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GENERAL AND LOCALIZED CORROSION OF AUSTENITIC AND BORATED STAINLESS STEELS IN SIMULATED CONCENTRATED GROUND WATERS

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ABSTRACT

Boron containing stainless steels are used in the nuclear industry for applications such as spent fuel storage, control rods and shielding. It was of interest to compare the corrosion resistance of three borated stainless steels with standard austenitic alloy materials such as type 304 and 316 stainless steels. Tests were conducted in three simulated concentrated ground waters at 90°C. Results show that the borated stainless were less resistant to corrosion than the witness austenitic materials. An acidic concentrated ground water was more aggressive than an alkaline concentrated ground water.

Keywords: S30400, S31600, Borated Stainless Steel, Neutrosorb, Neutronit, Concentrated Ground Water, Localized Corrosion, pH

INTRODUCTION

It is proposed to dispose of some 70,000 metric tons of nuclear waste in a repository located in Yucca Mountain, Nevada (1, 2). The safe disposal of radioactive waste requires that the waste be isolated from the environment until radioactive decay has reduced its toxicity to innocuous levels for plants, animals and humans. Engineered barriers are designed to limit the diffusion of the radioactive material to the surrounding geologic formation. The barriers may consist, for example, in a series of dissimilar materials such as zircaloy, glass, stainless steel, nickel alloy container and titanium drip

shield. The spent fuel itself will be located inside the containers in structural inserts or baskets made of corrosion resistant alloys containing neutron absorber elements (neutron poisons) such as boron or gadolinium, which will control criticality (3-5). The use of gadolinium may be preferred because of the lower thermal neutron absorption properties of boron (5).

In previous studies, the corrosion rate of Neutrosorb Plus and Neutronit A976 were compared to the corrosion rate of witness 304L SS using 96 h immersion corrosion tests (3). Testing was carried out in a solution containing 0.01 M formic acid plus 0.01 M sodium formate plus 0.02 M sodium oxalate plus 0.01 M nitric acid plus 0.01 M sodium chloride plus 0.01 M hydrogen peroxide in distilled water at 90°C. The pH of the solution was 4.06. Results showed that the corrosion rate of 304L SS was 10 µm/year, that of Neutronit A976 was 40 µm/year and that of Neutrosorb was 60 µm/year (3).

Waters that contact the engineered barriers (EB) are expected to be in the form of a multi-ionic solution. This solution may form through two different mechanisms; dripping from the drift wall and concentrating on the surface of the EB and/or via the eliquescence of salts (dust) that may accumulate on top of the EB during dry periods. In both cases, the aqueous solutions are expected to be concentrated. The degree of solution concentration would depend on the relative humidity at the emplacement site. The ground waters associated with the Yucca Mountain region have been characterized (2,6) and are consistent of multi-ionic solutions containing carbonate, sulfate, nitrate, chloride, alkalis and alkaline earth ions. Table 1

shows the composition of saturated zone water (from a well designated J-13), which is near the repository site and is carbonate-rich with a pH near neutral. Table 1 also shows the composition of three laboratory-prepared, aqueous, concentrated electrolyte solutions in which testing was performed. These electrolyte solutions range from pH ~3 to 10 and are designated as simulated acidified water (SAW), simulated concentrated water (SCW) and simulated cement modified water (SCMW).

The purpose of the present work was to determine the corrosion behavior of three borated stainless steels and compare the results with the behavior of witness materials such as 304 and 316 austenitic stainless steels. Immersion testing was conducted for more than 5 years in the concentrated simulated ground waters at 90°C using weight (mass) loss coupons.

EXPERIMENTAL PROCEDURE

The corrosion behavior of types 304, 316 and of three borated stainless steels was determined using standard immersion tests according to ASTM G 31 and G 1. The tests were conducted in the Long Term Corrosion Test Facility (LTCTF) at the Lawrence Livermore National Laboratory (LLNL) (7). The test vessels are rather large and each is half-filled with 1000 L of solution. The immersion tests were carried out in three different electrolyte solutions simulating concentrated ground waters. The solutions were called SAW (Simulated Acidified Water), SCW (Simulated Concentrated Water) and SCMW (Simulated Cement Modified Water) (Table 1). Each of the simulated solutions used in this study are concentrated variations of J-13 well water (Table 1). The coupons were immersed in the liquid phase, suspended in the vapor phase above the water line in the vessels and also at the water line itself. The nominal testing temperature was 90°C for the liquid phase. The simulated electrolyte solutions were naturally aerated, i.e., the solutions were not purged and the

ingress of air above the solution was not restricted. All tests were carried out under ambient pressure.

The corrosion test coupons of 304 and 316 SS measured nominally 2 in. × 1 in. × 1/16 in. (approximately 50 mm × 25 mm × 1.5 mm) and the borated stainless steels coupons (SSN and SSE) measured nominally 1 in. × 1 in. × 1/8 in. (25 mm × 25 mm × 3 mm). The SSC coupons were of the same dimensions but 2 mm thick (Table 2 for the acronyms of the steels). The surface areas of the weight loss coupons varied from approximately 14-15 cm² for the borated stainless to 28 cm² for the witness materials. The initial weight (mass) of the coupons varied between 12 to 14 g for 304, 316, SSN and SSE steels and were approximately 9 g for the SSC coupons. The coupons were fabricated from industrial plate stock. The chemical composition and alloy designation of the five studied materials are given in Table 2. The type 304 (S30400) and 316 (S31600) stainless steels were standard wrought materials supplied by Metal Samples. Stainless alloys S30400 and S31600 are used as witness materials. The density of 304 and 316 SS are 7.94 g/cm³ and 7.98 g/cm³, respectively. The heat numbers of these witness austenitic materials are unavailable. Neutrosorb Plus was a material produced by powder metallurgy according to ASTM A 887 by Carpenter Technologies (8). The UNS number of Neutrosorb Plus is S30467 and its heat number is unavailable. S30467 has a composition like S30400 with 1.75% of added boron (Table 2). The density of S30467 is 7.77 g/cm³. Neutronit alloys are wrought products of Bohler Bleche. These two alloys have a composition like 316 SS with 1.38 to 1.62% added boron. The heat numbers and compositions of Neutronit are given in Table 2. The density of the Neutronit stainless was unavailable, it was assumed to be 7.77 g/cm³. The weight loss coupons had a central hole, which was used for affixing them to the racks in the vessels using a bolt and 1/2" diameter PTFE washers or spacers to avoid electrical contact between them. That is, all the coupons had a small annulus around the central hole with an unintended crevice or an area of the metal surface that was shielded from the bulk of the solution in the vessels.

Table 1. Chemical composition of the electrolyte solutions (mg/L)

Ion	SCMW pH 7.75	SCW pH 10.3	SAW pH 2.8	J-13 Well Water pH 7.4
K ⁺	309	3400	3400	5.04
Na ⁺	14	40,900	40,900	45.8
Mg ²⁺	0.3	< 1	1000	2.01
Ca ²⁺	197.5	< 1	1000	13
F ⁻	1.3	1400	0	2.18
Cl ⁻	4.3	6700	24,250	7.14
NO ₃ ⁻	5.2	6400	23,000	8.78
SO ₄ ²⁻	864.7	16,700	38,600	18.4
HCO ₃ ⁻	6.4	70,000	0	128.9
SiO ₂ (aq)	22.7 (Si)	~40	~40	61.1

Table 2. Chemical composition of the studied stainless steel alloys (wt%)

Element	304 SS (S30400)	316 SS (S31600)	Neutrosorb Plus (S30467) Type 304B7	Neutronit A978 Type 316B7 (Heat E084295)	Neutronit A978 Type 316B7 (Heat N156129)
Name	304	316	SSC	SSE	SSN
Fe	~70.0	~68	~64	~64	~63
Cr	18.98	16.55	19.97	18.18	19.16
Ni	8.02	10.70	12.49	12.07	12.74
Mo	0.14	2.13	<0.005	2.11	2.22
B	---	---	1.57 (A)	1.00 (B)	1.17 (C)
C	0.070	0.034	0.034	0.056	0.039
Mn	1.96	1.84	1.70	1.43	0.97
Cu	0.35	0.21	0.02	0.11	0.10
Si	0.48	0.57	0.60	0.72	0.38

The nominal values are: (A) 1.75%, (B) 1.38% and (C) 1.62%.

The exposure time for the stainless steel specimens was over 5 years. The actual testing time for each vessel is shown in Table 3 along with each specimen label, vessel number and weight loss (or gain) during the testing period. Each specimen is designated with 3 letters or numbers (Table 2) followed by two characteristic sequential numbers. The results in Table 3 and further analysis are for dry specimens as removed from the vessels, that is, the specimens were not cleaned to remove corrosion products or salt deposits.

After more than five-year exposure to each solution at 90°C, the specimens were removed from their respective test vessels, rinsed in DI water and dried in air at ambient temperature. In all of the tested conditions, the coupons were covered with deposits, which formed by precipitation of salts from the environment. In some environments, the coupons also had corrosion products. In the analyses given in this report, the coupons were not cleaned. That is, actual corrosion rates are not calculated. Relative corrosion rates may be calculated using Equation 1:

$$CR(nm/yr) = \frac{8.76 \times 10^{10} \Delta W}{\rho A t} \quad (1)$$

where 8.76×10^{10} is the proportionality constant, ΔW is the mass loss in grams after 5+ years, ρ is the density of each of the stainless alloys in g/cm^3 , A is the exposed surface area of each coupon (cm^2) and t is the exposure time (hours).

RESULTS AND DISCUSSION

Table 3 shows the weight loss (gain) of the tested stainless coupons. A total of 135 coupons were studied. Most of the

coupons exposed to the vapor phase in each of the three vessels experienced weight gain due to the formation of deposits or corrosion products that cannot be fully washed out due to the restricted amount of condensed water in the vapor phase. The liquid phase would provide a constant supply of corroding solution and at the same time offer a vehicle to transport away the corrosion products. Both of these mechanisms are limited in the vapor phase. The coupons exposed to the water line had a distinctive behavior depending on the testing vessel. In the SAW vessel, most of the coupons suffered weight loss. The largest weight loss corresponded to the SSC coupons followed by the SSE/SSN coupons, then 304 SS and the lowest weight loss was for the 316 SS coupons (Table 3). In the SCW vessel, the coupons exposed to the water line also suffered weight loss, although a small amount compared to the SAW vessel. All the coupons exposed to the water line in the SCMW vessel experienced weight gain, mainly due to the heavy precipitation of a white/brown salt at the splash line (wet/dry) (Table 4). This salt is probably $CaSO_4$, but this needs to be confirmed.

Most of the coupons exposed to the liquid phase suffered weight loss due to corrosion. SAW was the most aggressive solution for all the materials and the borated stainless suffered the largest amount of weight loss. The second most corrosive solution was SCMW, which especially corroded the SSE/SSN borated stainless. The least corrosive solution was SCW, which caused little corrosion in the witness materials (SS 304 and 316). The least resistant material in SCW seemed to be the powder metallurgy alloy SSC.

Figures 1 and 2 show the weight loss (gain) for the coupons tested in SAW and SCMW liquid, respectively. Assuming that the same amount of deposits forms on both the witness alloys and the borated stainless, Table 3 and Figure 1

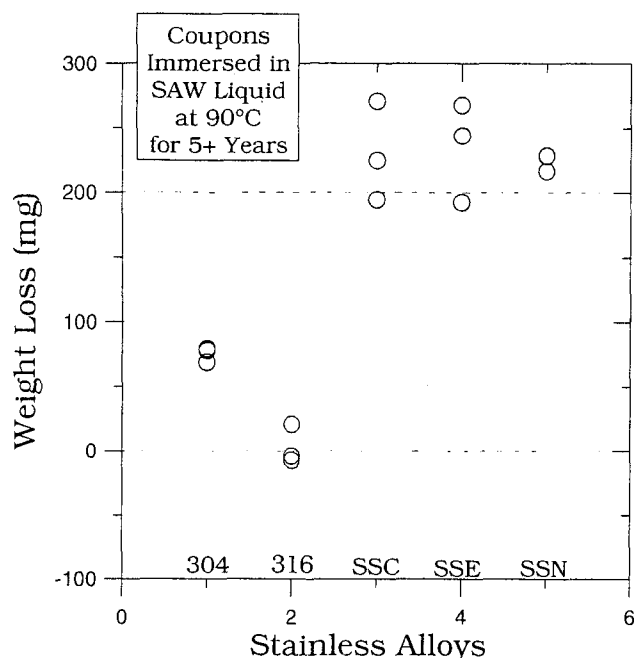


Figure 1. Weight Loss (Gain) for Coupons tested in SAW Liquid for over 5 years.

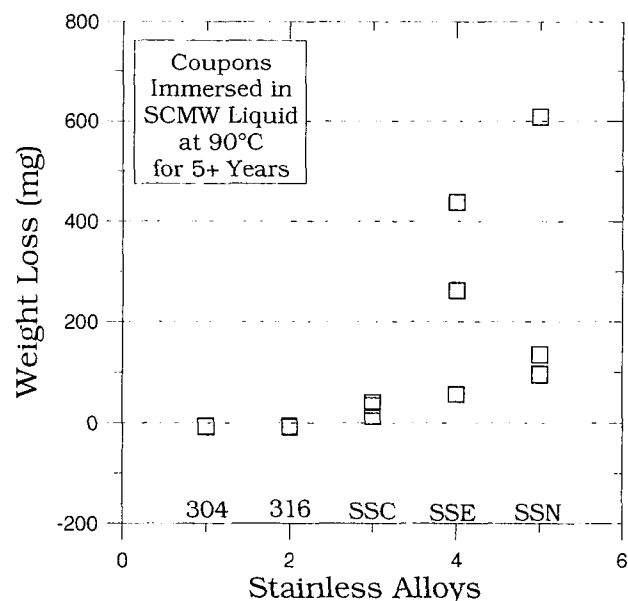


Figure 2. Weight Loss (Gain) for Coupons tested in SCMW Liquid for over 5 years.

show that the borated stainless, in liquid SAW, lost 3 to 10 times more mass than the witness materials. This ratio was even higher for SSE/SSN materials exposed to the liquid SCMW (Table 3 and Figure 2). Using equation 1, the approximate corrosion rates for the borated stainless steels (without cleaning) in liquid SAW is $3.5 \mu\text{m}/\text{year}$ while the approximate corrosion rate for 304 SS is $0.6 \mu\text{m}/\text{year}$. The calculation of these corrosion rates are given for comparative purposes only since most of the corrosion degradation was caused by localized attack in the annulus, that is, it is not justifiable to distribute the weight loss over the entire surface of the coupon to calculate a uniform penetration rate.

MODE OF ATTACK IN CORRODED COUPONS

Table 4 shows a description of the corroded coupons under an optical stereomicroscope. In general, results show that the borated stainless were less resistant to corrosion than the witness materials. For example, in liquid SAW, all five tested materials suffered localized corrosion in the annulus region, but the attack was more extended in the borated materials. On the other hand, in liquid SCMW, the witness materials did not suffer localized corrosion while all the borated stainless had localized corrosion (Table 4). The least aggressive solution was liquid SCW since it only irregularly induced little localized corrosion in the borated stainless and none in the witness materials.

The localized corrosion resistance of stainless steels depends mainly on three factors: (1) Redox potential, (2) Temperature and (3) Chloride concentration and pH. It can be assumed that the first two influencing factors are the same for the three vessels (SAW, SCW and SCMW). It may be obvious that SAW was the most aggressive electrolyte since it had the largest amount of chloride and the lowest pH (2.8) (Table 1). Even though SCW had more chloride concentration than SCMW, the former was probably less aggressive because its pH was higher (10.3)

Figures 3-5 show the corrosion of coupons of 316, SSC and SSN materials in liquid SAW. All these materials suffered localized corrosion in the annulus region (shown). The corrosion products (oxides) inside the corrosion pits in Figure 3 are rich in Mo and Cr. In the borated stainless, the corrosion action left behind particles of Fe and Cr borides. This was also evident in the corrosion of the boldly exposed surface of the borated stainless. For example, Figure 6 shows the boldly exposed surface of an SSC coupon corroded in liquid SCW solution. Corrosion around the iron and chromium boride particles is observed. Some of the boride particles seemed to be partially attacked as well (Figure 6).

CONCLUSIONS

- The largest weight loss was in the liquid phase where corrosion products were washed away. Most of the coupons exposed to the vapor phase experienced weight gain.
- The highest corrosion rate for all materials was in the acidic liquid SAW solution.
- The lowest corrosion damage for all materials was in the alkaline SCW solution.
- All materials suffered localized corrosion in SAW solution.
- Only the borated stainless suffered localized corrosion in the SCW and SCMW solutions.
- The borated stainless in general suffered weight losses 3 to 10 times higher than the witness materials (304 and 316 SS).
- Since weight loss was mostly caused by localized corrosion in the annulus, uniform penetration rates calculations are inappropriate.

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Table 3. Tested Stainless Steel Coupons and Weight Loss (or Gain).

	SAW, 90°C	Weight Loss (Gain) in mg	SCW, 90°C	Weight Loss (Gain) in mg	SCMW, 90°C	Weight Loss (Gain) in mg
Vessel	26	26	28	28	18 (32)	18 (32)
Vapor						
	304-11	(28.2)	304-20	(5)	304-82	(0.8)
	304-12	64.1	304-41	(6)	304-83	(0.6)
	304-13	(37.4)	304-42	(1.5)	304-84	(1.0)
	316-03	(0.6)	316-53	(4.4)	316-92	(0.7)
	316-04	(0.9)	316-54	(3.3)	316-93	(0.8)
	316-05	(0.5)	316-55	(1.9)	316-94	(0.9)
	SSC-01	37.1	SSC-10	(1.0)	SSC-21	(0.4)
	SSC-02	(29.5)	SSC-12	(1.7)	SSC-22	(0.3)
	SSC-03	(36.8)	SSC-13	(1.7)	SSC-23	0.1
	SSE-01	(49)	SSE-12	(1.5)	SSE-22	(0.5)
	SSE-02	18.5	SSE-13	(4.1)	SSE-23	(0.3)
	SSE-03	(79.2)	SSE-15	(3.4)	SSE-24	(2.4)
	SSN-02	(29.7)	SSN-11	0.6	SSN-21	1.1
	SSN-03	(42.1)	SSN-12	(0.5)	SSN-23	(0.2)
	SSN-04	(21.3)	SSN-13	(1.9)	SSN-24	0.1
Water Line						
	304-17	49.5	304-46	1.2	304-88	(135.3)
	304-18	47.0	304-47	0.7	304-89	(148.3)
	304-19	62.7	304-81	1.5	304-90	(142.8)
	316-09	17.3	316-59	1.7	316-98	(132.0)
	316-51	36.4	316-60	1.6	316-99	(158.0)
	316-52	32.3	316-91	2.0	316-100	(149.3)
	SSC-07	154.7	SSC-18	5.7	SSC-27	(163.2)
	SSC-08	130.2	SSC-19	5.1	SSC-28	(77.1)
	SSC-09	136.0	SSC-20	5.2	SSC-30	(154.4)
	SSE-09	61.1	SSE-19	5.7	SSE-28	(88.7)
	SSE-10	112.3	SSE-20	5.7	SSE-29	(163.5)
	SSE-11	129.7	SSE-21	5.0	SSE-30	(35.1)
	SSN-08	69.6	SSN-18	0.7	SSN-28	(139.9)
	SSN-09	133.6	SSN-19	2.8	SSN-29	(88.0)
	SSN-10	108.1	SSN-20	2.1	SSN-30	(137.6)

Table 3. Tested Stainless Steel Coupons and Weight Loss (or Gain) (Continued).

	SAW, 90°C	Weight Loss (Gain) in mg	SCW, 90°C	Weight Loss (Gain) in mg	SCMW, 90°C	Weight Loss (Gain) in mg
Vessel	26	26	28	28	18 (32)	18 (32)
Liquid						
	304-14	78.1	304-43	0.5	304-85	(6.7)
	304-15	79.5	304-44	0.7	304-86	(6.2)
	304-16	68.9	304-45	1.6	304-87	(6.4)
	316-06	(3.7)	316-56	0.7	316-95	(7.2)
	316-07	21.0	316-57	0.6	316-96	(6.2)
	316-08	(7.0)	316-58	1.9	316-97	(7.5)
	SSC-04	270.8	SSC-14	44.0	SSC-24	42.2
	SSC-05	224.6	SSC-15	312.7	SSC-25	13.5
	SSC-06	194.4	SSC-17	8.6	SSC-26	35.5
	SSE-05	244.1	SSE-16	8.8	SSE-25	262.1
	SSE-06	267.8	SSE-17	8.3	SSE-26	57.6
	SSE-08	192.4	SSE-18	9.4	SSE-27	437.9
	SSN-05	228.3	SSN-15	3.4	SSN-25	96.4
	SSN-06	216.4	SSN-16	3.3	SSN-26	608.1
	SSN-07	228.5	SSN-17	3.4	SSN-27	134.7
Date in	03Mar1998		03Mar1998		03Mar1998	
Date out	06Jan2004		20Aug2003 or 06Jan2004		06Jan2004	
Exp. Time, days (h)	2,134 (51,216)		1,994 (47,856) for 304 and 316 SS; 2,134 (51,216) for the Borated Alloys		2,134 (51,216)	

Table 4. Low Magnification (20X) Optical Observation of the Corroded Coupons.

	SAW, 90°C	SCW, 90°C	SCMW, 90°C
Vessel	26	28	18 (32)
Vapor			
304 SS	Spotted or stained, multicolor (brown, green, yellow, purple). No apparent general or localized corrosion	Dark blue/coal with areas of intense rust color. Abundant deposits (or corrosion products). No apparent localized corrosion.	Shiny metallic with a thin veil of brown and yellow deposits. No general or localized corrosion
316 SS	Coupons shiny gray with yellow/brown spots. Other spots red and green. No apparent general corrosion in the boldly exposed surface.	Dark gray and rusty in patches. Some areas of plum color. Accumulation of concentric deposits in annulus	Shiny gray with spots of yellow/rust color. Small amount of deposits. No apparent general or localized corrosion
Neutrosorb	Coupons covered by large amount of black, glass like, deposits. Also red, yellow and blue spots	Brown/yellow in patches with light shiny gray in between. Little crevice corrosion in annulus	Shiny light brown, spotted in brown. Crevice corrosion in annular section, cracks from hole
Neutronit E	Dark gray/greenish. Shiny black glassy like deposits. Crevice corrosion in annulus	Shiny gray in spots. Rust like deposits. Some apparent general corrosion. Negligible crevice corrosion	Gray/stained, with some wet-like spots. No obvious general or localized corrosion.
Neutronit N	Light dull gray. Scattered glass like black deposits. Also brown and yellow deposits. Crevice corrosion in annulus	Spotted dark gray, brown (rusty). Difficult to assess general corrosion due to deposits. Little or no crevice corrosion	Dark gray spotted with yellow and purple areas. Little or no crevice corrosion
Water Line			
304 SS	Pitting corrosion in annulus. One large bump with accumulated corrosion products (oxides) in each coupon. Stains in vapor phase, clear shiny in liquid phase.	Light golden in vapor phase. Clear at water line. Dark gray/bluish in liquid. No apparent general or localized corrosion	Thick crust of white/brown deposits above water line. Fewer deposits in liquid phase. No apparent general or localized corrosion
316 SS	Coupons dull with little deposits. No apparent corrosion in boldly exposed surface. Extensive attack in the annulus region. Each coupon has one large pit with accumulated corrosion products, which perforated the thickness.	Light golden in vapor phase, clear in water line and bright coal/blue in liquid. Concentric deposits in annulus. No apparent corrosion	Dark brown caked with brown and yellow salt like deposits, especially in the wet/dry area. Clear annulus and no apparent localized corrosion
Neutrosorb	The area exposed to the vapor was dark brown with black glassy deposits. The area in the liquid was light gray. Extensive crevice corrosion in the annulus	Shiny golden in vapor phase, shiny dark gray and bluish in liquid. Little crevice corrosion in annulus	Top half caked with brown/white deposits. Little crevice corrosion in annulus and cracks from hole.
Neutronit E	Shiny black/brown with large amount of deposits and corrosion products. Crevice corrosion in annulus. White salt like deposits in entire surface	Dark gray with deposits. General corrosion outside annulus. Little crevice corrosion	White deposits in wet/dry area. Large amounts of black and brown deposits (or corrosion products). Shiny glassy black products. Extensive crevice corrosion in annulus.
Neutronit N	In vapor phase caked black and brown deposits. In liquid phase light dull gray. Sugary crevice corrosion in annulus	Tan in the vapor phase. Even gray in the liquid phase. White deposits around annulus. Not obvious crevice corrosion. Probably general corrosion	Caked with white deposits in wet/dry area. Dark brown/black in the liquid phase. Extensive sugar like crevice corrosion with glassy black corrosion products

Table 4. Continued

	SAW, 90°C	SCW, 90°C	SCMW, 90°C
Vessel	26	28	18 (32)
Liquid			
304 SS	Coupons dull gray. Brown and black deposits over entire surface. One large bump with corrosion products in each coupon. Pitting corrosion in annulus.	Dark gray/bluish, shiny. Little or no deposits. No apparent general or localized corrosion	On label side, brown oily or wet appearance. On the back side, the same appearance of wetness but less rusty. No apparent general or localized corrosion
316 SS	Corrosion attack in the liquid phase is less pronounced than in the water line. Two coupons had corrosion pits in the annulus which perforated the thickness. Brown/white deposits at interface	Dark gray/bluish shiny. White concentric deposits in annulus. No general or localized corrosion	Brown wet with oily appearance. Clear annular section. No apparent general or localized corrosion
Neutrosorb	Light gray coupon. White deposits. Extensive crevice corrosion in annulus. Some pieces of the annulus might have fallen off. Black glass like crystals	Shiny dark gray and blue. Shallow cracks from hole. Crevice corrosion in annulus	Dark brown/rusty with patches of cobalt blue. Extensive pitting and crevice corrosion in annulus. Delamination and cracks at hole
Neutronit E	Light gray. Extensive crevice corrosion in annulus leaving behind a sugar like appearance. Black deposits around the hole	Even dark gray. Little deposits, some general corrosion, little crevice corrosion	Large mounds of crumbling rust especially by the hole. Extensive crevice corrosion in annulus. Shiny black glassy appearance of corrosion products. Wet oily appearance in label side.
Neutronit N	Light dull gray with black and yellow deposits. Extensive sugary crevice corrosion in annulus. Some cracks from hole	Uniform dark gray, little apparent crevice corrosion. General corrosion in boldly exposed surface.	Gray with brown deposits. Extensive crevice corrosion in annulus. Plenty of brown/black corrosion products.

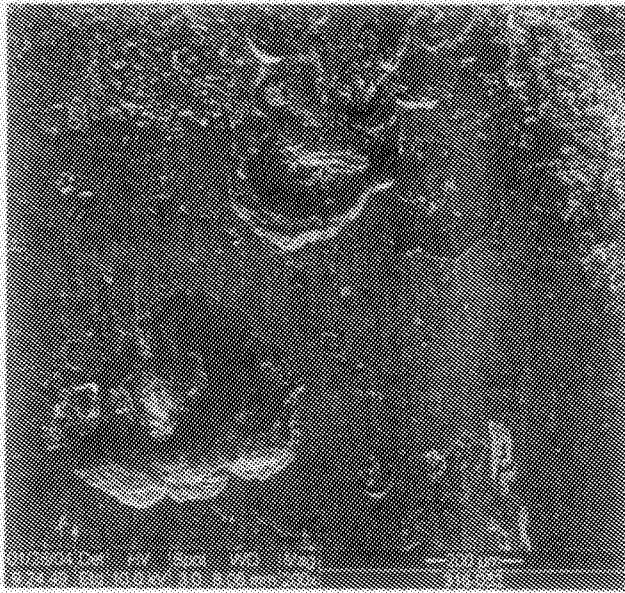


Figure 3: Annulus of 316-52 in SAW, Liquid for 5 + Years

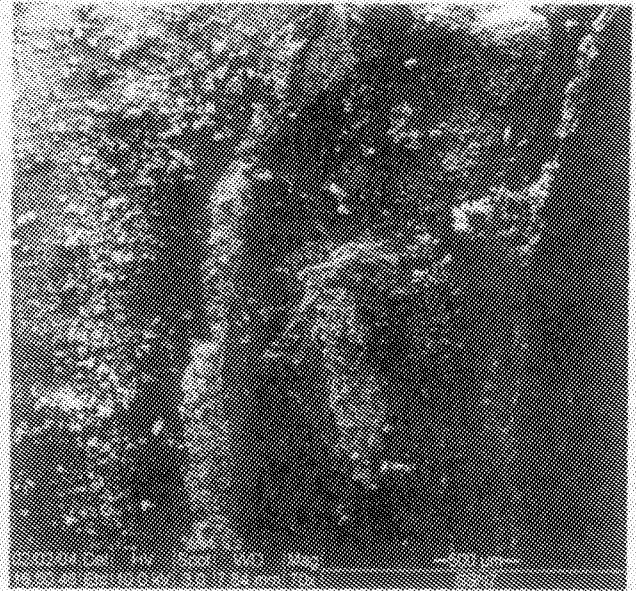


Figure 5: Annulus of SSN-07 in SAW, Liquid for 5 + Years

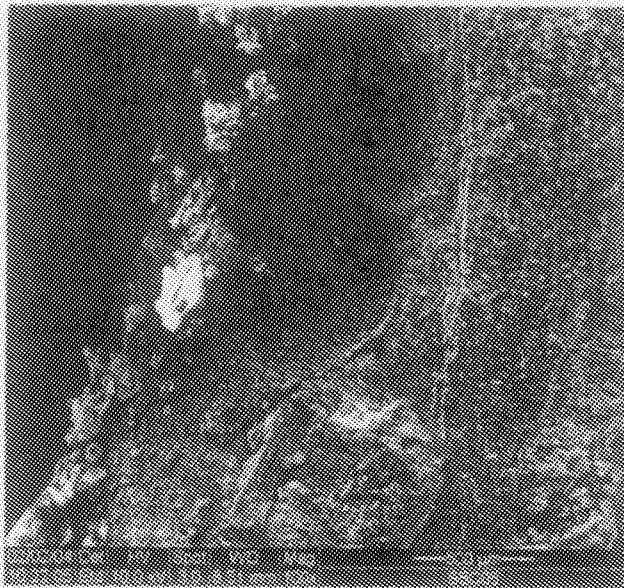


Figure 4: Annulus of SSC-05 in SAW, Liquid for 5 + Years

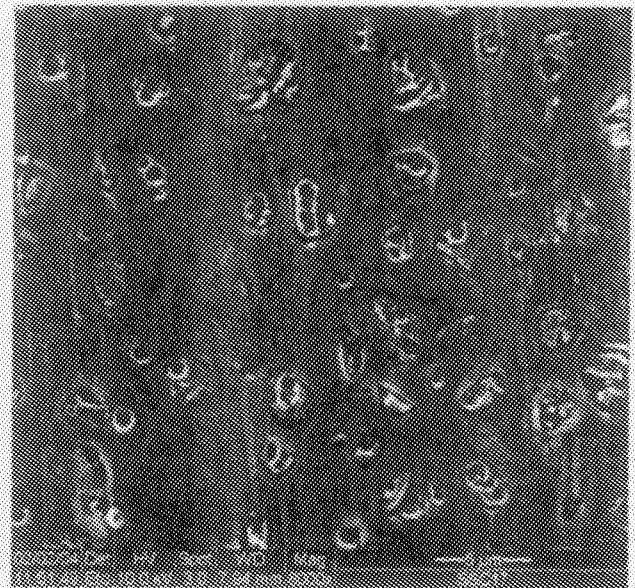


Figure 6: Bold Surface of SSC-17 in SCW, Liquid for 5 + Years