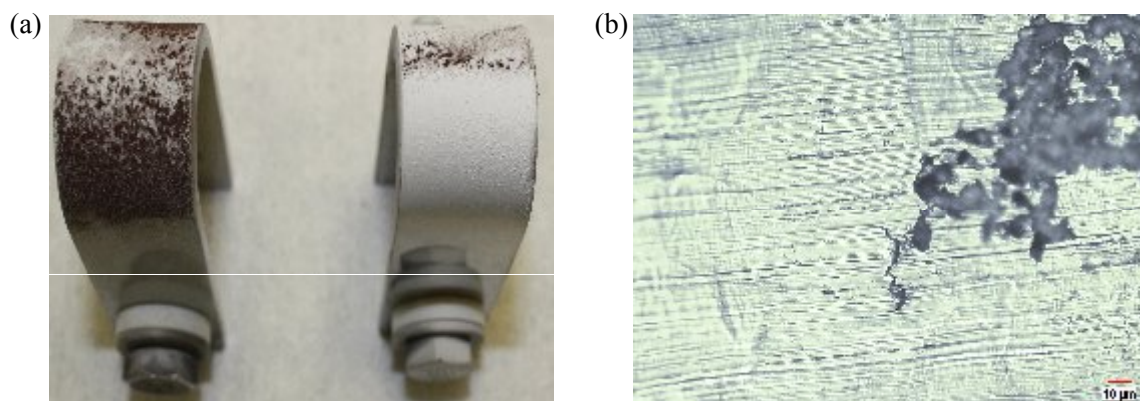


Fig. 4. (a) Photograph of U-bend specimens exposed to 80°C and 28% RH for 8 weeks. (b) Scanning electron micrograph of initiated cracks of one of the specimens shown in (a).

SCC Testing in High Humidity Conditions

The equilibrium chloride concentration in a saturated solution tends to decrease with increasing ambient RH. Figure 6 shows the plot of equilibrium chloride concentration as a function of RH at 30°C for sea salt, as calculated by OLI Analyzer Studio. Other investigators have reported this dilution phenomenon [17,18]. A conceptual model for chloride-induced SCC may consider that SCC will occur as long as the RH is above a threshold that corresponds to deliquescence of sea salt, all the way to 100%. An alternative conceptual model may posit that there is a window of susceptibility because SCC will not initiate above a certain RH because of dilution. To further investigate these potential scenarios, this activity involved SCC testing in conditions of high RH where the equilibrium chloride concentration in solution would be relatively low compared to the previously described tests.

Tests were performed by exposing U-bend specimens at 30°C and 90% RH. Some U-bend specimens were deposited with 10 g/m² of simulated sea salt in the manner described for the previous activities. Other U-bend specimens were immersed in prepared solutions of sea salt, NaCl, MgCl₂, and CaCl₂, respectively, at calculated equilibrium concentrations at 30°C and 90% RH. Table II shows the salt and chloride concentration in the prepared solution. Specimens were periodically removed from the test chamber for visual and microscopic examination to determine whether cracking had initiated.

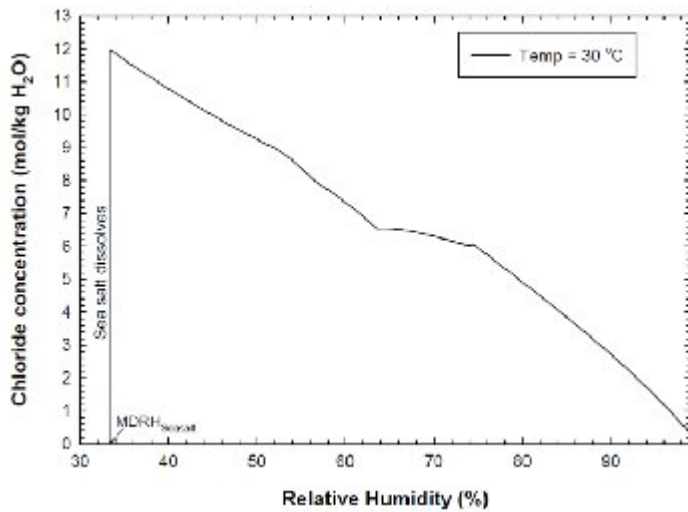


Fig. 5. Plot of equilibrium chloride concentration as a function of RH for sea salt at 30°C, as calculated by OLI Analyzer Studio.

TABLE II. Chloride and salt concentrations in equilibrium solutions at 30°C and 90% RH.

		Chloride Concentration (mol/kg H ₂ O)	Salt Concentration (g/kg H ₂ O)
Solution	Sea salt	2.71	203
	NaCl	2.79	163
	MgCl ₂	3.01	306
	CaCl ₂	3.16	232

For the U-bend specimens deposited with sea salt, the salt went into solution and quickly drained off the side, leaving the surface clean before SCC could occur, similar to the cyclic SCC tests at 27°C. For specimens immersed in solution, minor pitting and what appear to be small cracks were observed after 5 weeks of exposure. Tests are ongoing to verify the crack initiation. Figure 6(a) shows a photograph of the immersed specimens and 6(b) shows a micrograph of a pit on an examined specimen after exposure in sea salt solution.

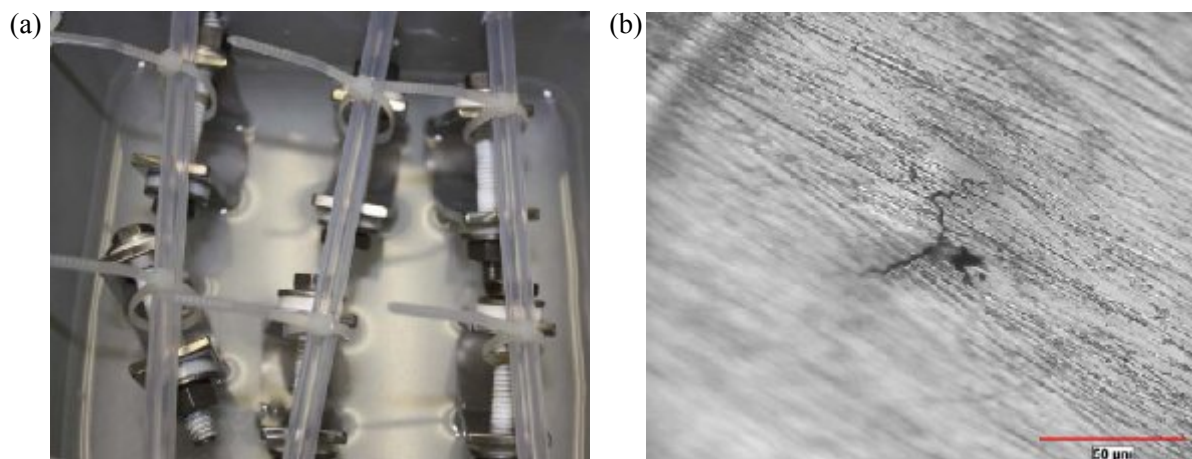


Fig. 6. (a) Photograph of U-bend specimens immersed in equilibrium sea salt solution at 30°C and 90% RH. (b) Micrograph of a pit and apparent cracks on a U-bend specimen immersed for 5 weeks in the sea salt solution.

SCC Testing at Various Stress Levels

The previously described activities, as well as the prior NRC investigation, used U-bend specimens to evaluate SCC susceptibility. These specimens are useful because they are relatively easy to fabricate by standard methods. The specimen configuration, however, represents a relatively stress or strain state, where the strain is in the range of about 14-16% at the apex. The residual stresses in canister welds are not well characterized but it is uncertain whether the U-bend specimen gives a realistic representation of the weld stress state. Therefore the objective of this activity is to perform SCC testing at a lower stress or strain levels than is associated with the U-bend specimens to determine whether this will affect the SCC initiation behavior.

SCC testing was performed using C-ring specimens following ASTM Standard G38-01, “Standard Practice for Making and Using C-Ring Stress-Corrosion Test Specimens” [19]. For the C-ring specimens, the applied stress is controlled by adjusting the deflection of the C-shaped specimen. A photograph of some test specimen is shown in Figure 7. Some C-ring specimens were strained to about 0.3%, where the tensile stress is close to the material yield stress. Other C-rings were strained to about 1.5%, which is still well less than the U-bend strain. The specimens were deposited with 1 or 10 g/m² of simulated sea salt in the same manner described for the U-bend specimens. Specimens were exposed in the environmental test chambers to static conditions of 35°C and 72% RH, 45°C and 44% RH, and 52°C and 32% RH. These represent conditions where the AH is approximately 30 g/m³ at the respective temperatures. Initial results indicate cracking in the 0.3% strain specimens at 35°C and 72% RH with 10 g/m² of salt. This indicates that SCC could initiate at strain or stress level below what is represented by the U-bend specimens.

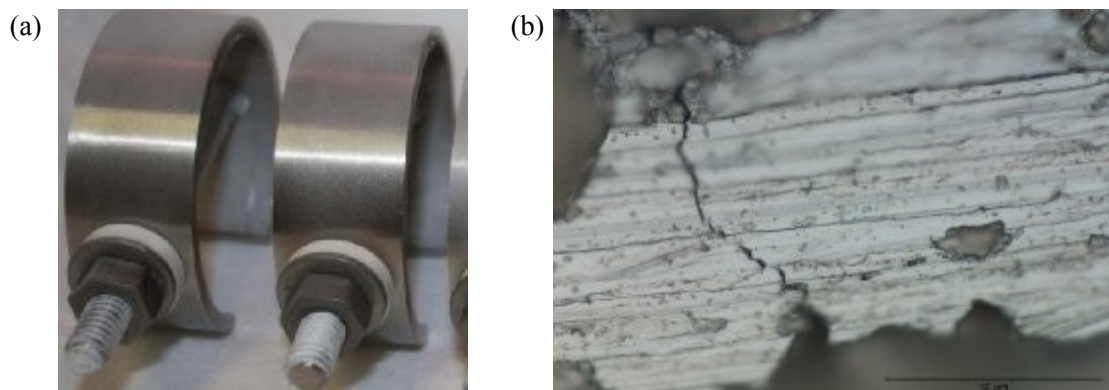


Fig. 7. Photograph of C-ring specimens used for SCC testing.

CONCLUSIONS

NRC has sponsored a research program to investigate various factors that could affect the SCC susceptibility of austenitic stainless steel dry storage canisters exposed to marine chlorides including temperature, humidity, salt concentration, and stress level. These were evaluated by systematic testing to identify conditions where SCC could occur. It is expected that moisture to support electrochemical corrosion reactions could be generated by deliquescence of dry salt. The DRH for sea salt was measured to be close to that of pure $MgCl_2$. Tests using U-bend specimens showed that SCC could initiate for conditions where the ambient RH was near to or greater than the DRH for sea salt. A sufficiently high RH could be achieved within a representative limit of natural AH, particularly at low temperatures. Cracks initiated on specimens with as little as 0.1 g/m^2 of salt on the surface. Tests are ongoing for conditions of high RH and lower stress levels to generate additional data. It is expected that the findings of this test program will help NRC to determine whether additional work by industry is needed to demonstrate that deployed canisters satisfy the safety requirements of 10 CFR, Part 72. Concerning this issue, NRC is engaged with industry and other stakeholders to share information through the Electric Power Research Institute Extended Storage Collaboration Program and Nuclear Energy Institute Risk Informed Resolution Protocol. A full report of this test program is expected as a NUREG/CR in summer of 2013.

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