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EFFECT OF HEATING RATE ON CAUSTIC STRESS CORROSION CRACKING

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INTRODUCTION

To evaluate effects of a large water leak into the sodium side of a steam generator in a Liquid Metal Fast Breeder Reactor the Liquid Metal Engineering Center (LMEC) at Canoga Park, California, is performing a series of tests in a Large Leak Test Rig (LLTR). This test series involves heating a large steam generator that possibly contains localized pockets of aqueous caustic retained from a previous sodium-water reaction. Such pockets of caustic solution could be in contact with welds and other components that contain residual stresses up to the yield point. The LMEC and General Electric (GE) ran a series of tests to evaluate the effect of heating rate on caustic stress corrosion cracking (SCC) for alloys either used or considered for the LLTR.

A summary of the temperatures and caustic concentration ranges that can result in caustic SCC for carbon steel and Type-304 stainless steel is given in Figure 1.[1] The intersection of the NaOH–H_2O boiling point curve with regions of caustic SCC are particularly important for alloys exposed to caustic solutions and heated from a low to high temperature at 1 atm. At low temperatures and low concentrations, no localized attack occurs. In the high-temperature, high-concentration regions, localized attack (manifested by the formation of blunt, finger-like penetrations) into the metal matrix

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occurs. In the intermediate regions indicated in Figure 1, caustic SCC occurs. The low-, intermediate-, and high-temperature interactions of the caustic solutions with [illegible] specimens are illustrated in Figure 2. In a system open to the atmosphere, it is possible to avoid the regions of caustic stress corrosion susceptibility by starting with a low caustic concentration and heating at a very low heating rate. In this case, the caustic solution can concentrate by low-temperature evaporation to a high concentration, but the solution temperature can be maintained below the minimum temperature necessary for caustic SCC. A second method that can be used to avoid caustic SCC is rapidly heating through the susceptible ranges of caustic concentration and temperature, such that the time spent in the susceptible regions is less than that required for crack initiation. The determination of the heating rates required to avoid caustic SCC was the subject of these studies.

SPECIMEN CONFIGURATION AND GENERAL STRESSING METHODS

Three types of stressed specimens were used. The first type (Figure 3) was formed from rectangular metal sections, 2.54 cm (1 in.) by 1.27 cm (0.5 in.) with a thickness of 0.32 cm (0.13 in.). A spot weld was placed on the face, and the specimen was bent around a cylindrical mandrel, 1.27 cm (0.5 in.) diameter, to form a C-ring with the spot weld on the convex side. Springback of the specimen then occurred which resulted in tensile stresses on the concave side and compressive stresses on the convex side. A second spot weld was then placed on the concave side which when combined with the springback tensile stresses, assured that tensile stresses up to the yield point were present adjacent to the spot weld. The specimen then had an area with high residual tensile stresses adjacent to the weld on the concave side and a second welded region with similar metallurgical structure, but not under residual tensile stresses on the convex side.

The second sample configuration was similar to the first except it consisted of two 1.27 cm (0.5 in.) by 1.27 cm (0.5 in.) sections welded together. By this technique, the effect of galvanic coupling on caustic SCC could be determined. The welded samples were bent around a mandrel to form a C-ring in the same manner as the first type sample and allowed to spring back. A spot weld was then placed on each half (convex side) of these C-rings. No spot welds were placed on the concave side. Thus the two types of C-ring specimens were self-stressed by forming into a C-ring and appropriate spot welding.

The third type specimen was a flat dog-bone tensile type (Figure 4). The tensile samples were loaded in a stressing facility and inserted into a 2-gallon Type-316 stainless steel pressure vessel. Figure 5 shows the stressing arrangement with one sample coupled to one piston. In an actual run, eight samples were connected to eight pistons. Because of the danger of caustic cracking, the pressure vessel was fitted with a nickel liner. The pistons were pressurized individually so that a predetermined stress was maintained on each sample during the experiment. All internal fixtures of the stressing facility were fabricated from nickel or high nickel alloys for protection against caustic cracking. The pull rods were fabricated from Inconel-X750. A complete description of the testing arrangements has been published.[2]

MATERIALS

The specimens used to form C-rings from the single alloys were fabricated from Type-304 stainless steel with 0.06% C, 2¼ Cr-1 Mo steel with 0.10% C, SAE-1008 carbon steel and Inconel 600. In addition, tab halves of some of the above alloys were welded together to produce four galvanic combinations; Type-304 stainless steel welded to Type-304 stainless steel, Type-304 stainless steel welded to 2¼ Cr-1 Mo steel, Type-304 stainless steel welded to carbon steel and 2¼ Cr-1 Mo welded to carbon steel. Because the C-ring specimens were used for preliminary experiments, the chemical analyses and mechanical properties are not presented.

The dog-bone tensile samples were fabricated from Type-304 stainless steel, 2¼ Cr-1 Mo steel and carbon steel (ASTM-A516 Grade 70). The above carbon steel in the plate form is similar to the replacement piping steel that could be used for the LLTR. The heat numbers, significant mechanical properties, and chemical composition of the alloys used for tensile samples are given in Table 1. The alloys investigated were used in several metallurgical conditions. Type-304 stainless steel was tested in the mill-annealed, solution-annealed,* and furnace-sensitized** conditions; 2¼ Cr-1 Mo was tested in the as-received (normalized and tempered) and annealed conditions,*** and the carbon steel was tested in the as-received condition.

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*Heated at 1900 F (1038 C) for 1 h, water quench.
**Heated at 1150 F (621 C) for 24 h.
***Heated at 1695 F (924 C) for 20 min, cool to 1350 F (732 C) and hold for 45 min, air cool.

GENERAL APPROACH

Two series of experiments were conducted. The first series was conducted at the LMEC. The experiments were with the C-ring samples fabricated from the single alloys as well as the welded tab halves. Those experiments conducted with the C-rings fabricated from a single alloy were conducted in the open atmosphere. The experiments with the C-rings fabricated from the welded halves, duplex samples, were conducted in a nitrogen atmosphere. The C-rings were heated in beakers of caustic on a hot plate at predetermined rates to specific temperatures. The purpose of this first series was to determine the general weight loss and caustic SCC trends, galvanic effects, and to establish the heating rates of interest for a more in-depth second series of experiments.

The second series of experiments was conducted with tensile samples maintained over a range of predetermined stresses and heated in a well-controlled atmosphere to a specific temperature. These experiments were performed at the General Electric Company (GE) Pleasanton, California. Each series of experiments is discussed below.

FIRST EXPERIMENTAL SERIES

In the initial series of experiments, solutions of 33% or 10% NaOH in distilled water were heated in Teflon beakers to 180 F (82 C) on a hot plate. At this temperature, three weighed duplicate C-ring specimens were introduced into the beakers. A nickel-clad thermocouple was used to measure temperature. The solutions were then heated at specific rates. Experiments were conducted at heating rates of 4.5 F/h (2.5 C/h), 9 F/h (5 C/h), 18 F/h (10 C/h), and 45 F/h (25 C/h).

A description of the first experiment with a starting solution of 33% NaOH provides the typical procedure used to test the C-rings. At 180 F (82 C) heating commenced at a rate of 18 F/h (10 C/h). Distilled water was added in small amounts up to 205 F (96 C) to keep the solution from concentrating before boiling. From 205 to 240 F (96 to 116 C) 33% NaOH solution was added, since a considerable amount of water boiled away. From 240 to 260 F (116 to 127 C), 50% NaOH was added to keep the solution level constant, as large quantities of water continued to boil away and the solution concentrated according to the 1 atm (101 kPa) boiling curve indicated in Figure 1. When the solution concentration reached 50% NaOH at about 290 F (143 C), no further additions were made and the solution continued to concentrate according to the boiling curve. At 450 F (232 C) the specimens were removed from the caustic and quenched to room temperature in a solution of 50% NaOH. The specimens were then ultrasonically cleaned, dried, and weighed. The C-rings were then examined macroscopically for evidence of caustic SCC and prepared for metallographic examination. The samples were cut through the spot welds in a manner that resulted in C-shaped cross sections. Appropriate sections in the vicinity of the welds were prepared from both the convex and concave sides. The section cut from a C-ring is shown in Figure 6.

The same procedure was used for the experiments conducted with an initial concentration of 10% NaOH, except the highest test temperature was 360 F (182 C). A single experiment was conducted at the fastest heating rate, 45 F/h (25 C/h) with a starting solution of 33% NaOH and a finishing temperature of 360 F (182 C).

The experiments with the duplex C-rings were all conducted with a starting solution of 33% NaOH, a heating rate of 18 F/h (10 C/h) and a finishing temperature of 450 F (232 C). In these experiments the beaker and hot plate were placed in a plastic bag-type glove box and a nitrogen purge was maintained.

SECOND EXPERIMENTAL SERIES

The second series of experiments with the tensile samples was run in the container and stressing assembly (Figure 5).

In the first two runs, D001 and D002, no attempt was made to insulate the tensile samples from the stressing fixture electrically. Since the samples are connected to pins, clevices, and pull-rods, galvanic coupling between samples or other internal portions of the stressing facility could occur. Data obtained from the welded tab halves tested at LMEC indicated the presence of an anodic alloy, carbon steel, coupled to Type–304 stainless steel could prevent the stainless steel from cracking in caustic. Thus, in the runs following D002, the specimens were insulated electrically from the retaining plate and clevices by using pre-oxidized Zr–2.5 Nb retaining pins, bottom shims of pre-oxidized Zircaloy–4 and strips of Teflon, to avoid any galvanic effects.

The experimental method used in the second series was to fill the nickel liner in the autoclave body with proper volume and concentration of NaOH solution, bolt the autoclave head containing the samples and stressing facility to the autoclave body, and evacuate the autoclave. The autoclave was then purged with nitrogen at atmospheric pressure and the purge maintained during the entire heating period. The pistons were then pressurized which stressed the samples, and the heating was initiated at a predetermined rate. During the heating period, the solution concentrated according to the NaOH-H_2O boiling point curve shown in Figure 1. At 600 F (316 C), the heaters were shut off, the pistons de-pressurized, the autoclave head unbolted, and the head containing the samples was quenched in a bucket of water. After cooling, the samples were removed from the stressing facility, dimensionally measured, and examined with an optical stereoscope to 140X. Selected samples were also examined nondestructively with a scanning electron microscope and eventually sectioned for metallographic examination.

In the first experiment, D001, a starting solution of 16% NaOH was used and a heating rate of 18 F/h (10 C/h) was desired. The heating rate (Figure 7a) was considerably below the required rate especially in the 220 to 240 F (104 to 116 C) range. In this temperature range, large amounts of steam must be formed and vented to increase the temperature and concentration of the internal solution. In this run, the solution level eventually dropped below the gage section of the stressed samples. By calculation and experimental measurements, it is believed the samples were covered with solution until 320 $\pm$ 10 F (160 $\pm$ 6 C). Run D001 was terminated at 500 F (260 C). All subsequent runs were conducted with a starting solution of 35% NaOH and sufficient volume such that the solution could concentrate to 95% without uncovering the samples.

Runs D002 and D003 were conducted at the 18 F/h (10 C/h) heating rate. Run D003 was conducted to determine whether the use of electrically conducting sample retaining pins had influenced the stress corrosion behavior by galvanic coupling to the more cathodic nickel structural alloys, as discussed previously.

Run D004 was conducted at nominal heating rates of 36 F/h (20 C/h). However, as shown in Figure 7b, the heating rate achieved was below the required rate in the lower temperature regime. Thus, the run was repeated (D005, shown in Figure 7c), to obtain an adequate heating rate in the temperature range where the caustic solution undergoes the greatest change in concentration.

Run D006 shown in Figure 7d was conducted at the slowest heating rate, 9 F/h (4.5 C/h) and was the final experiment of the third series.

TEST RESULTS AND DISCUSSION

FIRST SERIES OF TESTS

Type-304 Stainless Steel (0.06% Carbon)

All the Type-304 stainless steel C-rings heated at rates below 45 F/h (25 C/h) showed evidence of caustic SCC on the concave surfaces by the spot weld with residual tensile stresses. The surfaces by the spot weld on the convex side of the C-rings showed no SCC. The extent of SCC was very slight at 18 F/h (10 C/h), more at 9 F/h (5 C/h), and most severe at 4.5 F/h (2.5 C/h) as shown in Figures 8 and 9. The 33% NaOH initial concentration with the finishing temperature of 450 F (232 C) resulted in slightly more SCC than did the 10% NaOH initial concentration heated to 360 F (182 C). For example, at the 18 F/h (10 C/h) heating rate, SCC was extremely slight in the 10% NaOH test (Figure 10). The prevalent theory of caustic SCC indicates cracking of Type-304 stainless steel at atmospheric pressure should occur only in the 250 to 350 F (121 to 177 C) range. The concentration of the caustic that started initially at 10% NaOH and the caustic that started at 33% NaOH should be the same at 250 F (121 C), namely 33% NaOH. The difference then in SCC behavior may be related to the boiling experienced by the 10% solution under 250 F (121 C). The boiling may have reduced the amount of dissolved oxygen in the NaOH. Conversely, however, many more additions of liquid were made to the 10% solution which could well have added oxygen to the caustic, since the liquid was poured through air into the caustic.

The weight loss data converted to metal losses for Type-304 stainless steel are given in Table 2. It should be noted for the same heating rate, 9 F/h (4.5 C/h), weight losses were at least three times higher in the experiment where the starting solution was 33% NaOH. The lower weight losses for all the experiments where the starting solutions were 10% NaOH may be related to the reduced amount of dissolved oxygen as a result of increased boiling during the heat-up period.

2¼ Cr-1 Mo and Type-1008 Carbon Steel

No evidence of caustic cracking was found in the stress corrosion tests for any of these samples at any heating rate. Although these steels should be susceptible to caustic SCC in the region indicated in Figure 1, apparently the incubation periods were not sufficient even at the slowest heating rate. A second possibility is the surface residual tensile stresses were not sufficient to cause SCC. This second possibility was explored in the second series of experiments conducted at GE.

The calculated metal loss data for the ferritic steels are given in Tables 3 and 4. The data in Table 3 indicate the corrosion during the test for 2¼ Cr-1 Mo seems to be independent of the starting concentration or heating rate. This is surprising, as at the slower heating rates, the specimens were exposed to concentrated caustic for longer periods. Longer exposure should have resulted in greater corrosion. Carbon steel (Table 4) shows more predictable behavior. Slower heating rates resulted in more corrosion and the same effect of starting caustic solution on corrosion as noted above for stainless steel was observed.

Inconel-600

In tests with the initial 33% NaOH concentration, heated at 18 F/h (10 C/h), each of the three Inconel-600 specimens stress corrosion cracked from the wrought material on the surface until the crack blunted in the cast structure of the weld bead (Figure 11). When the 18 F/h (10 C/h) tests were performed with an initial 10% NaOH solution, one set of three Inconel-600 specimens was heated from 180 to 240 F (82 to 115 C), then replaced in the same solution by three new Inconel-600 specimens from 240 to 300 F (115 to 149 C), which, in turn, were replaced by a final set of specimens for the temperature range 300 to 360 F (149 to 182 C). One C-ring in the set from 180 to 240 F (82 to 115 C) stress corrosion cracked all through the wrought material (Figure 12), as did a C-ring in the set from 240 to 300 F (115 to 149 C). None of the specimens heated from 300 to 360 F (149 to 182 C) cracked. Metal losses for these tests were low, less than 0.02 mm.

Welded Tab Halves

The C-rings fabricated from tab halves of Type-304 stainless steel welded together cracked on the convex side by each spot weld. Fine cracks were also detected in those

C-rings fabricated from 2¼ Cr-1 Mo welded to Type-304 stainless steel. No stress corrosion cracks were found in the carbon steel to Type-304 stainless steel or the carbon steel to 2¼ Cr-1 Mo steel couples. The weight loss data for the welded couples are given in Table 5.

SECOND SERIES OF TESTS

The samples, metallurgical conditions, applied stresses and stress corrosion results are given in Tables 6 through 11. No stress corrosion occurred on the 2¼ Cr-1 Mo and carbon steel at any applied stress regardless of the heating rate. On the other hand, the stainless steel was quite susceptible to stress cracking. The degree of cracking depended on the applied stress and the heating rate. In runs D001 and D002, at 18 F/h (10 C/h), the stainless steel samples stressed to 75% of yield strength, based on the 300 F (149 C) yield strength, did not show any obvious cracking by examination up to 140X. However, those stressed to 100 and 150% of the yield were cracked. In run D003 also at 18 F/h (10 C/h) the Type-304 stainless steel samples stressed at 90,100, and 150% of yield cracked.

In our experiments, no galvanic effects appear between samples and the more cathodic fixtures in the test facility. Thus, no difference in the stress corrosion behavior of stainless steel was noted in run D003 where the samples were electrically insulated, compared to runs D001 and D002 where there was no attempt to insulate the specimens.

Figures 13a, 13b, and 13c are scanning electron micrographs of a mill-annealed stainless steel sample from run D003 stressed at 100% of the 300 F (149 C) yield strength. The corner cracks are clearly intergranular while surface cracks appear to be mixed mode. Some cracks appear to initiate at pits.

The morphology of the cracking of both solution-annealed and furnace-sensitized Type-304 stainless steel is especially interesting in run D004. The sensitized and solution-annealed samples show the networks of continuous carbides at the grain boundaries as well as transgranular cracks (Figures 14 and 15). The grain boundary carbides indicate that the "solution-annealed" alloy was somewhat sensitized. Non-metallic inclusions appear to be initiation sites for some transgranular cracks.

A comparison of scanning electron micrograph and a metallographic micrograph of a mill-annealed stainless steel sample is given in Figures 16 and 17 for run D005. Crack initiation sites can be seen with the scanning electron micrograph and the very shallow, mostly transgranular cracks are evident with the metallographic section. This sample was stressed at 100% of the yield strength and heated at 36 F/h (20 C/h). At 200% of the yield strength, the cracking depth increases significantly (Figure 18). Run D006, at 9 F/h (5 C/h) heating rate, the slowest heating rate studied, had the most severe cracking. In this run, Type-304 stainless steel samples stressed at 200% of the yield strength completely parted. The highly stressed 2¼ Cr–1 Mo and carbon steel samples were examined after test. Metallographic sections of these highly stressed samples showed no indication of caustic SCC although sustained stresses of 60 ksi on 2¼ Cr–1 Mo and 70 ksi on carbon steel were maintained during run D006.

To evaluate the general corrosion rate of the materials studied, all the gage sections of all specimens tested were measured before and after the test. Because samples stressed over the yield strength have reduced gage sections caused by strain as well as corrosion, their net dimensional changes are expected to be the greatest. For this reason, the only dimensional measurements given in Table 12 are for samples stressed at, or below, the yield strength.

Samples from run D001 were expected to have lower corrosion rates because these samples were uncovered above 320 F (160 C). Because of the formation of general surface oxides which are included in the measurements, a few selected samples were measured destructively by obtaining metallographic cross sections. In the metallographic sections, the oxide can readily be distinguished from the metal. Surface oxides were less than 0.001 in. (0.0254 mm) and thus too thin to affect the measurements significantly.

From Table 12, it appears the corrosion rates for 2¼ Cr-1 Mo and carbon steel are comparable and about a factor of 6 higher than Type-304 stainless steel. The amount of general corrosion increased with decreasing heating rate because the exposure to concentrated caustic occurred over a longer time. Thus, at 18 F/h (10 C/h), 2¼ Cr–1 Mo and carbon steel corroded about 0.0025 in. (0.063 mm) of corrosion on each surface. At 9 F/h (5 C/h) the corrosion was about 0.0035 in. (0.089 mm) for the same materials while at the fastest heating rate 36 F/h (20 C/h) the corrosion on a surface for the ferritic alloys was about 0.001 to 0.0015 in. (0.0254 to 0.0381 mm).

SUMMARY

It has been established by H. S. Isaacs and reported by Indig[3] that 2¼ Cr–1 Mo steel is susceptible to stress corrosion in boiling caustic solutions. It is also well known that carbon steel will crack in the same environment. However, in our tests no cracking of these ferritic alloys occurred. Apparently the amount of time spent in the susceptible temperature regions is too short to initiate cracking. The obvious implication is that with any reasonable heating rate following a sodium-water reaction in the LLTR, the 2¼ Cr–1 Mo will not stress corrosion crack, nor will 1008 carbon steel.

Type-304 stainless steel suffered caustic SCC when stressed greater than or equal to 90%

of the yield strength at heating rates less than 45 F/h (25 C/h). The slower the heating rate and the higher the stress, the greater the extent of caustic SCC. At 45 F/h (25 C/h), no caustic cracking was observed in the tests conducted at LMEC. Apparently stainless steel will crack in caustic solutions over a wide range of oxidizing potential, since cracking was observed in de-aerated and aerated caustic environments. Carbon steel welded to Type-304 stainless steel eliminated caustic SCC at 18 F/h (10 C/h), although cracking occurred in the 2¼ Cr-1 Mo Type-304 stainless steel welded couples.

Inconel-600, when under stresses approaching the yield strength, suffered extensive caustic SCC at 18 F/h (10 C/h) whereas unstressed Inconel-600 seemed unaffected by caustic. The cracking that occurred was intergranular, propagated in the wrought material, and terminated in the weld cast structure.

General corrosion rates of 2¼ Cr-1 Mo, 1008 carbon steel, and Type-304 stainless steel in aqueous caustic during heating were not known before these tests. Surfaces of the ferritic alloys lost from 1 to 3 mils during each test while Type-304 stainless steel corroded at about one-sixth that rate. The amount of general corrosion increased with decreasing heating rate.

ACKNOWLEDGMENTS

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TABLE 1. Alloy Compositions for Series Two Experiments

Material	Heat	Yield Point psi	Tensile Strength psi	% C	% Mn	% P	% S	% Si	% Ni	% Cr	% Mo	% Co	Cu
Type-304	X11224	38,000	85,100	0.06	1.36	0.023	0.028	0.74	8.36	18.69	0.37	0.14	0.27
2¼Cr-1Mo	88541	59,500	83,200	0.12	0.47	0.017	0.025	0.39	0.12	2.25	0.97	–	0.14
A516-70	D1923A4	51,700	80,300	0.23	1.05	0.012	0.023	0.18	–	–	–	–	–

TABLE 2. Type-304 Stainless Steel Corrosion Rate Based on Weight Loss Data, C-Rings

Temperature °F (°C)	Heating Rate °F/h (°C/h)	Concentration (% NaOH)	Corrosion Rate μ/Test	Corrosion Rate mils/Test
180-450 (82-232)	9 (5)	33	11	0.4
			18	0.7
			12	0.4
180-360 (82-182)	18 (10)	10	3	0.1
			4	0.2
			3	0.1
180-360 (82-182)	9 (5)	10	4	0.2
			4	0.2
			4	0.2
180-360 (82-182)	4.5 (2.5)	10	9	0.4
			7	0.3

TABLE 3. 2¼ Cr-1 Mo Steel Corrosion Rate Based on Weight Loss Data, C-Rings

Temperature °F (°C)	Heating Rate °F/h (°C/h)	Concentration (% NaOH)	Corrosion Rate μ/Test	Corrosion Rate mils/Test
180-450 (82-232)	9 (5)	33	30	1.2
			33	1.3
			33	1.3
180-360 (82-182)	18 (10)	10	36	1.4
			30	1.2
			43	1.7
180-360 (82-182)	9 (5)	10	33	1.3
			33	1.3
			30	1.2
180-360 (82-182)	4.5 (2.5)	10	33	1.3
			36	1.4
			33	1.3

TABLE 4. Type-1008 Carbon Steel Corrosion Rate Based on Weight Loss Data, C-Rings

Temperature °F (°C)	Heating Rate °F/h (°C/h)	Concentration (% NaOH)	Corrosion Rate μ/Test	mils/Test
180-450	9	33	25	1.0
(82-232)	(5)		140	5.6
			30	1.2
180-360	18	10	13	0.5
(82-182)	(10)		16	0.6
			16	0.6
180-360	9	10	20	0.8
(82-182)	(5)		20	0.8
			18	0.7
180-360	4.5	10	35	1.4
(82-182)	(2.5)		28	1.1
			30	1.2

TABLE 5. Weight Losses for Welded Tab Halves

Welded Couple	Weight Losses (mg/cm^2)
Type-304/Type-304	9 ± 2
Type-304/2¼ Cr-1 Mo	34 ± 3[a]
Type-304/Carbon Steel	35 ± 5[a]
2¼ Cr-1 Mo/Carbon Steel	24 ± 2

[a]Calculated on the assumption that corrosion for Type-304 stainless steel was negligible.

TABLE 6. Run D001 (18 F/h or 10 C/h) Materials, Stresses, and Observations

Alloy	Metallurgical Condition	Stress (ksi)	Observation[a]
2¼ Cr-1 Mo	Normalized and Tempered	35	No SCC[b]
2¼ Cr-1 Mo	Normalized and Tempered	52.5	No SCC
Type-304 Stainless Steel	Mill Annealed	23.3	No SCC
Type-304 Stainless Steel	Mill Annealed	31	Slight Cracks, SCC
Type-304 Stainless Steel	Mill Annealed	46	Deep Cracks, SCC
Carbon Steel[c]	As-Received	34.5	No SCC
Carbon Steel[c]	As-Received	46	No SCC
Carbon Steel[c]	As-Received	69	No SCC

[a]Initial sample examinations at 140X; [b]Stress corrosion cracking; [c]ASTM-A516 Grade 70.

TABLE 7. Run D002 (18 F/h or 10 C/h) Materials, Stresses, and Observations

Alloy	Metallurgical Condition	Stress (ksi)	Observation[a]
2¼ Cr-1 Mo	*Normalized and Tempered*	35	No SCC[b]
2¼ Cr-1 Mo	Normalized and Tempered	52.5	No SCC
Type-304 Stainless Steel	Mill Annealed	23.3	No SCC
Type-304 Stainless Steel	Mill Annealed	31	Edge Cracks, SCC
Type-304 Stainless Steel	Mill Annealed	46	SCC over entire gage length
Carbon Steel[c]	As-Received	34.5	No SCC
Carbon Steel[c]	As-Received	46	No SCC
Carbon Steel[c]	As-Received	69	No SCC

[a]Initial sample examinations at 140X; [b]Stress corrosion cracking; [c]ASTM-A516 Grade 70.

TABLE 8. Run D003 (18 F/h or 10 C/h) Materials, Stresses, and Observations

Alloy	Metallurgical Condition	Stress (ksi)	Observation[a]
2¼ Cr-1 Mo	Annealed	30	No SCC[b]
2¼ Cr-1 Mo	Annealed	40	No SCC
2¼ Cr-1 Mo	Annealed	60	No SCC
Type-304 Stainless Steel	Mill Annealed	27.9	Minor Edge SCC
Type-304 Stainless Steel	Mill Annealed	31	Minor Edge SCC
Type-304 Stainless Steel	Mill Annealed	46.5	Edge SCC
Carbon Steel[c]	As-Received	51.7	No SCC
Carbon Steel[c]	As-Received	70	No SCC

[a]Initial sample examination at 150X; [b]Stress corrosion cracking;
[c]ASTM-A516 Grade 70; [d]Appears to be some grain dropping.

TABLE 9. Run D004 (36 F/h or 20 C/h) Materials, Stresses, and Observations

Alloy	Metallurgical Condition	Stress (ksi)	Observation[a]
2¼ Cr-1 Mo	Annealed	30	No SCC[b]
2¼ Cr-1 Mo	Annealed	40	No SCC
2¼ Cr-1 Mo	Annealed	60	No SCC
Type-304 Stainless Steel	Solution Annealed	27.9	No SCC
Type-304 Stainless Steel	Solution Annealed	31	Minor Edge SCC[d]
Type-304 Stainless Steel	Solution Annealed	46.5	Minor SCC
Type-304 Stainless Steel	Furnace Sensitized	46.5	Edge SCC
Carbon Steel[c]	As-Received	69	No SCC

[a]Initial sample examination at 150X; [b]Stress corrosion cracking;
[c]ASTM-A516 Grade 70; [d]Appears to be some grain dropping.

TABLE 10. Run D005 (36 F/h or 20 C/h), Materials, Stresses, and Observations

Alloy	Metallurgical Condition	Stress (ksi)	Observation[a]
2¼ Cr-1 Mo	Annealed	30	No SCC
2¼ Cr-1 Mo	Annealed	40	No SCC
2¼ Cr-1 Mo	Annealed	60	No SCC
Type-304 Stainless Steel	As-Received	27.9	Minor SCC
Type-304 Stainless Steel	As-Received	31	SCC at edges
Type-304 Stainless Steel	As-Received	62	SCC
Type-304 Stainless Steel	Furnace Sensitized	62	SCC
Carbon Steel	As-Received	70	SCC

[a]Initial sample examination at 140X.

TABLE 11. Run D006 (9 F/h or 5 C/h), Materials, Stresses, and Observations

Alloy	Metallurgical Condition	Stress (ksi)	Observation[a]
2¼ Cr-1Mo	Annealed	30	No SCC
2¼ Cr-1 Mo	Annealed	40	No SCC
2¼ Cr-1 Mo	Annealed	60	No SCC
Type-304 Stainless Steel	As-Received	27.9	Minor SCC
Type-304 Stainless Steel	As-Received	31	SCC
Type-304 Stainless Steel	As-Received	62	Fractured
Type-304 Stainless Steel	Furnace Sensitized	62	Fractured
Carbon Steel	As-Received	70	No SCC

[a]Initial sample examination at 140X.

TABLE 12. Dimensions of Sample Gage Sections Before and After Series 2 Tests – Samples Stressed at or Below Yield Strength

Run	Sample	Alloy	Cross Sections (in.) Before Test	After Test
D001	1048	2¼ Cr-1 Mo	0.120 x 0.097	0.116 x 0.094
	1049	2¼ Cr-1 Mo	0.120 x 0.098	0.116 x 0.093
	1314	Carbon Steel	0.122 x 0.095	0.122 x 0.095
	1315	Carbon Steel	0.122 x 0.095	0.120 x 0.093
	15	Type-304 Stainless Steel	0.123 x 0.098	0.123 x 0.098
	16	Type-304 Stainless Steel	0.123 x 0.099	0.124 x 0.099
D002	1050	2¼ Cr-1 Mo	0.121 x 0.097	0.115 x 0.091
	1051	2¼ Cr-1 Mo	0.120 x 0.098	0.113 x 0.091
	1316	Carbon Steel	0.123 x 0.096	0.118 x 0.091
	11	Type-304 Stainless Steel	0.121 x 0.098	0.119 x 0.098
	12	Type-304 Stainless Steel	0.123 x 0.098	0.121 x 0.097
D003	1045	2¼ Cr-1 Mo	0.119 x 0.098	0.115 x 0.092
	1046	2¼ Cr-1 Mo	0.119 x 0.098	0.115 x 0.092
	1320	Carbon Steel	0.122 x 0.096	0.122 x 0.095
	8	Type-304 Stainless Steel	0.122 x 0.098	0.121 x 0.098
	9	Type-304 Stainless Steel	0.120 x 0.097	0.118 x 0.097
D004	1055	2¼ Cr-1 Mo	0.119 x 0.097	0.116 x 0.092
	32	Type-304 Stainless Steel	0.121 x 0.097	0.119 x 0.098
	33	Type-304 Stainless Steel	0.123 x 0.096	0.120 x 0.095
D005	1058	2¼ Cr-1 Mo	0.120 x 0.097	0.119 x 0.096
	1340	Type-340 Stainless Steel	0.123 x 0.095	0.123 x 0.096
	1341	Type-340 Stainless Steel	0.124 x 0.095	0.122 x 0.095
D006	1062	2¼ Cr-1 Mo	0.120 x 0.098	0.114 x 0.091
	1343	Type-304 Stainless Steel	0.124 x 0.096	0.123 x 0.095
	1344	Type-304 Stainless Steel	0.124 x 0.097	0.122 x 0.096

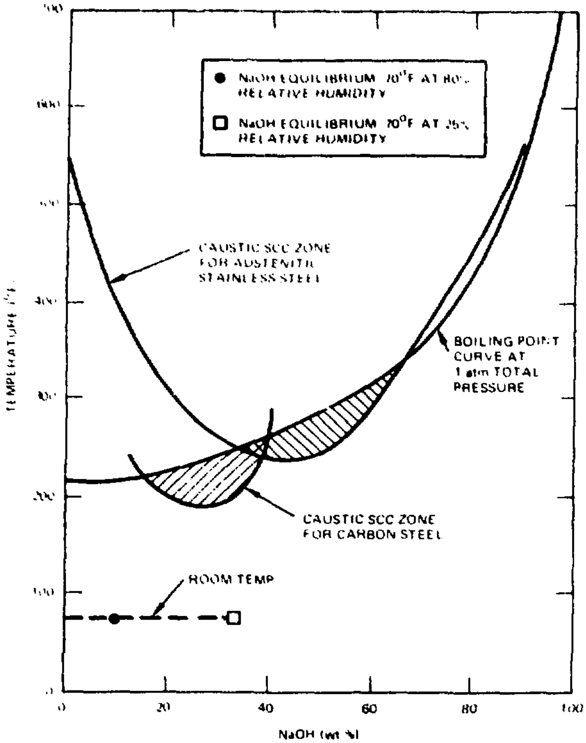


Figure 1: Boundaries of Caustic Stress Corrosion Cracking Zones and Boiling Point Curve

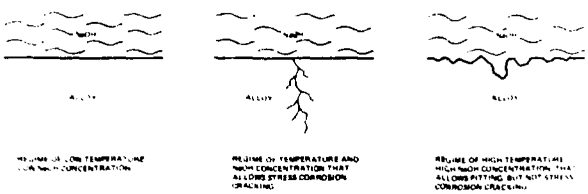


Figure 2: Interactions of Caustic Solutions and Metals

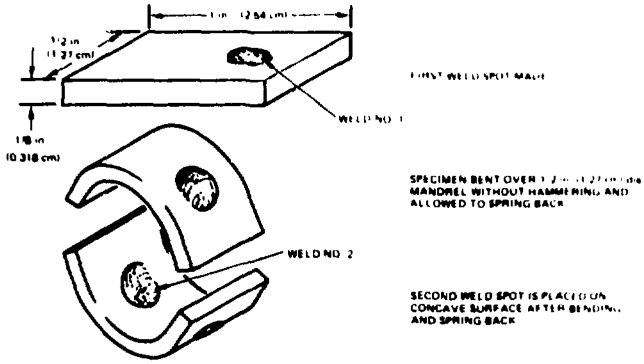


Figure 3: C-Ring Specimen with Spot Weld

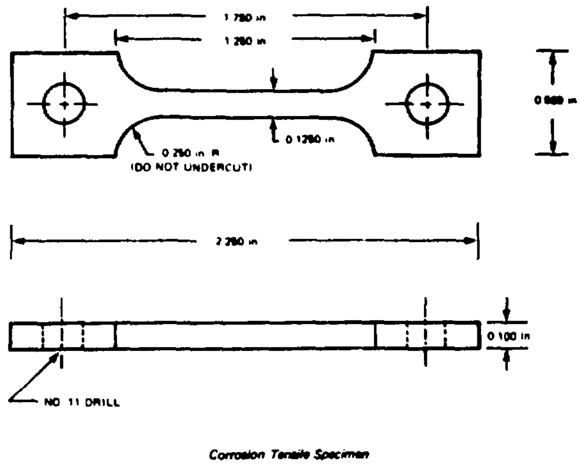
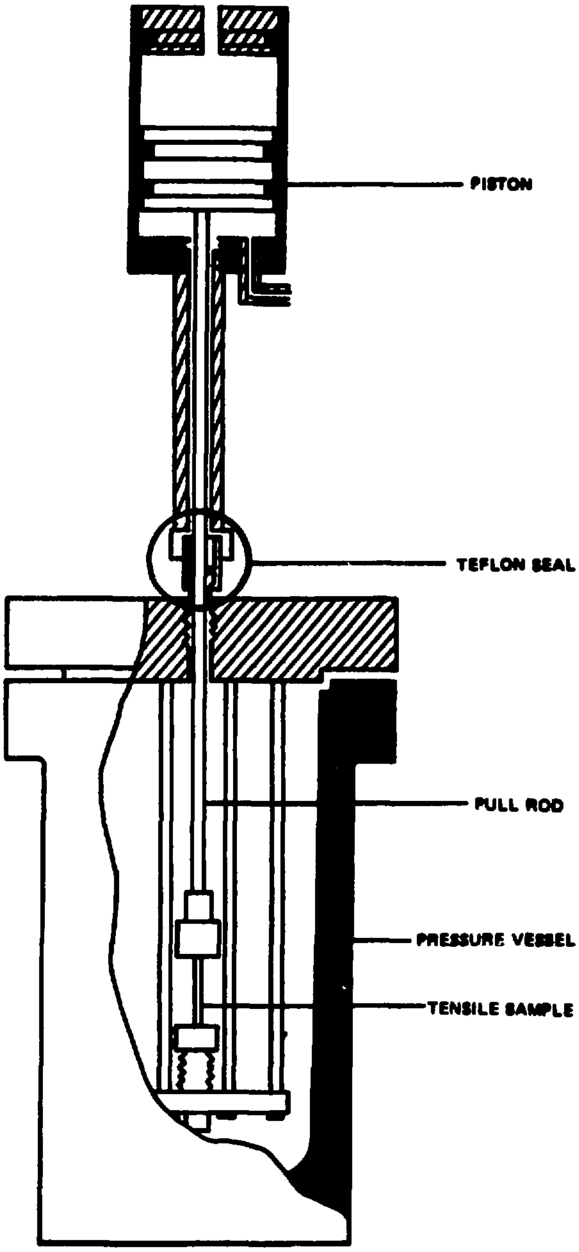


Figure 4: Dog-Bone Tensile SCC Sample



Schematic Drawing of the Test Apparatus

Figure 5: Schematic Drawing of the Test Apparatus for Stressing Tensile Samples

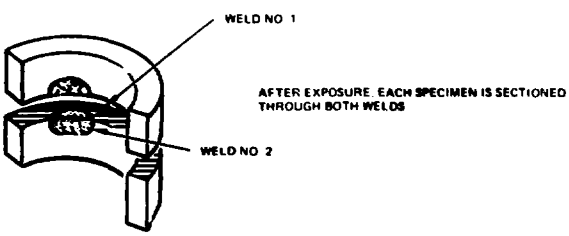
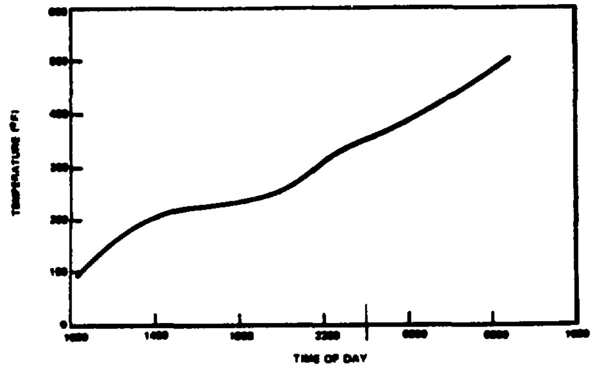
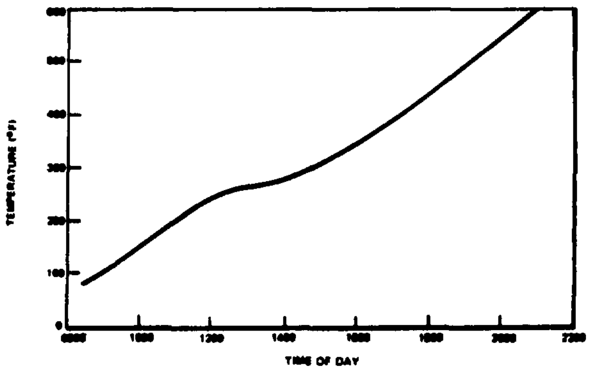


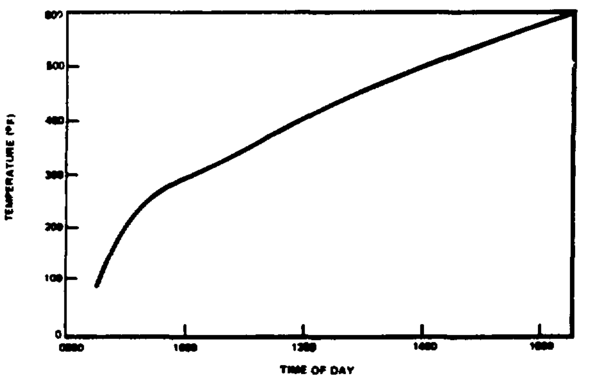
Figure 6: C-Ring Sectioning Method



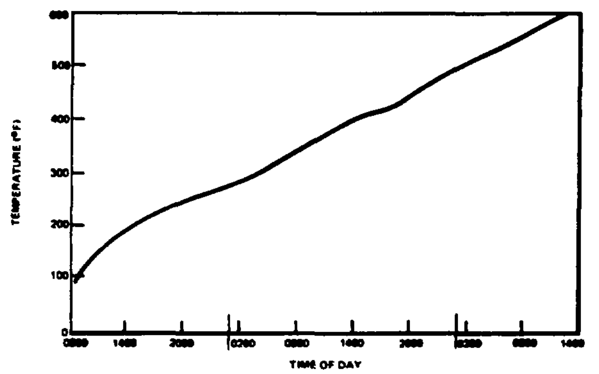
Time – Temperature Curve (Run D001)



Time – Temperature Curve (Run D004)



Time – Temperature Curve (Run D005)



Time – Temperature Curve (Run D006)

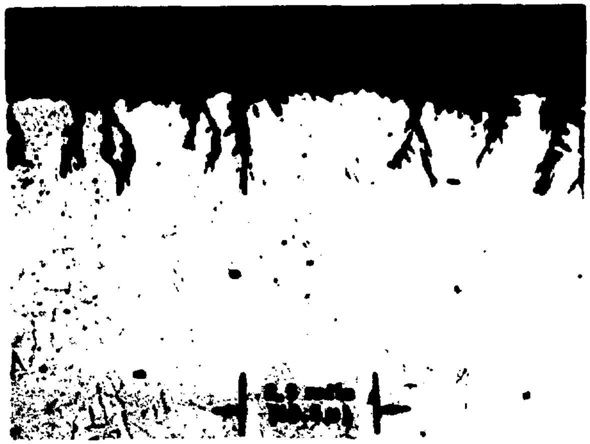
Figure 7a: Time-Temperature Curve (Run D001)
7b: Time-Temperature Curve (Run D004)
7c: Time-Temperature Curve (Run D005)
7d: Time-Temperature Curve (Run D006)

a.

b.

Figure 8: Type-304 Stainless Steel Heated at 18 F/h (10 C/h) to 450 F (232 C) in Caustic with an Initial Concentration of 33% NaOH - Under Residual Tensile Stresses

a.

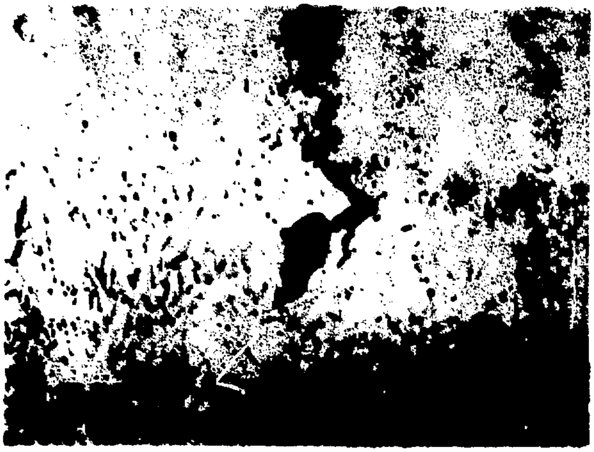


b.

Figure 9: Type-304 Stainless Steel Heated 4.5 F/h (2.5 C/h) to 450 F (232 C) in Caustic with an Initial Concentration of 33% NaOH - Under Residual Tensile Stresses

Figure 10: Type-304 Stainless Steel Heated 18 F/h (10 C/h) to 360 F (182 C) in Caustic with an Initial Concentration of 10% NaOH - Under Residual Tensile Stresses

a.



b.

Figure 11: Inconel-600 Heated 18 F/h (10 C/h) to 450 F (232 C) in Caustic with an Initial Concentration of 33% NaOH - Under Residual Tensile Stresses

Figure 12: Inconel-600 Heated 18 F/h (10 C/h) to 240 F (115 C) in Caustic with an Initial Concentration of 10% NaOH - Under Residual Tensile Stresses

Figure 13a:, 13b:, 13c: Three SEM Views of Mill-Annealed Type-304 Stainless Steel, Run D003, Stressed in Tension to 100% of the 300 F (150 C) σy

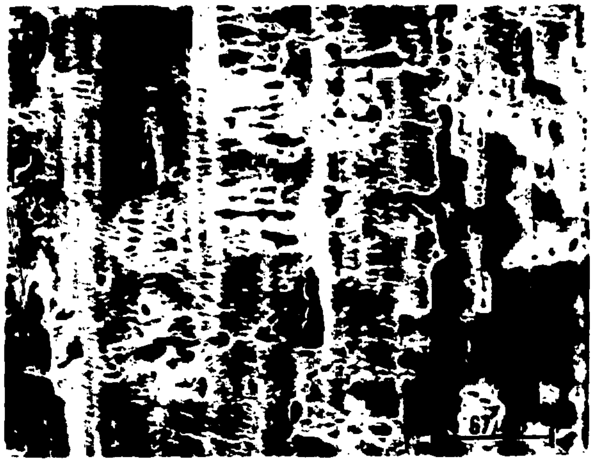


Figure 14: SEM of Solution Annealed Type-304 Stainless Steel, Run D004, Stressed in Tension to 150% of the 300 F (150 C) σy

Figure 16: SEM of Mill-Annealed Type-304 Stainless Steel, Run D005, Stressed in Tension to 100% of the 300 F (150 C) σy

Figure 15: SEM of Furnace Sensitized Type-304 Stainless Steel, Run D004, Stressed in Tension to 150% of the 300 F (150 C) σy

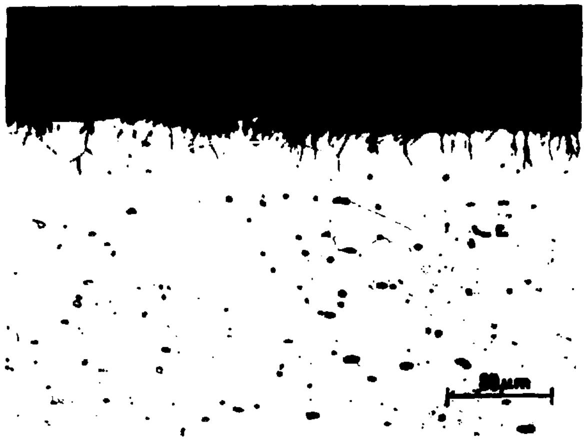


Figure 17: Photomicrograph of Mill-Annealed Type-304 Stainless Steel, Run D005, Stressed in Tension to 100% of the 300 F (150 C) σy

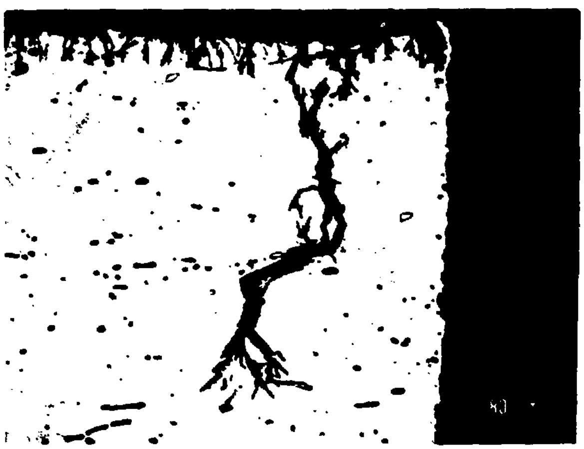


Figure 18: Photomicrograph of Mill-Annealed Type-304 Stainless Steel, Run D005, Stressed in Tension to 200% of the 300 F (150 C) σy