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Stress Corrosion Cracking Behavior of Irradiated Model Austenitic Stainless Steel Alloys*

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Abstract

Slow-strain-rate tensile tests (SSRTs) and posttest fractographic analyses by scanning electron microscopy were conducted on 16 austenitic stainless steel (SS) alloys that were irradiated at 289°C in He. After irradiation to $\approx 0.3 \times 10^{21}$ n·cm^{-2} and $\approx 0.9 \times 10^{21}$ n·cm^{-2} (E > 1 MeV), significant heat-to-heat variations in the degree of intergranular and transgranular stress corrosion cracking (IGSCC and TGSCC) were observed. Following irradiation to a fluence of $\approx 0.3 \times 10^{21}$ n·cm^{-2}, a high-purity laboratory heat of Type 316L SS (Si $\approx$ 0.024 wt.%) exhibited the highest susceptibility to IGSCC. The other 15 alloys exhibited negligible susceptibility to IGSCC at this low fluence. The percentage of TGSCC on the fracture surfaces of SSRT specimens of the 16 alloys at $\approx 0.3 \times 10^{21}$ n·cm^{-2} (E > 1 MeV) could be correlated well with N and Si concentrations; all alloys that contained <0.01 wt.% N and <1.0 wt.% Si were susceptible, whereas all alloys that contained >0.01 wt.% N or >1.0 wt.% Si were relatively resistant. High concentrations of Cr were beneficial. Alloys that contain <15.5 wt.% Cr exhibited greater percentages of TGSCC and IGSCC than those alloys with $\approx$18 wt.% Cr, whereas an alloy that contains >21 wt.% Cr exhibited less susceptibility than the lower-Cr alloys under similar conditions.

Introduction

As nuclear plants age and accumulated neutron fluence increases, austenitic stainless steel (SS) core internal components of boiling and pressurized water reactors (BWRs and PWRs) become susceptible to irradiation-assisted stress corrosion cracking (IASCC). Although most failed components can be repaired or replaced, such repairs are difficult and expensive. Therefore, extensive research has been conducted to develop our understanding of this form of degradation.[1-18]

Irradiation–induced grain–boundary depletion of Cr has been considered by most investigators to be the primary metallurgical process that causes IASCC. Chromium–depleted zones at grain boundaries have been observed, and the effects of electrochemical potential (ECP) on susceptibility to SCC of nonirradiated, thermally sensitized material (where Cr–depletion is widely recognized as the primary factor) and on susceptibility to IASCC of BWR–irradiated solution–annealed material have been reported to be similar.[1-3] However, contrary to expectations based on the strong effect of ECP on SCC associated with the Cr–depletion mechanism, cracking of highly stressed components has been reported in PWRs, which operate at low ECPs, and the susceptibility of PWR–irradiated components to cracking at low ECP has been demonstrated in several hot-cell experiments.[4,5] Other investigators have suggested radiation–induced segregation of impurities such as Si, P, and S as a process that causes IASCC.[4,6,7] The superior resistance to IASCC of a heat of Type 348 SS that contained very low levels of C, Si, P, and S seemed to provide evidence for this.[4] However, later investigations indicated that the resistance of high–purity (HP) heats (low in C, Si, S, and P) of Type 304 SS is no better than that of commercial–purity (CP) Type 304 SSs.[8-14]

Although it has been known for many years that IASCC in general is characterized by strong heat-to-heat variations even among the same grade of SSs, the origin of the variations is not well understood. Therefore, a joint irradiation testing program was initiated to systematically investigate the effects of alloying and impurity elements (Cr, Ni, Si, P, S, Mn, C, and N) on the susceptibility of austenitic SSs to IASCC. In the joint program, many austenitic SSs were irradiated in the Halden reactor, and postirradiation tests and examinations are being conducted at Argonne National Laboratory. This paper describes the results obtained to date from slow-strain-rate-tensile (SSRT) tests on specimens irradiated to $\approx 0.3 \times 10^{21}$ n·cm^{-2} and $\approx 0.9 \times 10^{21}$ n·cm^{-2} (E > 1 MeV). A companion paper describes results from compact-tension (CT) J-R tests.

Experimental Procedure

An optimized test matrix was constructed according to the method of Taguchi.[19,20] Based on the optimized matrix, 8 commercial and 19 laboratory heats of model austenitic SS alloys were procured.[20] High- and commercial-purity (HP and CP) heats of Types 304, 316 and 348 SS were included in the test matrix. The compositions of the 27 model alloys are given in Table 1. Slow-strain-rate-tensile and 1/4T compact-tension specimens were prepared from the alloys. The specimens were irradiated in the Halden reactor at 289°C in He to three fluence levels, 0.3, 0.9, and 2.5×10^{21} n·cm^{-2} (E > 1 MeV), as shown in Table 2. Susceptibility to IASCC was determined by SSRT tests of the irradiated specimens in simulated-BWR water, and posttest fractographic analysis was conducted in a scanning electron microscope to measure the degree of transgranular and intergranular fracture.

Slow-Strain-Rate-Tensile Tests

Slow–strain–rate tensile tests and fractographic analysis by scanning electron microscopy have been completed of the 16 alloys that were irradiated to a fluence of $\approx 0.3 \times 10^{21}$ n·cm^{-2} (E > 1 MeV). Tests have also been conducted on 9 alloys that were irradiated to $\approx 0.9 \times 10^{21}$ n·cm^{-2} (E > 1 MeV). In addition to the irradiated specimens, unirradiated control specimens of some of the alloys have been tested to provide baseline properties.[21] All of the SSRT tests were conducted at 289°C in simulated BWR water that contained ≈8 ppm dissolved oxygen (DO). The conductivity and pH of the water were held at 0.07-0.10 µS·cm^{-1} and 6.3-6.8, respectively. The strain rate was 1.65×10^{-7} s^{-1}. The electrochemical potential (ECP) was measured at regular intervals on the effluent side .

SSRT Testing and Fractographic Analysis of Low-Fluence Specimens

Feedwater chemistry (i.e., DO, ECP, conductivity, and pH) and results from SSRT tests (i.e., 0.2%-offset yield strength, maximum strength, uniform plastic strain, and total plastic strain) are summarized in Tables 3 and 4, respectively, for the "low-fluence" specimens, i.e., the specimens irradiated to $\approx 0.3 \times 10^{21}$ n·cm^{-2} (E > 1 MeV). Also shown in the tables are results of SEM fractographic analysis of the failure mode (i.e., ductile, intergranular, and transgranular fracture surface morphology) of the specimens. In Table 4, the results of SSRT and SEM fractographic analysis [percent intergranular stress corrosion cracking (IGSCC), percent transgranular stress corrosion cracking (TGSCC), and combined percent IGSCC + TGSCC] are correlated with the compositions of the low-fluence specimens.

Significant heat-to-heat variations in susceptibilities to IGSCC and TGSCC were observable even at a fluence of $\approx 0.3 \times 10^{-21}$n cm^{-2}. A HP laboratory heat of Type 316L SS, Heat L22, which contains a very low Si concentration of ≈0.024 wt.%, exhibited a relatively low ductility and the greatest susceptibility to IGSCC (highest percent IGSCC). At this low fluence, susceptibility of all other commercial and laboratory-fabricated alloys to IGSCC was insignificant. The threshold fluence of Heat L22 appears to be lower than the commonly accepted threshold fluence for IASCC, i.e., $\approx 0.5 \times 10^{-21} n\ cm^{-2}$. As shown later, this alloy also exhibited relatively high susceptibility to IGSCC after irradiation to $\approx 0.9 \times 10^{-21} n\ cm^{-2}$.

Table 1. Elemental composition of twenty-seven commercial and laboratory model austenitic stainless steel alloys irradiated in the Halden Reactor.

ANL ID[a]	Source Heat ID	Ni	Si	P	S	Mn	C	N	Cr	O	B	Mo or Nb
C1	DAN-70378	8.12	0.50	0.038	0.002	1.00	0.060	0.060	18.11	-	<0.001	-
L2	BPC-4-111	10.50	0.82	0.080	0.034	1.58	0.074	0.102	17.02	0.0065	<0.001	-
C3	PNL-C-1	8.91	0.46	0.019	0.004	1.81	0.016	0.083	18.55	-	<0.001	-
L4	BPC-4-88	10.20	0.94	0.031	0.010	1.75	0.110	0.002	15.80	-	<0.001	-
L5	BPC-4-104	9.66	0.90	0.113	0.028	0.47	0.006	0.033	21.00	-	<0.001	-
L6	BPC-4-127	10.00	1.90	0.020	0.005	1.13	0.096	0.087	17.10	0.0058	<0.001	-
L7	BPC-4-112	10.60	0.18	0.040	0.038	1.02	0.007	0.111	15.40	0.0274	<0.001	-
L8	BPC-4-91	10.20	0.15	0.093	0.010	1.85	0.041	0.001	18.30	-	<0.001	-
C9	PNL-C-6	8.75	0.39	0.013	0.013	1.72	0.062	0.065	18.48	-	<0.001	-
C10	DAN-23381	8.13	0.55	0.033	0.002	1.00	0.060	0.086	18.19	-	<0.001	-
L11	BPC-4-93	8.15	0.47	0.097	0.009	1.02	0.014	0.004	17.40	-	<0.001	-
C12	DAN-23805	8.23	0.47	0.018	0.002	1.00	0.060	0.070	18.43	-	<0.001	-
L13	BPC-4-96	8.18	1.18	0.027	0.022	0.36	0.026	0.001	17.40	-	<0.001	-
L14	BPC-4-129	7.93	1.49	0.080	0.002	1.76	0.107	0.028	15.00	0.0045	<0.001	-
L15	BPC-4-126	8.00	1.82	0.010	0.013	1.07	0.020	0.085	17.80	0.0110	<0.001	-
C16	PNL-SS-14	12.90	0.38	0.014	0.002	1.66	0.020	0.011	16.92	-	<0.001	-
L17	BPC-4-128	8.00	0.66	0.090	0.009	0.48	0.061	0.078	15.30	0.0092	<0.001	-
L18	BPC-4-98	8.13	0.14	0.016	0.033	1.13	0.080	0.001	18.00	-	<0.001	-
C19	DAN-74827	8.08	0.45	0.031	0.003	0.99	0.060	0.070	18.21	-	<0.001	-
L20	BPC-4-101	8.91	0.017	0.010	0.004	0.41	0.002	0.002	18.10	-	<0.001	-
C21[b]	DAN-12455	10.24	0.51	0.034	0.001	1.19	0.060	0.020	16.28	-	<0.001	Mo 2.08
L22[c]	BPC-4-100	13.30	0.024	0.015	0.004	0.40	0.003	0.001	16.10	-	<0.001	Mo 2.04
L23[d]	BPC-4-114	12.04	0.68	0.030	0.047	0.96	0.043	0.092	17.30	0.0093	<0.001	Nb 1.06
L24[e]	BPC-4-105	12.30	0.03	0.007	0.005	0.48	0.031	0.002	16.90	0.0129	<0.001	Nb 1.72
L25C3	BPC-4-133	8.93	0.92	0.020	0.008	1.54	0.019	0.095	17.20	0.0085	0.010	-
L26C19	BPC-4-131	8.09	0.79	0.004	0.002	0.91	0.070	0.089	17.20	0.0080	<0.001	-
L27C21	BPC-4-132	10.30	0.96	0.040	0.002	0.97	0.057	0.019	15.30	0.0058	0.030	Mo 2.01

[a]First letters "C" and "L" denote commercial and laboratory heats, respectively.
[b]Commercial-purity Type 316 SS.
[c]High-purity Type 316 SS.
[d]Commercial-purity Type 348 SS.
[e]High-purity Type 348 SS.

In an SSRT experiment similar to those in the present study, Jenssen and Ljungberg[18] irradiated U-notched rod specimens that had been fabricated from two heats of Type 316 SS. They conducted postirradiation SSRT tests in a BWR loop under normal oxidizing water chemistry. The CP heat of Type 316 SS (Heat K) was resistant to IASCC, whereas the HP heat of Type 316L SS (Heat F) was susceptible. The chemical composition, irradiation and test conditions, and test results for the 4 heats of Type 316 SS are summarized in Table 5. The two relatively more susceptible heats in the table (i.e., Heats

L22 and F) are characterized by an unusually low Si concentration of <0.26 wt.%, whereas the two relatively more resistant heats (i.e., Heats C21 and K) contain a higher Si concentration of >0.5 wt.%. The greater susceptibility of HP heats of Type 316L SS, when compared to CP heats observed in the present tests and in the work of Jenssen and Ljungberg,[18] is similar to that observed for BWR neutron absorber tubes fabricated from HP heats of Type 304 SS.[14]

Table 2. Specimen number per alloy, irradiation fluence, and postirradiation test type.

ANL Alloy	Number of SSRT Test per Fluence Level			Number of Constant Load Test			Number of J-R or Crack Growth Rate Test		
ID	high[a]	medium[a]	low[a]	high	medium	low	high	medium	low
C1	1	1	1						
L2	1	1					1	1	
C3	1	1	1				1	1	1
L4	1	1	1						
L5	1	1	1				1		
L6	1	1							
L7	1	1							
L8	1	1	1						
C9	1	1	1						
C10	1	1	1						
L11	1	1	1						
C12	1	1	1						
L13	1	1	1						
L14	1	1					1		
L15	1	1							
C16	1	1	1				1	1	
L17	1	1							
L18	1	1	1				1	1	
C19	5	1	1	4	4		1	1	1
L20	5	1	1	4	4		1	1	1
C21	1	1	1				1	1	1
L22	1	1	1				1	1	
L23	1	1					1		
L24	1	1					1		
L25C3	3								
L26C19	3								
L27C21	2								

[a]Fluence level in 10^{21} n cm^{-2} (E > 1 MeV), high ≈2.5, medium ≈0.9, and low ≈0.3.

Of the 13 alloys in Table 4, 3 alloys (L22, L11, and L20) contain low concentrations of Si, C, and N, whereas 1 alloy (L13) contains low concentrations of C and N but a high concentration of Si. The 3 alloys that contained low concentrations of Si (0.02-0.47 wt.%) exhibited consistently greater strengths than the alloy that contains Si concentrations >1.18 wt.%; this is shown in Fig. 1. Consistent with this greater strength, the three low-Si alloys exhibited significantly lower ductility than the high-Si alloy, i.e., 3.8-9.4 vs. 24.8%. The degree to which these differences connote a difference in irradiation hardening vs. the degree to which they reflect differences in the strength of the materials due to the variations in composition can't be determined definitively until more data are available. Additional baseline tests on the unirradiated materials will be performed.

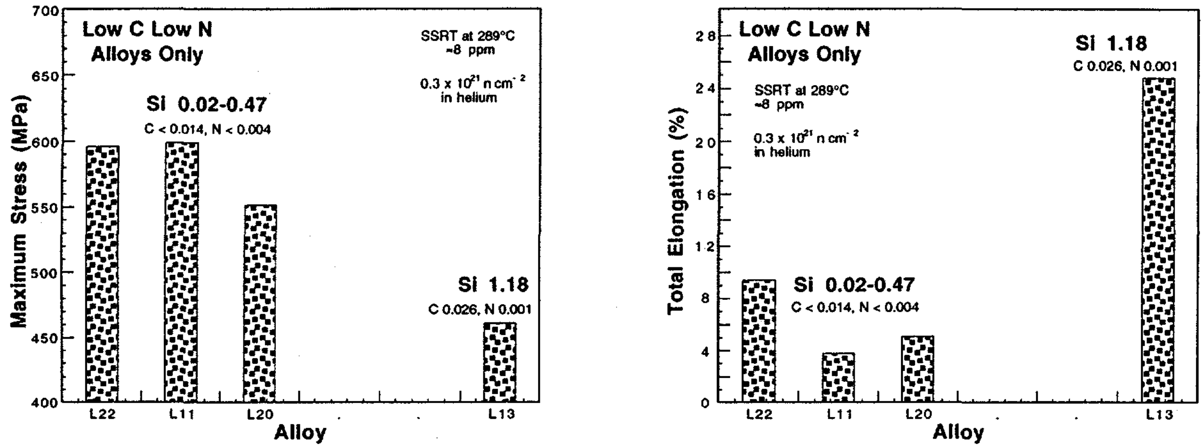


Figure 1. Effects of Si concentration on maximum strength (left) and total elongation (right) of model stainless steel alloys that contain low carbon (<0.03 wt.%) and low nitrogen (<0.004 wt.%) and were irradiated to ≈0.3 x 10^{-21}n cm^{-2} (E > 1 MeV).

Table 3. Results of SSRT[a] test and SEM fractography for model austenitic stainless steels irradiated in helium at 289°C to a fluence of ≈0.3 x 10^{21} n·cm^{-2} (E > 1 MeV)

Alloy and Specimen Ident. No.	SSRT No.	Feedwater Chemistry: Oxygen Conc. (ppm)	Feedwater Chemistry: Average ECP (mV SHE)	Feedwater Chemistry: Cond. at 25°C (μS·cm^{-1})	Feedwater Chemistry: pH at 25°C	SSRT Parameters: Yield Stress (MPa)	SSRT Parameters: Max. Stress (MPa)	SSRT Parameters: Uniform Elongation (%)	SSRT Parameters: Total Elongation (%)	Fracture Behavior: TGSCC (%)	Fracture Behavior: IGSCC (%)	Fracture Behavior: TGSCC + IGSCC (%)
C1-1	HR-1	8.3	+184	0.07	7.03	490	680	13.4	16.6	4	0	4
L5-1	HR-2	9.7	+208	0.07	6.89	413	539	29.5	32.7	2	2	4
L22-1	HR-3	8.0	+236	0.07	6.80	360	596	6.6	9.4	50	15	65
C3-1	HR-4	8.7	+161	0.07	6.68	338	491	27.7	31.6	5	0	5
C16-1	HR-5	8.3	+204	0.08	6.74	370	527	17.6	20.6	2	0	2
L4-1	HR-6	9.0	+202	0.08	6.70	367	542	19.7	22.3	46	0	46
L18-1	HR-7	9.0	+203	0.08	6.33	503	572	6.3	8.8	54	0	54
C10-1	HR-8	8.2	+174	0.07	6.35	523	640	17.4	18.9	6	0	6
C21-1	HR-9	8.1	+149	0.08	6.49	480	620	15.9	19.4	4	0	4
L11-1	HR-10	9.0	+157	0.08	6.17	487	599	2.3	3.8	62	0	62
L13-1	HR-11	8.7	+164	0.08	6.17	248	461	22.1	24.8	8	0	8
L20-1	HR-12	8.4	+174	0.07	6.20	454	552	2.9	5.1	32	2	34
C19-1	HR-13	9.5	+132	0.12	6.36	554	682	10.5	14.7	7	0	7
C9-1	HR-14	8.0	+192	0.11	6.30	522	607	13.4	14.6	24	0	24
C12-1	HR-15	9.0	+195	0.08	6.40	404	589	20.4	24.2	5	0	5
L8-1	HR-16	9.0	+215	0.08	6.60	411	571	15.6	17.9	54	0	54

[a]Tested at 289°C at a strain rate of 1.65 x 10^{-7} s^{-1} in BWR-simulated water containing ≈8 ppm DO.

The percentages of TGSCC and IGSCC observed on the fracture surfaces of the 16 alloys for a fluence of ≈0.3 x 10^{21} n·cm^{-2} are shown in Figs. 2 and 3, respectively. Significant TGSCC was observed in 7 of the alloys, but only one alloy showed significant IGSCC at this fluence. As shown in Fig. 4, all of the alloys that contain <0.01 wt.% N and <1.0 wt.% Si exhibited significant TGSCC, whereas all of the alloys that contain >0.01 wt.% N or >1.0 wt.% Si were relatively resistant to TGSCC. Practically all commercial heats of Types 304 or 304L SS contain >0.01 wt.% N, but the Si concentration would typically be on the order of 0.5 wt.%.

Table 4. SSRT[a] test and SEM fractography results for austenitic stainless steel model irradiated to 0.3 x 10^{21} n cm^{-2} in He at 289°C in the Halden reactor (composition in wt.%)

Alloy ID	Ni	Si	P	S	Mn	C	N	Cr	Mo/Nb	Remarks	YS (MPa)	UTS (MPa)	UE (%)	TE (%)	TGSCC (%)	IGSCC (%)	TG+IGSCC (%)
C1	8.12	0.50	0.038	0.002	1.00	0.060	0.060	18.11	-	Low S, CP 304	490	680	13.4	16.6	4	0	4
L5	9.66	0.90	0.113	0.028	0.47	0.006	0.033	21.00	-	High P, Cr; Low C	413	539	29.5	32.7	2	2	4
L22	13.30	0.024	0.015	0.004	0.40	0.003	0.001	16.10	Mo 2.04	HP 316L, low Si, N	360	596	6.6	9.4	50	15	65
C3	8.91	0.46	0.019	0.004	1.81	0.016	0.083	18.55	-	CP 304L, Low Si	338	491	27.7	31.6	5	0	5
C16	12.90	0.38	0.014	0.002	1.66	0.020	0.011	16.92	-	High Ni; Low Si, S	370	527	17.6	20.6	2	0	2
L4	10.20	0.94	0.031	0.010	1.75	0.110	0.002	15.80	-	High Ni, Mn, C; Low N	367	542	19.7	22.3	38	0	38
L18	8.13	0.14	0.016	0.033	1.13	0.080	0.001	18.00	-	Low Si, N	503	572	6.3	8.8	54	0	54
C10	8.13	0.55	0.033	0.002	1.00	0.060	0.086	18.19	-	Low S, CP 304	523	640	17.4	18.9	6	0	6
C21	10.24	0.51	0.034	0.001	1.19	0.060	0.020	16.28	Mo 2.08	CP 316	480	620	15.9	19.4	4	0	4
L11	8.15	0.47	0.097	0.009	1.02	0.014	0.004	17.40	-	High P; Low Si, C, S, N	487	599	2.3	3.8	62	0	62
L13	8.18	1.18	0.027	0.022	0.36	0.026	0.001	17.40	-	High Si; Low Mn, C, N	248	461	22.1	24.8	8	0	8
L20	8.91	0.017	0.010	0.004	0.41	0.002	0.002	18.10	-	HP 304L, Low Si, N	454	552	2.9	5.1	32	2	34
C19	8.08	0.45	0.031	0.003	0.99	0.060	0.070	18.21	-	Low Si, S	554	682	10.5	14.7	7	0	7
C9	8.75	0.39	0.013	0.013	1.72	0.062	0.065	18.48	-	Low Si; High Mn	522	607	13.4	14.6	24	0	24
C12	8.23	0.47	0.018	0.002	1.00	0.060	0.070	18.43	-	Low Si, P, S	404	589	20.4	24.2	5	0	5
L8	10.20	0.15	0.093	0.010	1.85	0.041	0.001	18.30	-	High Ni, P, Mn; Low Si, N	411	571	15.6	17.8	64	0	64

[a]Test at 289°C at a strain rate of 1.65 x 10^{-7} s^{-1} in simulated BWR water; DO ≈ 8 ppm, effluent ECP +140 to +236 mV SHE, conductivity at 25°C 0.07-0.11 $\mu S\ cm^{-1}$, and pH 6.2-7.0.

Table 5. Composition (in wt.%) and relative susceptibility to IASCC of Type 316 stainless steels irradiated and tested under BWR-like conditions

Heat ID	Steel Type[a]	Source	Ni	Si	P	S	Mn	C	N	B	Cr	Mo	Irradiated in Reactor	Fluence, 10^{21} n cm^{-2}	Type of SCC Test	Relative Susceptibility
L22	HP 316L	this work	13.30	0.024	0.015	0.004	0.40	0.003	0.001	<0.001	16.10	2.04	Halden, He	0.3 and 0.9	SSRT in hot cell	high
C21	CP 316	this work	10.24	0.51	0.034	0.001	1.19	0.060	0.020	<0.001	16.28	2.08	Halden, He	0.3	SSRT in hot cell	low
F	HP 316L	Ref. 18	11.60	0.26	0.021	0.001	1.44	0.009	0.062	0.001	16.69	2.65	BWR	0.3-9.0	SSRT in BWR loop	high
K	CP 316	Ref. 18	12.40	0.64	0.016	0.006	1.73	0.055	0.029	<0.0004	16.51	2.25	BWR	0.3-9.0	SSRT in BWR loop	low

[a]HP = high purity, CP = commercial purity.

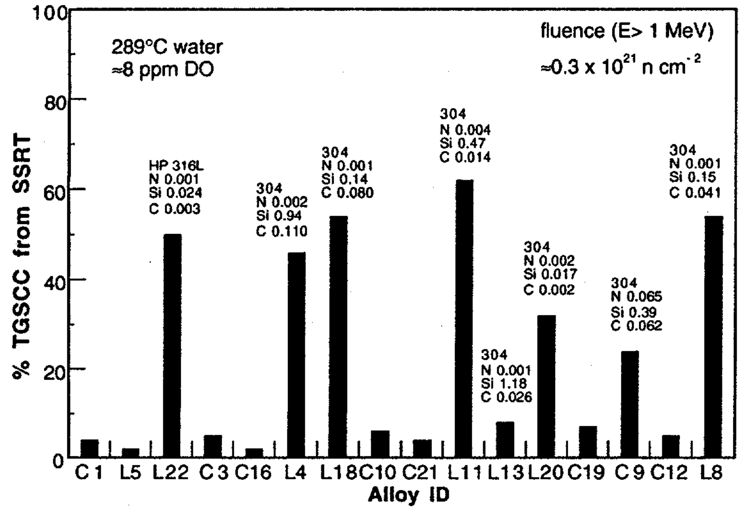


Figure 2.

Percent TGSCC on fracture surfaces of stainless steel alloys irradiated to fluence of ≈0.3 x 10^{21} n cm^{-2} and tested at 289°C in simulated BWR water.

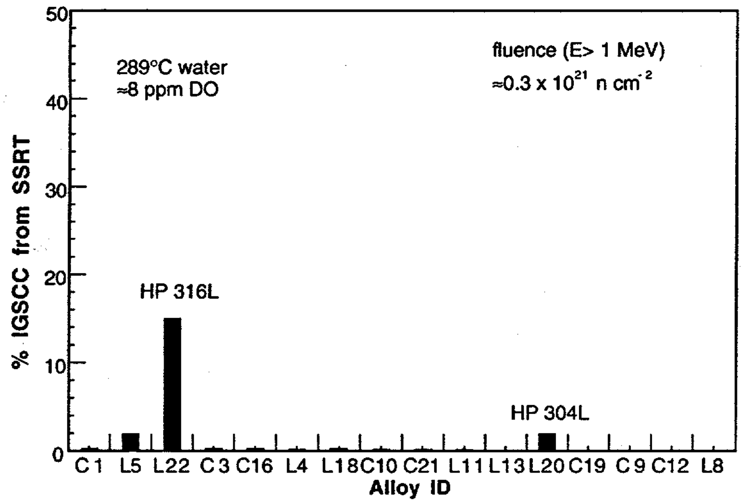


Figure 3.

Percent IGSCC on fracture surfaces of stainless steel alloys irradiated to fluence of ≈0.3 x 10^{21} n cm^{-2} (E > 1 MeV) and tested at 288°C in simulated BWR water that contained DO ≈8 ppm.

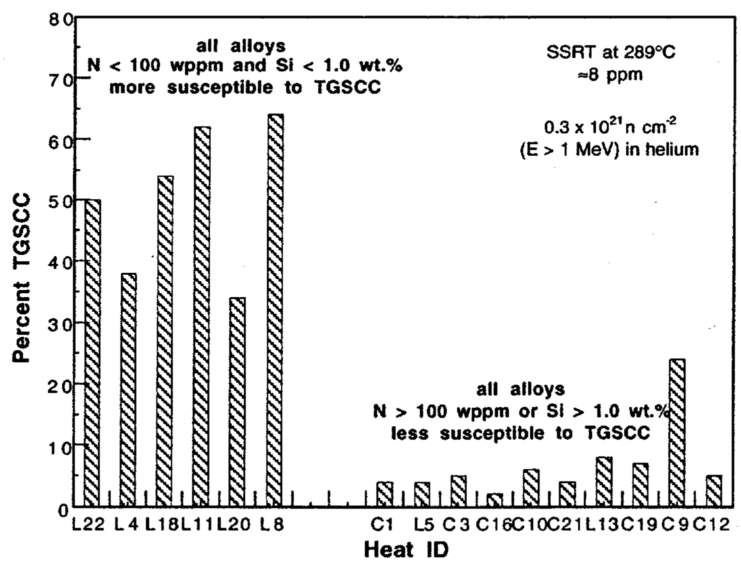


Figure 4.

Percent TGSCC on fracture surfaces of stainless steel alloys, irradiated to fluence of ≈0.3 x 10^{21} n cm^{-2} (E > 1 MeV) and tested at 288°C in simulated BWR water, classified as a function of N and Si content of the alloys.

Miwa et al.[22] and Tsukada et al.[23] irradiated two Type 304L SS specimens at ≈240°C to a fluence of ≈0.67 x 10^{21} n cm^{-2}. One specimen contained low Si, the other was doped to ≈0.69% Si. In subsequent SSRT tests at ≈300°C, the high-Si specimen exhibited significantly greater ductility than the low-Si specimen, namely, total elongation was ≈21% vs. ≈11% for the low-Si specimen. Miwa et al.[22] observed that the number density of Frank loops was significantly lower in the high-Si specimen than in the low-Si specimen, although the number densities of "black-dot" defect clusters appeared similar. Because the SSRT test temperature was ≈60°C higher than the irradiation temperature, some fraction of the irradiation-induced defects, defect clusters, and loops probably annealed out during the test. Furthermore, different types of irradiation-induced interactions may occur between irradiation-induced defect clusters and impurities at ≈240°C and ≈300°C. Susceptibilities to IGSCC of the two specimens were similar, but the high-Si specimen showed more TGSCC than the low-Si specimen, a finding that is the opposite of the trend observed in the present study.

SSRT Testing and Fractographic Analysis of Medium-Fluence Specimens

Tests were conducted on nine "medium-fluence" specimens irradiated to ≈0.9 x 10^{21} n·cm^{-2}. The results are summarized in Tables 6 and 7 and in Fig. 5. The effects of the higher fluence on yield stress, maximum stress, uniform strain, total strain, percent IGSCC, and percent TGSCC were significant. When the fluence was increased from ≈0.3 x 10^{21} n·cm^{-2} to ≈0.9 x 10^{21} n·cm^{-2} in the low-N and low-Si alloys (e.g., Heats L22, L18, and L11), the percentage of TGSCC observed on the fracture surfaces decreased, but at the same time, the percentage of IGSCC increased (see Figs. 5E and F). This trend is consistent with that observed on field-cracked BWR components. The threshold fluence for the transition from TGSCC to IGSCC appears to differ from alloy to alloy. For example, the percentage of TGSCC of Alloy C9, a commercial heat of Type 304 SS, increased when fluence increased from ≈0.3 x 10^{21} n cm^{-2} (E > 1 MeV) to ≈0.9 x 10^{21} n cm^{-2}.

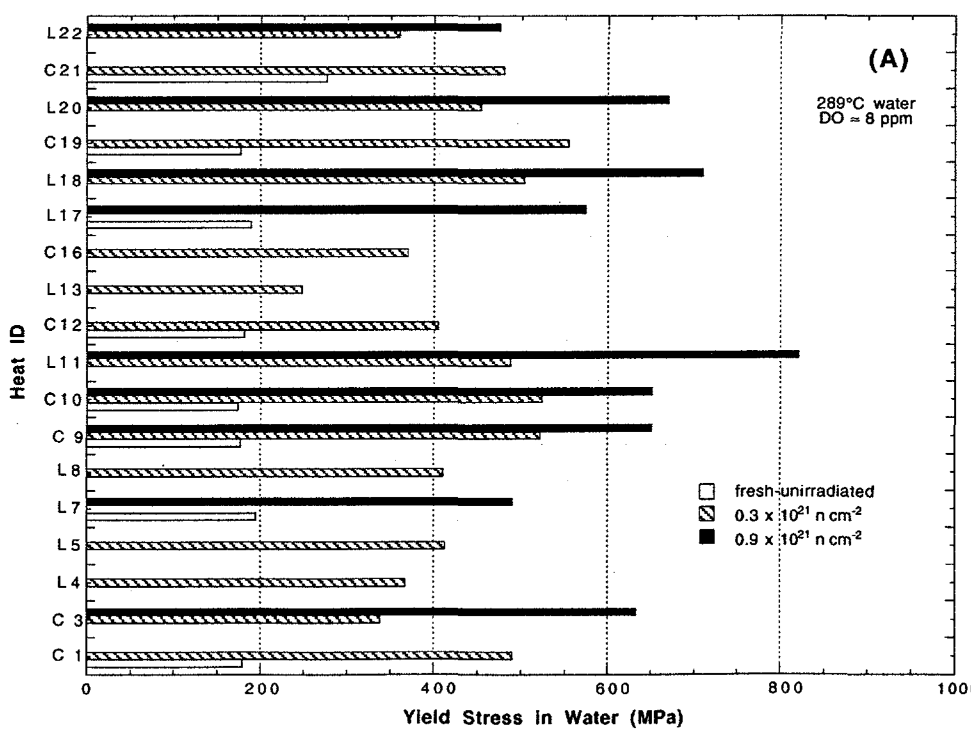
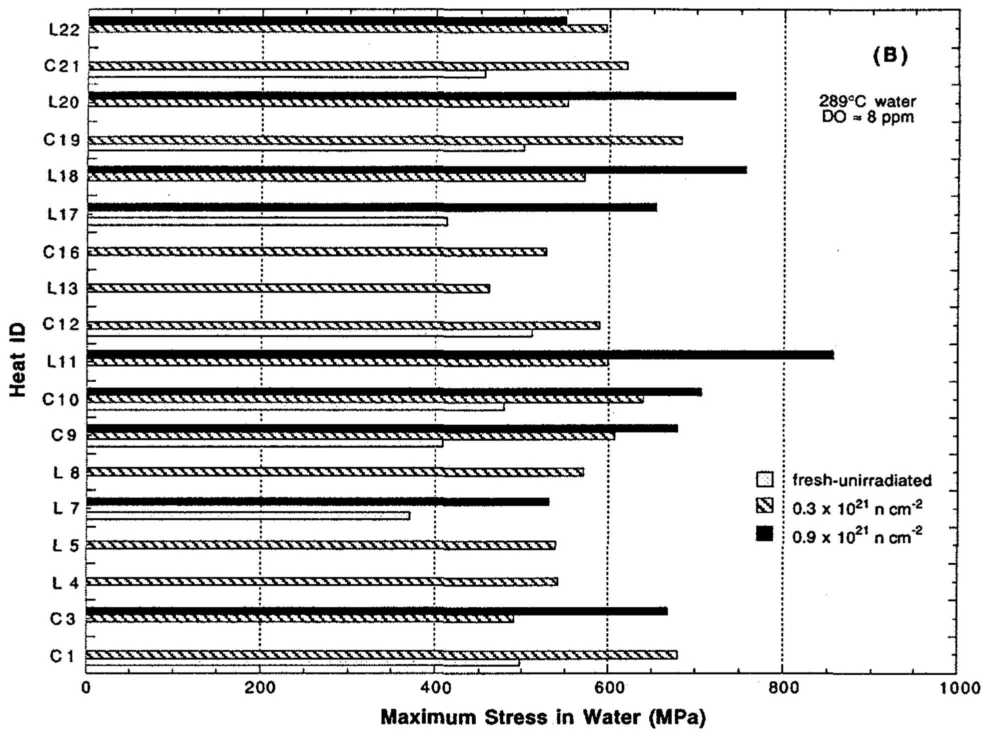


Figure 5. Effects of fluence on: (A) yield strength, (B) maximum strength measured in 289°C water containing ≈8 ppm DO.



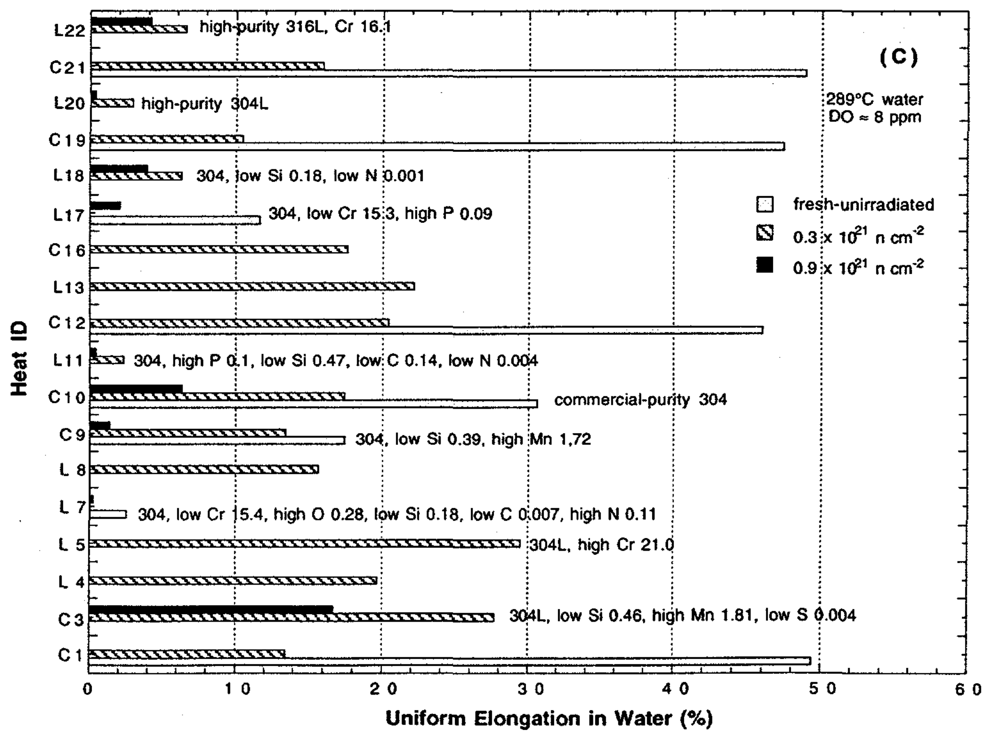
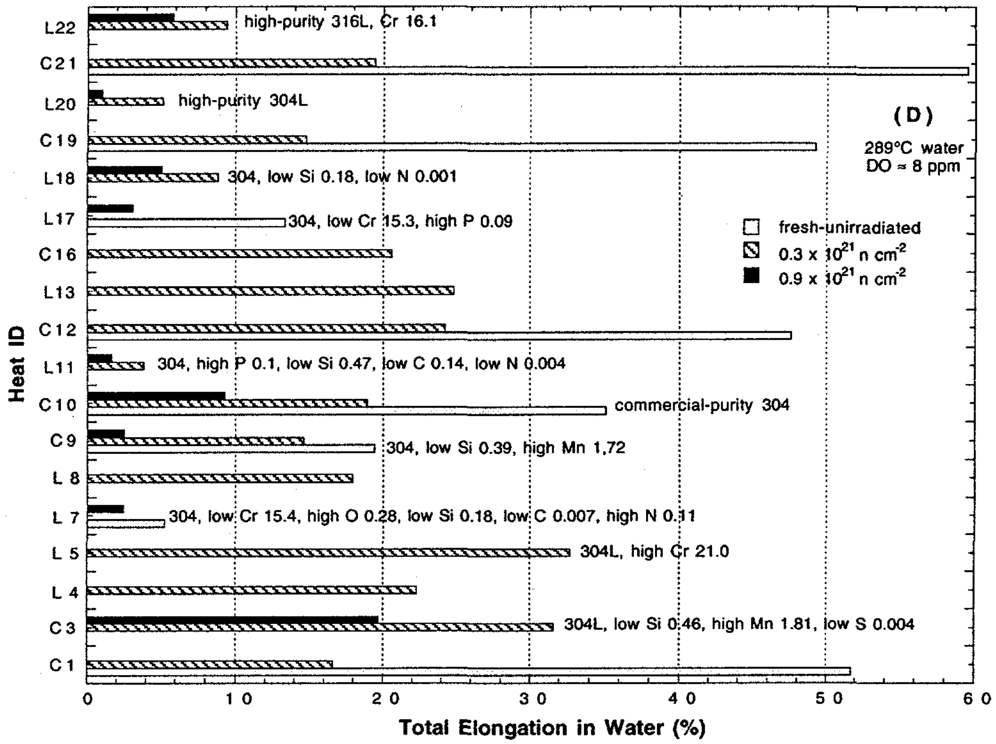


Figure 5. Continued.



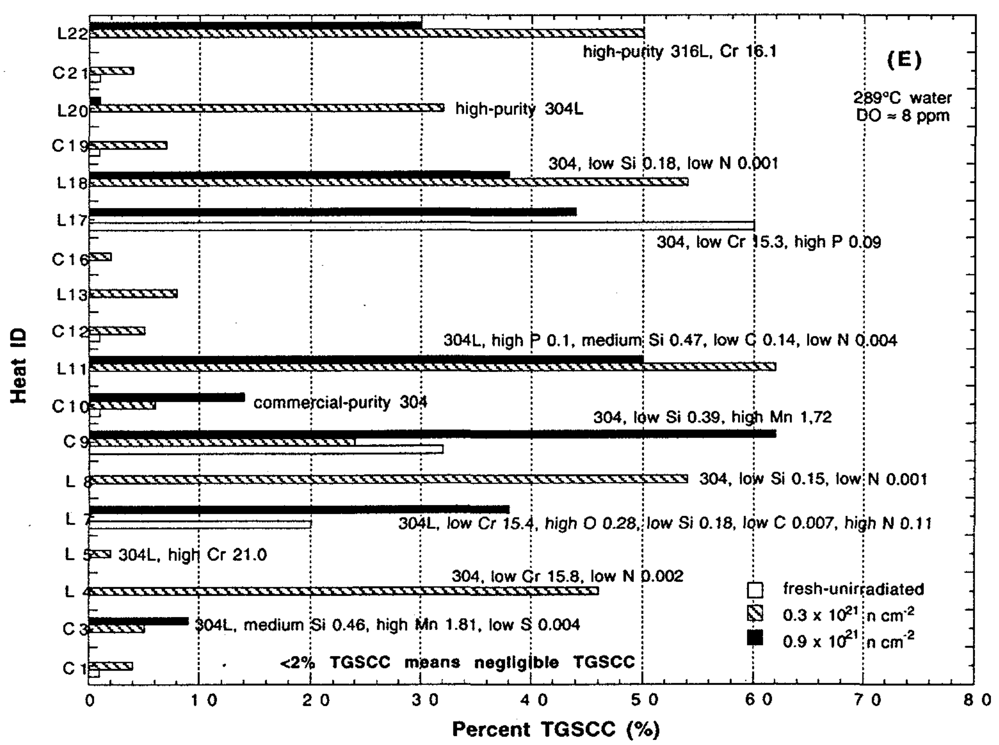


Figure 5. Continued.

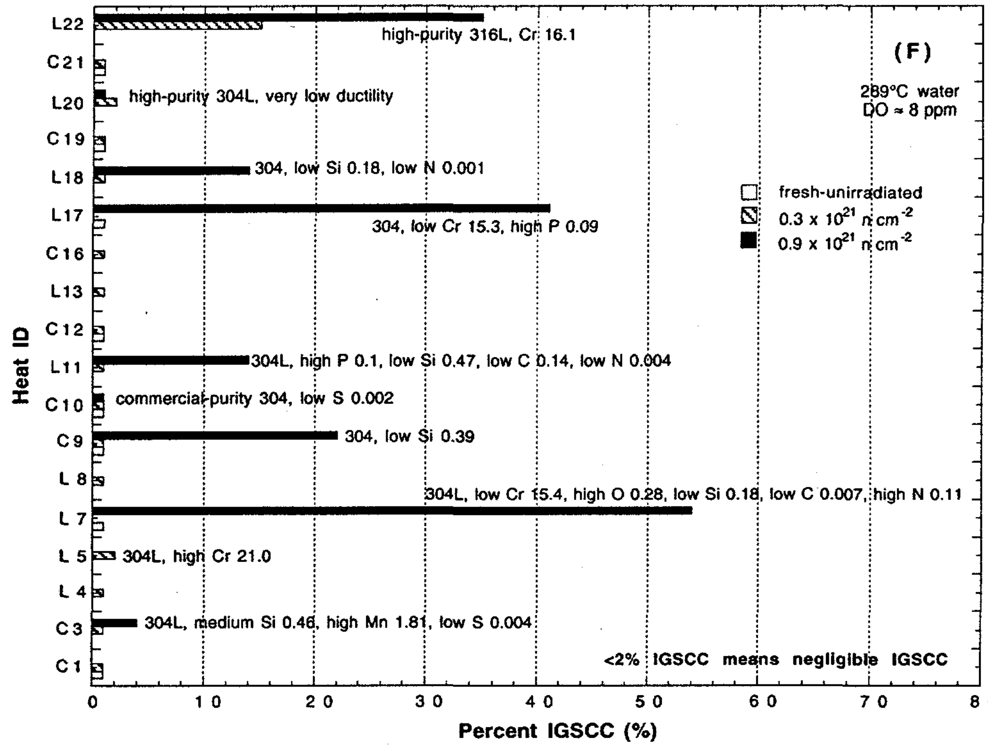


Figure 5. Continued.

The percentage IGSCC on the fracture surfaces of all of the alloys that contain <0.5 wt.% Si (i.e., L22, L18, L11, C9, and L7) increased significantly when the fluence was increased from ≈0.3 x 10^{21} n·cm^{-2} to ≈0.9 x 10^{21} n·cm^{-2} (see Fig. 5F).

For the fluence level of ≈0.9 x 10^{21} n·cm^{-2}, the percentages of IGSCC on the fracture surfaces of L17 (Cr ≈ 15.3 wt.%) and L7 (Cr ≈ 15.4 wt.%) were significantly greater than those observed for alloys that contain more typical Cr concentrations of ≈18 wt.% (see Fig. 5F). Consistent with this observation, Alloy L5, which contains an unusually high Cr concentration of ≈ 21.0 wt.%, showed little TGSCC or IGSCC at ≈0.3 x 10^{21} n·cm^{-2} (see Figs. 5E and F, respectively). This alloy also contains the relatively high concentration of Si of ≈0.90 wt.%. Alloy L5 also exhibited the highest ductility among all of the alloys that were irradiated to ≈0.3 x 10^{21} n·cm^{-2} (see Fig. 5D).

Alloy L7, a laboratory heat of Type 304L SS, exhibited significant TGSCC even in the unirradiated state[21] and the greatest degree of IGSCC after irradiation to ≈0.9 x 10^{21} n·cm^{-2}. The high susceptibility to IASCC of this alloy may be related to several deleterious compositional characteristics, i.e., low concentration of Cr (≈15.3 wt.%), high concentration of O (≈0.027 wt.%),[10,14,20,24] low concentration of Si (≈0.18 wt.% Si), and low concentration of C (≈0.007 wt.%).[14]

There are indications that a combination of a high concentration of Mn and low concentration of S may be beneficial. For example, Alloy C3, a CP heat of Type 304L SS that contains ≈1.81 wt.% Mn and ≈0.004 wt.% S exhibited unusually high ductility (>20%), low percentage of TGSCC (<9%), and low percentage of IGSCC (<4%) after irradiation to ≈0.3 x 10^{21} n·cm^{-2} and ≈0.9 x 10^{21} n·cm^{-2} (see Figs. 5D, E, and F, respectively). However, conclusions on the effects of Mn, S, N, O, and C must be established on the basis of the data to be obtained from the whole test matrix.

Table 6. Results of SSRT[a] test and SEM fractography for austenitic stainless steel model alloys irradiated in He to fluence of ≈0.9 x 10^{-21} n cm^{-2} (E > 1 MeV) at 289°C.

Specimen Ident. No.	SSRT No.	Feedwater Chemistry: Oxygen Conc. (ppm)	Average ECP (mV SHE)	Cond. at 25°C ($\mu S \cdot cm^{-1}$)	pH at 25°C	SSRT Parameters: Yield Stress (MPa)	Max. Stress (MPa)	Uniform Elongation (%)	Total Elongation (%)	Fracture Behavior: TGSCC (%)	IGSCC (%)	TGSCC + IGSCC (%)
L22-02	HR-17	8.0	+181	0.08	6.77	475	549	4.20	5.82	30	35	65
L11-02	HR-18	8.0	+191	0.08	6.55	820	856	0.43	1.65	50	14	64
L18-02	HR-19	8.0	+193	0.10	6.07	710	755	3.98	5.05	38	14	52
L20-04	HR-20	8.0	+225	0.07	6.75	515	574	1.85	3.36	erratic	pressure,	test invalid
L20-05	HR-26	9.0	+182	0.09	3.62	670	743	0.37	1.03	0	0	0
C9-02	HR-21	8.0	+240	0.07	6.47	651	679	1.42	2.50	62	22	84
L17-02	HR-22	8.0	+198	0.07	6.42	574	654	2.02	3.08	44	41	85
L7-02	HR-23	8.0	+215	0.07	6.03	490	531	0.24	2.44	38	54	92
C10-02	HR-24	7.0	+221	0.07	5.26	651	706	6.35	9.25	14	0	14
C3-02	HR-25	8.0	+240	0.07	6.34	632	668	16.72	19.74	9	4	13

[a]Test at 289°C at a strain rate of 1.65 x 10^{-7} s^{-1} in simulated BWR water that contained ≈8 ppm DO.

Table 7. Composition (in wt.%) of model austenitic stainless steels irradiated to fluence of ≈0.9 x 10^{21} $n \cdot cm^{-2}$ (E > 1 MeV), correlated with results of SSRT[a] test and SEM fractography

Specimen ID	Ni	Si	P	S	Mn	C	N	Cr	O, B, Mo, Nb	Remark	YS (MPa)	UTS (MPa)	UE (%)	TE (%)	TGSCC (%)	IGSCC (%)	TG+IGSCC (%)
L22-02	13.30	0.024	0.015	0.004	0.40	0.003	0.001	16.10	Mo 2.04	HP 316L; Low Si, N	475	549	4.20	5.82	30	35	65
L11-02	8.15	0.47	0.097	0.009	1.02	0.014	0.004	17.40	-	high P; low Si, C, S, N	820	856	0.43	1.65	50	14	64
L18-02	8.13	0.14	0.016	0.033	1.13	0.080	0.001	18.00	-	low Si, N	710	755	3.98	5.05	38	14	52
L20-04	8.91	0.017	0.010	0.004	0.41	0.002	0.002	18.10	-	HP 304L; low Si, N, Mn	515	574	1.85	3.36	erratic	pressure,	test invalid
L20-05	8.91	0.017	0.010	0.004	0.41	0.002	0.002	18.10	-	HP 304L; low Si, N, Mn	670	743	0.37	1.03	0	0	0
C9-02	8.75	0.39	0.013	0.013	1.72	0.062	0.065	18.48	-	low Si; high Mn	651	679	1.42	2.50	62	22	84
L17-02	8.00	0.66	0.090	0.009	0.48	0.061	0.078	15.30	-	high P; low Cr, Mn, S	574	654	2.02	3.08	44	41	85
L7-02	10.60	0.18	0.040	0.038	1.02	0.007	0.111	15.40	O 0.0274	high N, O; low Si, C	490	531	0.24	2.44	38	54	92
C10-02	8.13	0.55	0.033	0.002	1.00	0.060	0.086	18.19	-	CP 304; low S; high N	651	706	6.35	9.25	14	0	14
C3-02	8.91	0.46	0.019	0.004	1.81	0.016	0.083	18.55	-	CP 304L; high Mn, N; low S	632	668	16.72	19.74	9	4	13

[a]Test at 289°C at a strain rate of 1.65 x 10^{-7} s^{-1} in simulated water water that contained ≈8 ppm DO.
[b]HP = high purity, CP = commercial purity.

Summary

1. Slow-strain-rate tensile tests and posttest fractographic analyses by scanning electron microscopy were conducted on 16 austenitic stainless steel alloys that were irradiated at 289°C in He. After irradiation to $\approx 0.3 \times 10^{21}$ n·cm^{-2} and $\approx 0.9 \times 10^{21}$ n·cm^{-2} (E > 1 MeV), significant heat-to-heat variations in the degree of IGSCC and TGSCC were observed. After irradiation to a fluence of $\approx 0.3 \times 10^{21}$ n·cm^{-2}, a high-purity laboratory heat of Type 316L SS, which contains very low Si, exhibited the highest susceptibility to IGSCC. The other 15 alloys exhibited negligible susceptibility to IGSCC at this low fluence.

2. The percentage of TGSCC on the fracture surfaces of SSRT specimens of the 16 alloys at $\approx 0.3 \times 10^{21}$ n·cm^{-2} (E > 1 MeV) could be correlated with N and Si concentrations. All of the alloys that contained <0.01 wt.% N and <1.0 wt.% Si were susceptible, whereas all of the alloys that contained >0.01 wt.% N or >1.0 wt.% Si were relatively resistant. Practically all commercial heats of Type 304 or 304L SSs contain >0.01 wt.% N, but the Si concentration would typically be ≈0.5 wt.%. Results of initial tests on alloys irradiated to a fluence of $\approx 0.9 \times 10^{21}$ n·cm^{-2} are consistent with the finding that a low level of Si (<0.5 wt.%) is conducive to relatively higher susceptibility to IASCC. Consistent with the effect of Si concentration in Type 304 SSs, a lower concentration of Si (e.g., <0.26 wt.%) appears to promote higher susceptibility of high-purity heats of Type 316 SS to IASCC when compared with commercial-purity heats that contain higher concentrations of Si. A certain minimum concentration of Si, and probably N, may be helpful in stabilizing the irradiation-induced defect structure by suppressing formation of some type(s) of hardening centers.

3. High concentrations of Cr were beneficial. Alloys that contain <15.5 wt.% Cr exhibited greater percentages of TGSCC and IGSCC than alloys with ≈18 wt.% Cr, whereas an alloy that contains >21 wt.% Cr exhibited less susceptibility than the lower Cr alloys under similar conditions.

4. Susceptibility to IASCC is influenced by many alloying and impurity elements in a complex manner. Higher concentrations of Cr and Si appear to be beneficial, whereas high concentrations of O and very low concentrations of N (<0.01 wt.%) appear to be deleterious. There are indications that a combination of a high concentration of Mn and low concentration of S is beneficial. However, conclusions on the effects of Mn, S, N, O, and C must be established on the basis of data to be obtained from the whole test matrix.

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References

1. M. E. Indig, J. L. Nelson, and G. P. Wozadlo, in *Proc. 5th Intl. Symp. on Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors*, D. Cubicciotti, E. P. Simonen, and R. Gold, eds., American Nuclear Society, La Grange Park, IL, 1992, p. 941.

2. M. Kodama, S. Nishimura, J. Morisawa, S. Shima, S. Suzuki, and M. Yamamoto, ibid., p. 948.

3. H. M. Chung, W. E. Ruther, J. E. Sanecki, A. G. Hins, and T. F. Kassner, in *Proc. 7th Intl. Symp. on Environmental Degradation of Materials in Nuclear Power Systems - Water Reactors*, NACE International, Houston, 1995, p. 1133-1143.

4. F. Garzarolli, P. Dewes, R. Hahn, and J. L. Nelson, in *Proc. 6th Intl. Symp. on Environmental Degradation of Materials in Nuclear Power Systems - Water Reactors*, R. E. Gold and E. P. Simonen, eds., The Minerals, Metals, and Materials Society, Warrendale, PA, 1993, p. 511.

5. I. Suzuki, M. Koyama, H. Kanasaki, H. Mimaki, M. Akiyama, T. Okubo, Y. Mishima, and T. R. Mager, in *Proc. ASME-JSME 4th Intl. Conf. on Nuclear Engineering*, March 10–14, 1996, New Orleans, American Society of Mechanical Engineers, New York, NY, 1996, Vol. 5, pp. 205–213.

6. A. J. Jacobs, G. P. Wozadlo, K. Nakata, T. Yoshida, and I. Masaoka, in *Proc. 3rd Int. Symp. Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors*, G. J. Theus and J. R. Weeks, eds., The Metallurgical Society, Warrendale, PA, 1988, p. 673.

7. K. Fukuya, K. Nakata, and A. Horie, in *Proc. 5th Intl. Symp. on Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors*, D. Cubicciotti, E. P. Simonen, and R. Gold, eds., American Nuclear Society, La Grange Park, IL, 1992, pp. 814–820.

8. H. M. Chung, W. E. Ruther, J. E. Sanecki, A. G. Hins, and T. F. Kassner, in *Effects of Radiation on Materials: 16th Int. Symp.*, ASTM STP 1175, A. S. Kumar, D. S. Gelles, R. K. Nanstad, and T. A. Little, eds., American Society for Testing and Materials, Philadelphia, 1993, pp. 851-869.

9. H. M. Chung, W. E. Ruther, J. E. Sanecki, and T. F. Kassner, in *Proc. 6th Intl. Symp. on Environmental Degradation of Materials in Nuclear Power Systems - Water Reactors*, R. E. Gold and E. P. Simonen, eds., The Minerals, Metals, and Materials Society, Warrendale, PA, 1993, pp. 511-519.

10. J. M. Cookson, D. L. Damcott, G. S. Was, and P. L. Anderson, ibid., pp. 573-580.

11. M. Kodama, J. Morisawa, S. Nishimura, K. Asano, S. Shima, and K. Nakata, *J. Nucl. Mater.*, 1509 (1994) pp. 212–215.

12. T. Tsukada and Y. Miwa, in *Proc. 7th Intl. Symp. on Environmental Degradation of Materials in Nuclear Power Systems - Water Reactors*, NACE International, Houston, 1995, pp. 1009–1018.

13. F. Garzarolli, P. Dewes, R. Hahn, and J. L. Nelson, ibid., 1055–1065.

14. H. M. Chung, W. E. Ruther, J. E. Sanecki, A. G. Hins, N. J. Zaluzec, and T. F. Kassner, *J. Nucl. Mater.*, 239 (1996) 61.

15. J. M. Cookson, G. S. Was, and P. L. Anderson, *Corrosion* 54 (1998) 299.

16. S. Kasahara, K. Nakata, K. Fukuya, S. Shima, A. J. Jacobs, G. P. Wozadlo, and S. Suzuki, in *Proc. 6th Intl. Symp. on Environmental Degradation of Materials in Nuclear Power Systems - Water Reactors*, R. E. Gold and E. P. Simonen, eds., The Minerals, Metals, and Materials Society, Warrendale, PA, 1993, pp. 615-623.

17. A. J. Jacobs, G. P. Wozadlo, K. Nakata, S. Kasahara, T. Okada, S. Kawano, and S. Suzuki, in *Proc. 6th Intl. Symp. on Environmental Degradation of Materials in Nuclear Power Systems - Water Reactors*, R. E. Gold and E. P. Simonen, eds., The Minerals, Metals, and Materials Society, Warrendale, PA, 1993, pp. 597-606.

18. A. Jenssen and L. G. Ljungberg, in *Proc. 7th Intl. Symp. on Environmental Degradation of Materials in Nuclear Power Systems - Water Reactors*, NACE International, Houston, 1995, 1043–1052.

19. G. Taguchi, in *Quality Engineering Using Robust Design*, M. S. Phadke, Prentice Hall, Englewood Cliffs, NJ, 1989.

20. H. M. Chung, W. E. Ruther, R. V. Strain, and T. M. Karlsen, in *Environmentally Assisted Cracking in Light Water Reactors, Semiannual Report, January-June 1998*, NUREG/CR-4667, Vol. 26, ANL-98/30, pp. 10-21 (March 1999).

21. H. M. Chung, W. E. Ruther, R. V. Strain, and T. M. Karlsen, in *Environmentally Assisted Cracking in Light Water Reactors, Semiannual Report, July 1997 - December 1997*, NUREG/CR-4667, Vol. 25, ANL-98/18, pp. 26-38 (Sept. 1998).

22. Y. Miwa, T. Tsukada, S. Jitsukawa, S. Kita, S. Hamada, Y. Matsui, and M. Shindo, *J. Nucl. Mater.* 1393, (1996) pp. 233-237.

23. T. Tsukada, Y. Miwa, and H. Nakajima, in *Proc. 8th Intl. Symp. on Environmental Degradation of Materials in Nuclear Power Systems - Water Reactors*, August 10-14, 1997, Amelia Island, FL, S. M. Bruemmer, ed., American Nuclear Society, 1997, pp. 846-856.

24. H. M. Chung, J.-H. Park, W. E. Ruther, J. E. Sanecki, R. V. Strain, and N. J. Zaluzec, in *Proc. 8th Intl. Symp. on Environmental Degradation of Materials in Nuclear Power Systems - Water Reactors*, August 10-14, 1997, Amelia Island, FL, S. M. Bruemmer, ed., American Nuclear Society, 1997, pp. 846-856.