

# EVALUATION OF AUSTENITIC STAINLESS STEEL DRY STORAGE CASK STRESS CORROSION CRACKING SUSCEPTIBILITY

Mekonen Bayssie (mekonen.bayssie@nrc.gov), Darrell Dunn (darrell.dunn@nrc.gov),  
and Aladar Csontos (aladar.csontos@nrc.gov),  
U.S. Nuclear Regulatory Commission, Washington, DC 20555-0001

Leonardo Caseres (leonardo.caseres@swri.org) and Todd Mintz (todd.mintz@swri.org)  
Southwest Research Institute, 6220 Culebra Road, San Antonio, TX 78238-5166

Many nuclear plants are now storing spent nuclear waste in NRC licensed onsite dry storage facilities, because of limited remaining space in spent fuel pools. Some of these plants are located close to the coast and are in chloride-rich environments. Because most dry storage containers are manufactured from austenitic stainless steel, a potential concern is the susceptibility of the cask materials to chloride induced stress corrosion cracking. The objective of this work was to evaluate the atmospheric chloride stress corrosion cracking susceptibility of austenitic type 304, 304L, and 316L stainless steels. U-bend samples of these alloys were assembled, placed in an environmental chamber heated to various temperatures to simulate decay heat from the spent fuel, and subjected to an accelerated exposure test involving dry deposition of sea salt and cyclic humidity exposure. To date, stress corrosion cracking accompanied by pitting corrosion was observed on all three alloys exposed under conditions where partial deliquescence of the deposited salt occurred.

## I. INTRODUCTION

There are currently over 100 operating nuclear reactors in the United States, many of which have been in operation for multiple decades. During the routine operation of these nuclear power plants, spent fuel must be removed from the reactors and replaced with new fuel. The spent fuel initially is stored in spent fuel pools. However, as the spent fuel pools are reaching capacity, some licensees also have a NRC licensed independent spent fuel storage installation (ISFSI) where the spent fuel is contained in dry storage casks. Most of these dry storage casks are made out of austenitic stainless steel (SS) including UNS S30400 (304 SS), UNS S30403 (304L SS), and S31603 (316L SS).<sup>1,2</sup> Several storage systems have been licensed and utilized. In most of these designs, the SS casks are placed in a concrete bunker that shields the cask from precipitation. The bunkers have air passages for passive cooling. In these designs, airborne

deposition of salts may be possible but periodic exposure to precipitation that may wash away any deposited salts cannot occur. Although there are no reports to date, it is not known if these dry storage containers are susceptible to chloride stress corrosion cracking (SCC) in marine atmospheres which are known to be corrosive as a result of both high relative humidity (RH) and the deposition of chloride containing salt aerosols.<sup>3-5</sup>

Three conditions must be met in order for SCC to occur: the material must be susceptible, sufficient applied or residual stresses must be present, and the exposure environment must promote stress corrosion cracking. The welding process of the canisters can result in residual stresses close to the yield strength of the material. High residual stresses from welding have been shown to be sufficient to initiate SCC.<sup>6</sup>

The susceptibility of austenitic SSs to chloride SCC has been the subject of research investigations for many years.<sup>7-14</sup> The relative susceptibility of the materials are generally known to be 304SS > 304L > 316L.<sup>15-17</sup> The difference in the SCC susceptibility for the various austenitic SSs can be related to the chemical composition. Lower carbon content in the austenitic SS, such as type 316L or 304L tends to decrease the SCC susceptibility. It has been suggested that the carbon, which can form precipitates at the grain boundaries, may lead to the formation of a less protective passive film resulting in an increased SCC susceptibility.<sup>18</sup> The main compositional difference in the type 316L alloy versus the type 304 and 304L alloys, is the addition of 2-3 percent molybdenum that enhances passivity and reduces the localized corrosion and SCC susceptibility.<sup>19</sup>

The effect of chloride concentration in solution has been studied extensively on material degradation including SCC.<sup>20,21</sup> For atmospheric corrosion, the concentration of chloride containing salt was found to be highest closest to large bodies of salt water.<sup>5,22,23</sup> Temperature has also been shown to affect the SCC susceptibility of austenitic SSs. It has been reported that in acidic chloride solutions (e.g., pH 2), SCC was unlikely to occur below 65°C [149°F].<sup>21</sup> Ford and Silverman have

conducted some testing on sensitized type 304 SS, which showed the importance of oxidizing compounds (i.e., air, chloride, etc.). SCC was observed at temperatures as low as 40 °C [104 °F], but only in the presence of significant concentrations of dissolved oxygen.<sup>24</sup> In another study, type 304 SS was observed to be susceptible to SCC in 45°C [113°F] magnesium chloride solutions.<sup>12</sup> While the initiation of SCC at low temperatures may be dependent on material and environment combinations, testing has shown that the SCC susceptibility of austenitic SSs increases with temperature.

Previous tests on austenitic SSs at Southwest Research Institute (SwRI) were conducted using simulated seawater solution directly sprayed onto the heated test specimens.<sup>25</sup> Stress corrosion cracking was reported on type 304, 304L, and 316L specimens. While these results indicate that periodic exposure to simulated seawater can induce SCC, it was noted that the test method used was highly conservative and not representative of conditions expected for spent fuel storage casks.

Recently, specific testing has been conducted to evaluate atmospheric SCC of multiple austenitic and ferritic-austenitic SSs.<sup>26-28</sup> Cracking of austenitic and ferritic-austenitic SS have been evaluated as a function of temperature, alloy composition, RH, and salt composition.<sup>26</sup> No cracking of type 304 SS or 316L SS was observed with NaCl even at RH values sufficient for deliquescence. Cracking of type 304 SS and 316L SS was noted in CaCl<sub>2</sub> at temperatures of 30°C [86°F] and RH of 30 percent. It should be noted that the surface chloride concentrations with CaCl<sub>2</sub> were determined to be 290,000 mg/m<sup>2</sup>. Fairweather et al.<sup>27</sup> reported cracking of type 304L SS contaminated with MgCl<sub>2</sub> with surface chloride concentrations of 20 and 100 mg/m<sup>2</sup> at temperatures as low as 45°C [113°F]. Cracking was dependent on RH, with maximum susceptibility between 30 and 60 percent. Rough machining that imparted higher surface stresses (based on hardness measurements) also increased SCC susceptibility. Hayashibara et al.<sup>28</sup> reported SCC of type 304 SS tensile specimens at temperatures in the range of 60 to 80°C [140 to 176°F] at RH values of 35 and 50 percent. Chloride was deposited by placing a 10 µL drop of artificial sea water on the specimens.

Research conducted to date has addressed many important environmental and metallurgical factors important to the SCC of SS. However, there is insufficient information to determine the range of conditions where austenitic SS spent fuel dry storage casks contained in ventilated concrete enclosures may be susceptible to SCC. The objectives of the work described in this paper were to develop an accelerated SCC test method, considering the range of possible exposure

environments, and evaluate the effects of temperature, welding, and heat affected zones on the SCC susceptibility of type 304, 304L, and 316L SS in marine atmospheres.

## II. MATERIALS AND METHODS

Three different SS alloys 304, 304L, and 316L were prepared for testing. The chemical compositions of the alloys and the weld filler metals are shown in Table 1. Both single U-bend and double U-bend samples were machined from 0.318 cm [0.125 in] thick alloy sheets. The specimens were machined with the rolling direction perpendicular to the width of the sample. Both the single and double U-bend specimens were bent around a 1.27 cm [0.50 in] diameter mandrill following the ASTM G 30 procedure.<sup>29</sup> Double U-bend specimens were only produced from type 304 SS and type 316L SS, while all three SS alloys were used to construct the single and welded U-bend specimens.

Welded specimens were prepared using a procedure and welder qualified in accordance with ASME Section IX.<sup>30</sup> After the Gas Tungsten Arc Weld (GTAW) was completed, the weld was non-destructively examined using radiographic testing<sup>31</sup> in accordance with ASME Section III and the acceptance criteria in ASME Section III WB-5000. Satisfactory material was then machined into U-bend specimens that met the requirements of ASTM G 58 with the weld located at the apex of the specimen after bending.<sup>32</sup> All specimens were individually marked for identification. After bending, each specimen was also examined using a stereomicroscope capable of 8 to 20× magnification to verify that no cracks or fissures were present prior to exposure.

Salt deposit test specimens were similar to the U-bend specimens except that the specimens were shorter and they did not have extended legs or use a pinch bolt. The design of the salt deposit specimen was intended to be similar to the strained portion of a U-bend specimen but allow ease of placement into, and removal from, the test chamber. Eliminating the pinch bolt also simplified the determination of salt deposition rates.

The U-bend and salt deposition specimens were placed in the test chamber on cylindrical heaters. Specimens were positioned with the apex of the bend facing up so that salt deposits could accumulate on the specimen surfaces with the highest residual stresses. Calibrated thermocouples installed in instrumented U-bend specimens were used to monitor temperature. Salt deposition was accomplished using short fogging periods with ASTM D1141-52 simulated sea water<sup>32</sup> while the specimens were maintained at a temperature of 95°C

[203°F] for a period of 2 weeks. Prior to implementing this protocol specific tests were conducted to determine the condition of the specimens after salt deposition. These initial tests revealed significant accumulation of salt on the specimens but no indication of uniform, localized or stress corrosion cracking. After the accelerated salt deposition was completed, the operation of the chamber was changed to cyclic humidity with periodic salt deposition. This testing protocol was designed to expose the specimen with deposited salt to humidity variations while maintaining or increasing the amount of accumulated salt on the specimens. Specimens were maintained at the designated test temperatures of 40°C [104 °F], 85°C [185°F], and 120°C [248°F]. The details of the chamber operation for each protocol is provided in Tables 2 and 3. A calibrated combination probe was used to measure temperature and humidity variations as a function of location within the chamber. A segment of the relative humidity vs. time during the cyclic humidity with periodic salt deposition protocol is shown in Figure 1. Specimens were exposed for periods of 4, 16 and 32 weeks after the initial 2 week salt deposition period. Additional specimens will be tested for a period of 52 weeks. Triplicate samples for each material (i.e. type 304 SS, 304L SS or 316L SS) and specimen configuration (i.e., double, single, and welded) were exposed for each temperature and time combination.

### III. RESULTS

Measured salt deposition during the 2 week accelerated salt deposition protocol and the first 16 weeks of the cyclic humidity with periodic salt deposition are shown in Table 4. The amount of salt deposited in the initial 2 week deposition period was reasonably consistent for the samples examined. After the 2 week salt deposition period, the specimen temperatures were adjusted and maintained at predetermined values according to the test plan. Salt deposition specimens retrieved after 4 and 16 weeks indicated that the amount of salt on the specimens was being maintained at a value recorded after the 2 week deposition period. Deposition measurements for the specimens at 40°C [104 °F] were likely confounded by the presence of corrosion products however, visual examination of the specimens indicated significant salt deposits were present even after 16 weeks of testing. Specimens maintained at 85°C [185°F], and 120°C [248°F] had no visual indications of corrosion products.

Appearance of the U-bend specimens after accelerated salt deposition plus 4 and plus 16 week exposures are shown in Figure 2. After the accelerated salt deposition, all specimens were mostly covered with a

uniform salt deposit. Specimens maintained at 85°C [185°F], and 120°C [248°F] after accelerated salt deposition maintained a similar appearance and showed no signs of corrosion product accumulation. Salt deposits on these specimens appeared to be maintained even after 16 weeks of cyclic humidity exposure. In contrast, specimens maintained at 40°C [104 °F], showed visual evidence of corrosion after 4 weeks. Specimens exposed for 16 weeks at 40°C [104 °F] also showed visual evidence of corrosion suggestive of cracking and the appearance of the corrosion products (Figure 2d).

Results of post exposure examination of the specimens are shown in Table 5. After 4 weeks, cracking was observed on all of the type 304 specimens maintained at 40°C [104 °F]. This included both single and double U-bend specimens and welded U-bend specimens. For the welded specimens, cracking occurred in the base metal next to the weld and likely included the heat affected zone. No cracking was observed in the type 308 weld metal. After 16 weeks, SCC was observed in both the type 304 and 304L specimens. Consistent with the results obtained at 4 weeks, cracking of the welded specimens occurred in the heat affected zone next to the weld for both the welded type 304/308 and 304L/308L specimens. None of the type 304, 304L, and 316L specimens maintained at 85°C [185°F], and 120°C [248°F] cracked. Also, no type 316L specimens tested at 40°C [104 °F] cracked after testing for 4 and 16 weeks. However, cracking of the type 316L specimens tested at 40°C [104 °F] was observed after 32 weeks.

Optical photos of the cracked type 304 SS material after 16 weeks of testing are shown in Figure 3. The cracks appear to be intergranular and have characteristic branching. Comparison of the 4 week and 16 week samples suggests that cracking of the type 304 and 304L specimens may have initiated prior to the 4 week inspection and continued to propagate. After 4 weeks of cyclic humidity exposure, the cracks on the type 304 and 304L specimens were small and narrow. Wider and longer cracks were observed on the 16 week specimens.

### IV. DISCUSSION

SCC was only observed on specimens tested at 40°C [104°F]. No SCC was observed on the type 304, 304L and 316L specimens tested at 85 and 120°C [185 and 248°F]. In addition, no SCC was observed on the type 316L specimens at any temperature for exposure periods of 4 and 16 weeks.

Profiles of the absolute humidity, RH and temperature as a function of distance from the test specimens are shown in Figure 4. For these data, the chamber temperature was fixed at 35°C [95°F] and the

ambient RH (i.e. away from the specimens) was maintained at 94 percent. Although no data was obtained with specimens at 40°C [104°F], the trend measured for specimens at 60 to 120°C [140 to 248°F] provides an indication of how the RH varied with both specimen temperature and distance from the specimen. Because the test specimens were at an elevated temperature compared to the ambient environment inside the chamber, the temperature and RH were both dependent on distance from the specimen especially between 4.45 cm [1.75 in] and 1.3 cm [0.5 in]. The temperature increased sharply over this distance and the RH dropped significantly. The relative humidity surrounding the 60 °C [140 °F] samples remained above 40 percent at a distance of 1.3 cm [0.5 in]. Closer to the surface of the U-bend, the relative humidity was estimated to be about 30 percent which is near the deliquescence RH for MgCl<sub>2</sub>. For the 120°C [248°F] specimens, the RH near the specimen surface was approximately 30 percent. As expected, the absolute humidity was virtually independent of distance from the specimen so the RH variation is strictly a consequence of higher local temperature adjacent to the heated specimen surface.

Cracking of only the specimens tested at 40°C [104°F] indicates that the environment for SCC is present at that temperature. For the data shown in Figure 4, it is apparent that salt deposits on specimens maintained at 40°C [104°F] may undergo partial deliquescence during the high humidity periods. This is consistent with observed changes in appearance of the salt deposits on the 40°C [104°F] specimens at high humidity reported by Mintz and Dunn<sup>25</sup> for both simulated and natural sea salt. Under these conditions, MgCl<sub>2</sub> and CaCl<sub>2</sub> salts absorbed enough water to support the electrochemical reactions necessary for pitting and SCC.

In this study, cracking was first observed with type 304 and 304L specimens. Cracking of type 316L specimens was not observed until the specimens had a cumulative exposure of 32 weeks. This result suggests that type 316L SS is more resistant to cracking than either type 304 or 304L SSs at 40°C [104°F]. However the previous work reported by Prosek et al.<sup>26</sup> suggests that type 316L SS is susceptible to chloride SCC at even lower temperatures with CaCl<sub>2</sub> solutions. Prosek et al.<sup>26</sup> also reported that no SCC occurred in NaCl solutions. It is likely that the specimens tested in this study were exposed to a much lower chloride concentration as a result of partial deliquescence of sea salt deposits compared with the high chloride concentrations obtained with the CaCl<sub>2</sub> solutions used by Prosek et al.<sup>26</sup>

No cracking was observed in the weld metal on any of the test specimens. Cracking in the welded type 304 SS and 304L SS specimens was located in the base metal adjacent to the welds. This result may be caused by

several factors including the formation of a heat affected zone with sensitized grain boundaries especially for type 304 SS. Uneven bending is possible with welded U-bend specimens as a result of the slightly higher yield strength of the weld metal. The adjacent base metal that includes a sensitized heat affected zone, may also have a smaller bend radius resulting in higher residual stresses.

The SCC susceptibility of austenitic SS in chloride solutions generally increases with temperature. However, at elevated temperatures, no cracking was observed on any of the test specimens. It is apparent from the results shown in Figure 4 that at 85°C [185°F], and 120°C [248°F] the RH adjacent to the heated specimen surfaces was too low for even partial deliquescence to occur. Because there was no chloride containing solution in contact with the specimens at elevated temperatures, SCC was not initiated.

It should be noted that the results obtained in these tests are also likely conservative compared to the actual condition of a SS storage cask contained within a concrete bunker. Although natural processes were simulated for salt deposition and RH variations, it is possible that the temperature gradient within a concrete bunker may be lower owing to the insulating properties of the concrete containment even with natural ventilation. In turn, the RH inside the bunker will be lower and deliquescence of the salt may not occur even at temperatures at or near 40°C [104°F]. In addition, the maximum humidity inside the test chamber was close to 60 g/m<sup>3</sup>. While theoretically possible, such conditions have not been observed in natural environments. A review of atmospheric data suggests that maximum absolute humidity values were typically less than 30 g/m<sup>3</sup> even in warm coastal environments. With lower absolute humidity, RH near the specimens would also be lower and partial deliquescence of any deposited salts would be unlikely.

While the tests conducted were likely conservative, the results are significant because it has been demonstrated that deliquescence of dry deposited sea salts can lead to SCC of type 304, 304L and 316L SS at slightly elevated temperatures.

## V. CONCLUSIONS

An accelerated test protocol to evaluate the atmospheric SCC of austenitic stainless steels was developed. The test protocol consisted of accelerated salt deposition followed by cyclic humidity exposure with periodic salt deposition. The test protocol is significantly different from previously developed atmospheric exposure tests because there are no spraying cycles employed that directly expose the specimens to aqueous test solutions.

Testing of type 304 SS, 304L SS and 316L SS U-bend specimens was conducted. SCC of the type 304 SS and 304L SS exposed at 40°C [104°F] was observed. No SCC was observed on any specimens tested at 85°C [185°F], and 120°C [248°F]. The observation of SCC of type 304, 304L, and 316L SS at 40°C [104°F] is consistent with the observation of partial deliquescence of the dry deposited sea salt during periods of high relative humidity. No deliquescence occurred on specimens maintained at 85°C [185°F] and 120°C [248°F]. The delayed observation of SCC on the type 316L SS specimens tested at 40°C [104°F] is consistent with the increased localized corrosion and SCC resistance of type 316L SS compared to either type 304 SS or 304L SS.

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Table 1. Composition of Stainless Steel Alloys Tested

Material	Fe	Cr	Ni	Mo	Mn	C	S	P	N	Si	Cu
Type 304 Heat 2N379	Bal	18.19	8.07	N/A	1.21	0.039	0.002	0.026	0.042	0.55	N/A
Type 304L Heat 7470395	Bal	18.14	8.07	0.18	1.29	0.025	0.001	0.025	0.032	0.34	0.27
Type 316L Heat 7470663	Bal	16.43	10.13	2.06	1.35	0.019	0.0006	0.027	0.022	0.51	0.32
ER308 Heat E72000	Bal	19.92	9.61	0.10	1.55	0.051	0.002	0.023	0.019	0.36	0.14
ER308L Heat D88069A	Bal	20.12	9.79	0.05	1.75	0.009	0.010	0.014	0.043	0.47	0.05
ER316L Heat S66892	Bal	18.10	11.05	2.22	1.63	0.023	0.016	0.026	0.04	0.41	0.39

Table 2. Atmospheric Test Chamber Protocol for Accelerated Salt Deposition

Cycle #	Chamber cycle	Time, minutes	Chamber temp, °C	Comments
1	Salt fog	5	30	Deposit salt on specimens
2	Dry	15	30	Drying of specimens after fog cycle

Table 3. Atmospheric Test Chamber Protocol for Accelerated Atmospheric Testing and Periodic Salt Deposition

Cycle #	Chamber cycle	Time, minutes	Comments
1	Salt fog	5	Deposit salt on specimens
2	Ambient	60	
3	Salt fog	5	Deposit salt on specimens
4	Ambient	60	
5	Salt fog	5	Deposit salt on specimens
6	Ambient	60	
7	Salt fog	5	Deposit salt on specimens
8	Ambient	60	
9	Dry	100	Low relative humidity
10	Increase Humidity	125	Relative humidity in chamber increased
11	High Humidity	55	High relative humidity exposure
12	Extended dry	180	Low relative humidity

Table 4. Weight Change for Salt Deposition Specimens

Exposure Temperature, °C	Weight change, mg/m <sup>2</sup>		
	2 weeks salt deposition (all specimens at 95 °C)	2 weeks salt deposition (95 C) + 4 weeks at exposure temperature	2 weeks salt deposition (95 C) + 16 weeks at exposure temperature
40	18,677	51,847*	81,400*
95	23,405	27,667	34,700
130	18,290	35,262	70,900

\*Corrosion products visible on specimen after exposure

Table 5. Results of U-Bend Test Specimens

Alloy and Specimen Configuration	Temperature °C	SCC Observed vs. Exposure Time			
		4 weeks	16 weeks	32 weeks	52 weeks
Type 304 Single U-bend	40	<b>Yes</b>	<b>Yes</b>	<b>Yes</b>	In test (TBD)
	85	No	No	No	In test (TBD)
	120	No	No	No	In test (TBD)
Type 304/308 Welded Single U-bend	40	No	<b>Yes</b>	<b>Yes</b>	In test (TBD)
	85	No	No	No	In test (TBD)
	120	No	No	No	In test (TBD)
Type 304 Double U-bend	40	<b>Yes</b>	<b>Yes</b>	<b>Yes</b>	In test (TBD)
	85	No	No	No	In test (TBD)
	120	No	No	No	In test (TBD)
Type 304L Single U-bend	40	No	<b>Yes</b>	<b>Yes</b>	In test (TBD)
	85	No	No	No	In test (TBD)
	120	No	No	No	In test (TBD)
Type 304L/308L Welded Single U-bend	40	No	<b>Yes</b>	<b>Yes</b>	In test (TBD)
	85	No	No	No	In test (TBD)
	120	No	No	No	In test (TBD)
Type 316L Single U-bend	40	No	No	<b>Yes</b>	In test (TBD)
	85	No	No	No	In test (TBD)
	120	No	No	No	In test (TBD)
Type 316L/316L Welded Single U-bend	40	No	No	<b>Yes</b>	In test (TBD)
	85	No	No	No	In test (TBD)
	120	No	No	No	In test (TBD)
Type 316L Single U-bend	40	No	No	<b>Yes</b>	In test (TBD)
	85	No	No	No	In test (TBD)
	120	No	No	No	In test (TBD)

TBD: To be determined when samples are removed and examined

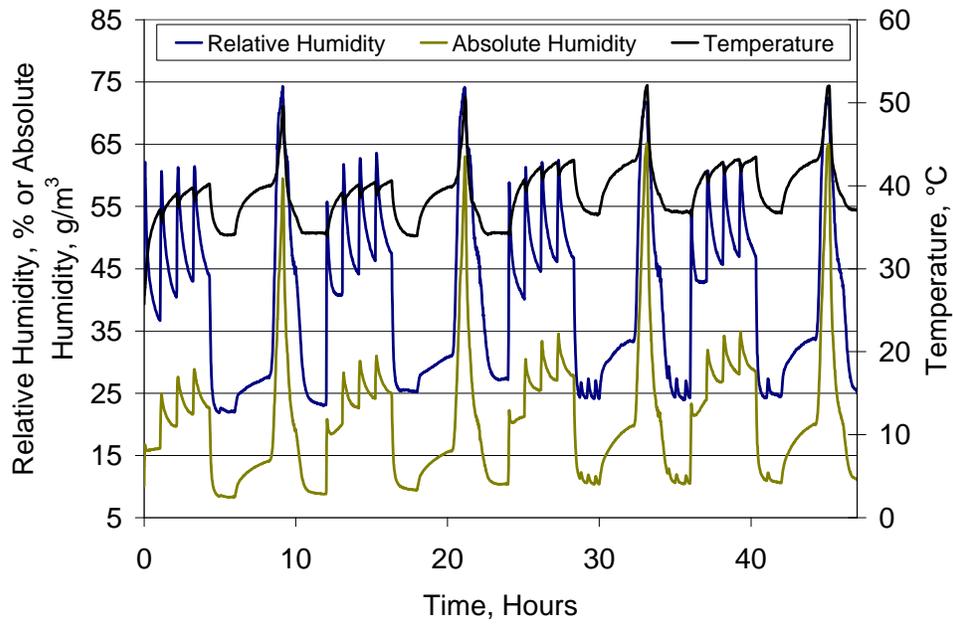


Figure 1. Atmospheric Conditions In The Test Chamber During The Accelerated Test Protocol After The 4-Week U-Bend Sample Pull

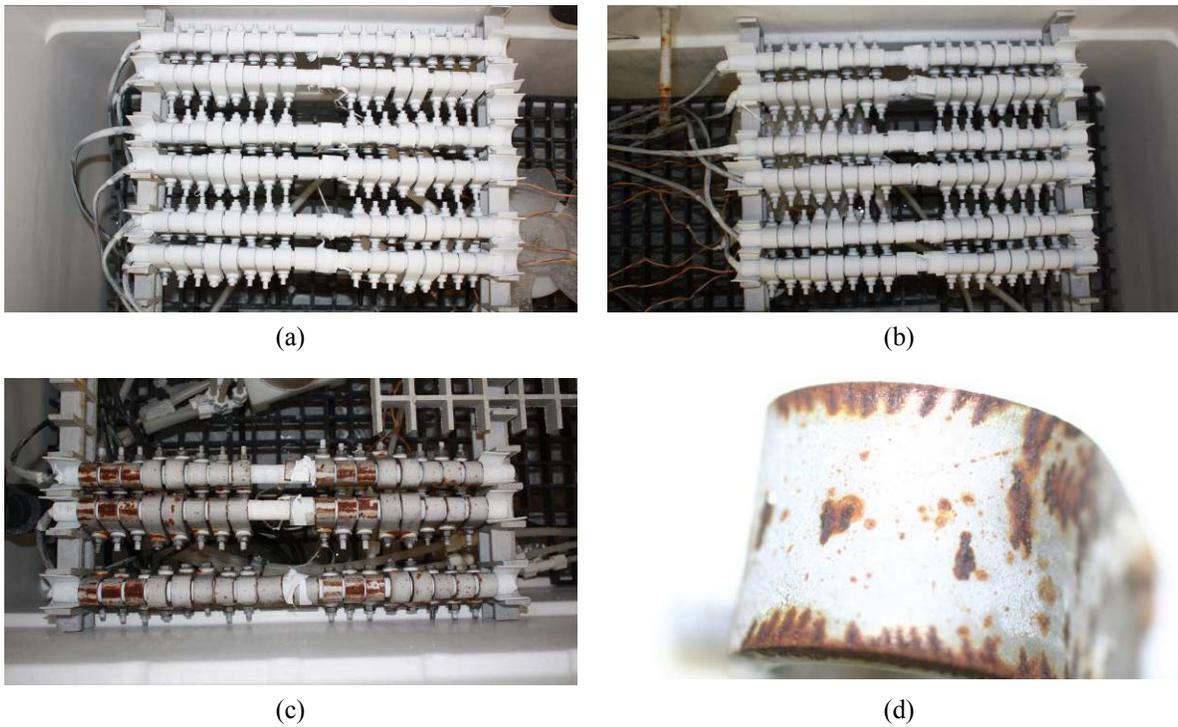


Figure 2. Images Of U-Bend Samples Exposed For 4-Weeks Of Accelerated Atmospheric Testing At A Temperature Of (a) 135 °C [248 °F], (b) 85 °C [185 °F], (c) 40 °C [104 °F] and (d) Type 304 specimen exposed for 16 weeks at 40 °C [104 °F].

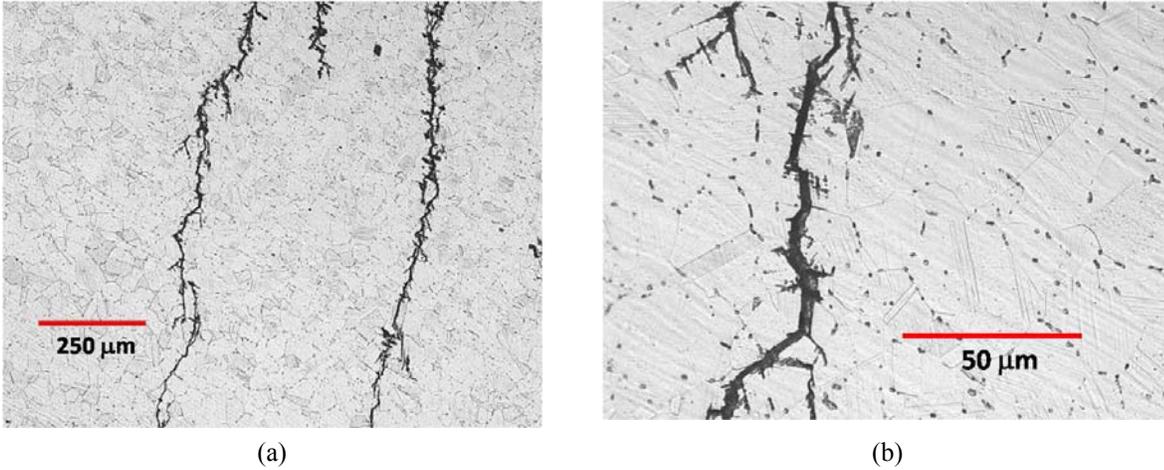


Figure 3. Image of Stress Corrosion Crack of 304 43 °C [109 °F] Single U-Bend Sample Tested for 16 Weeks

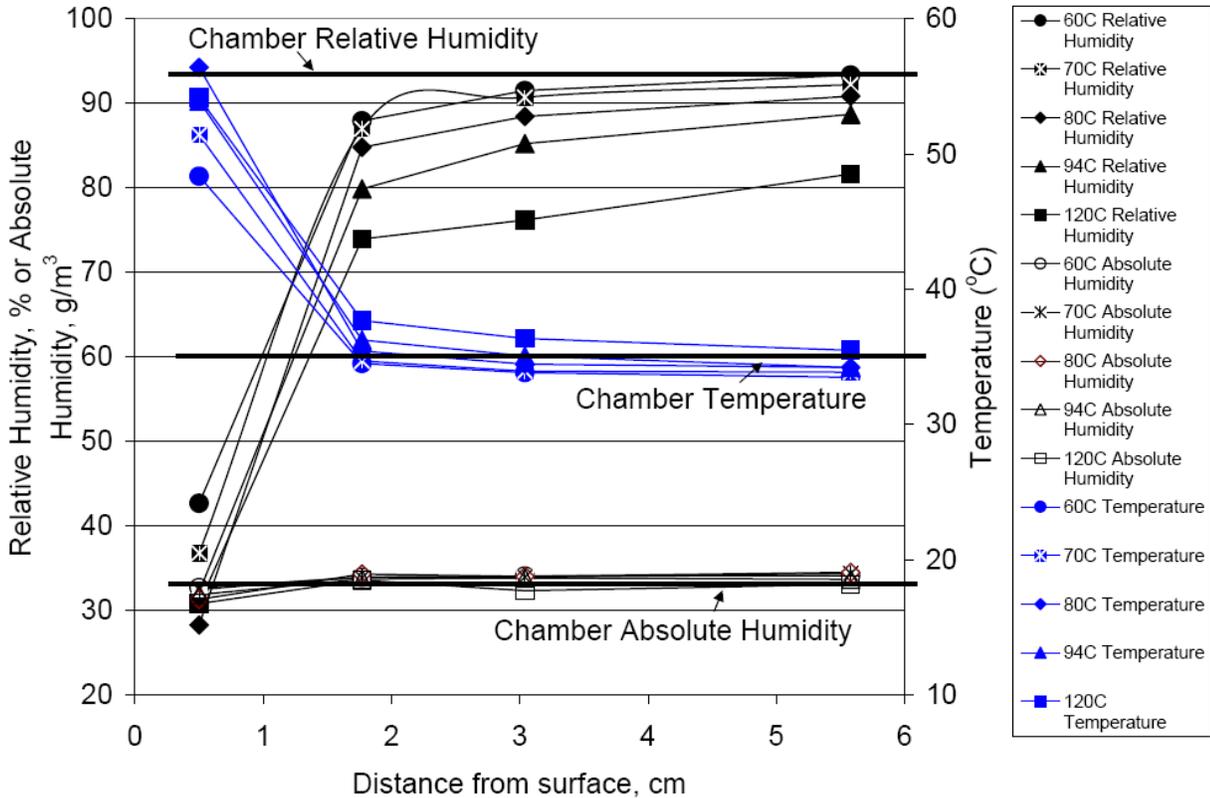


Figure 4. Relative Humidity, Absolute Humidity, and Temperature as a Function of Distance and Specimen Temperature.