

Stress Corrosion Crack Initiation and Propagation under Different Corrosion Environmental conditions, and the Role of Post-Irradiation Annealing on Irradiation Assisted Stress Corrosion Cracking

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CONTENTS

1 EXECUTIVE SUMMARY	1-1
2 INTRODUCTION.....	2-1
3 EXPERIMENT	3-1
3.1 Materials and specimens.....	3-1
3.2 Microstructure Characterization	3-9
3.3 Test systems and procedures	3-11
3.3.2 Procedures for CERT test.....	3-13
3.3.3 Procedures for CGR Test.....	3-18
3.3.4 Crack Initiation Studies using Four Point Bend Test.....	3-22
4 RESULTS	4-1
4.1 Hardness	4-1
4.2 Irradiated Microstructure	4-3
4.2.1 Dislocation Loops.....	4-3
4.2.2 Solute Clusters.....	4-6
4.2.3 Grain Boundary Segregation.....	4-8
4.2.4 Overview of Microstructure of CIR Alloys.....	4-12
4.3 Crack initiation in CERT tests on PIA conditions.....	4-14
4.3.1 Prediction of Yield Stress and Incremental Straining Experiments.....	4-14
4.3.2 Stress-Strain Behavior.....	4-17
4.3.3 Mechanical Properties.....	4-18
4.3.4 SCC Behavior.....	4-19
4.3.5 Dislocation Channeling.....	4-23
4.4 Crack initiation in CERT tests on neutron-irradiated CIR alloys	4-27
4.4.1 Stress-Strain Curves.....	4-27
4.4.2 Mechanical Properties.....	4-29
4.4.3 SCC Behavior.....	4-31
4.5 Four-Point Bend Test Results.....	4-36

4.5.1 FEA results.....	4-36
4.5.2 Four-point bend tests of unirradiated samples.....	4-40
4.5.3 Four-point bend tests of irradiated samples.....	4-43
4.5.4 Comparison to Literature.....	4-48
4.6 CGR test results.....	4-50
4.6.1 304L Irradiated in Barsebackk 1 BWR.....	4-50
4.6.2 Neutron-irradiated CIR Alloys.....	4-60
5 DISCUSSION.....	5-1
5.1 Role of Environment on IASCC.....	5-1
5.1.1 Crack Initiation.....	5-1
5.1.2 Crack Growth Rate.....	5-3
5.2 Post irradiation Annealing.....	5-4
5.2.1 Crack Initiation.....	5-4
5.2.2 Crack Growth Rate.....	5-6
5.3 Irradiated Microstructure.....	5-6
5.4 Dislocation Channeling in IASCC.....	5-8
6 CONCLUSION.....	6-1
7 REFERENCES.....	7-1

1 EXECUTIVE SUMMARY

This program addresses the cause of IASCC in LWR core internals in both BWR normal water chemistry/hydrogen water chemistry, and PWR primary water, with the objective of providing a foundation for materials performance models and mitigation strategies by determining the key factors controlling the process. IASCC is generic in that all water reactors have exhibited susceptibility over a wide range of alloys and components. The problem will gain increasing importance in the coming years due to the need for life extension to 60 and perhaps to 80 years for the existing reactor fleet, and the introduction of advanced reactors that will be subjected to similar environments.

The program consists of two major tasks; determination of the factors affecting crack initiation in irradiated alloys strained in high temperature water, and utilizing post-irradiation annealing as both a mitigation technique for IASCC and to identify microstructure features controlling IASCC initiation. The first task focused on improving the capability to detect crack initiation and identifying the factors affecting it. This included development of the four-point bend test to determine the stress at initiation and the role of dislocation channels in the initiation process. It also includes the assessment of alloys and environments on IASCC initiation. The second part involves the use of post-irradiation annealing to identify the microstructure features responsible for IASCC initiation. By isolating the PIA conditions that mark the disappearance of IASCC, a comparison of microstructures may provide insight as to what change is responsible for the elimination of IASCC susceptibility. The outcome of this program is a firmer understanding of parameters and microstructures governing IASCC and a prescription for post-irradiation annealing to mitigate IASCC.

Crack initiation was studied by both CERT and four-point bend tests. CERT studies on neutron irradiated stainless steel alloys showed that the increase in the corrosion potential of the environment results in an increase in the cracking susceptibility of the alloy. At high potentials, the cracking susceptibility was determined by crack propagation since very few cracks nucleate and the nucleated crack rapidly grows to cause failure. At low potentials, cracking was predominantly controlled by crack initiation resulting in high crack density on the surface of the specimen. Thus, the degree of susceptibility depends on the figure of merit. When measured by %IG on the fracture surface, samples in NWC show greater susceptibility. But when measured by surface crack density, samples in PW are more susceptible.

Four-point bend tests were conducted to correlate the crack initiation behavior with dislocation channeling. The advantage of these tests is that the growth of crack nuclei was suppressed due to the increasingly compressive stress field normal to the surface. These tests were highly successful in catching crack initiation and linking it to the microstructure and dislocation channels. Depending on the dpa, dislocation channels form at stresses as low as 40% of the bulk yield stress. Cracks always nucleated at dislocation channel-grain boundary intersections either with the

formation of the channel or after it. MnS inclusion on the grain boundary were often the site of crack nucleation and their dissolution in NWC is likely a key factor in producing an environment that is conducive to cracking. It was also found that increasing dpa resulted in a lower stress to both initiate dislocation channels and cracks.

Post irradiation annealing was found to mitigate the cracking susceptibility in 304L irradiated in the Barseback 1 BWR. With one exception, higher degrees of annealing (time+temperature) resulted in a decrease in SCC susceptibility. All measures of IGSCC susceptibility (max stress, uniform strain, total strain, %IG) changed monotonically with heat treatment severity. The exception was an increase in IGSCC in the most severely annealed sample, which is not explainable with the resulting microstructure. Close inspection of the microstructure showed that all features (dislocation loops, clusters, RIS) changed smoothly and monotonically and comparison of the microstructures at annealing treatments that bracketed the onset of cracking were not able to identify the feature or features responsible for cracking.

The CGRs of as-irradiated (5.9 dpa) and irradiated + annealed (550 °C:20 h) 304L stainless steel samples have been measured in NWC and HWC environments. The results show that HWC can significantly mitigate the SCC propagation of both samples. The mitigating effect may be dependent on the inherent SCC susceptibility of material. The post-irradiation treatment has a significant mitigating effect on the SCC propagation of this material with a larger effect in HWC. The reduced SCC susceptibility of the annealed sample is consistent with the recovery in the irradiated microstructure (dislocation loop, solute cluster and grain boundary segregation). However, the determining microstructure change could not be specifically identified.

2 INTRODUCTION

Objectives and scope

This program addresses the cause of IASCC in LWR core internals in both BWR normal water chemistry and PWR primary water, with the objective of providing a foundation for materials performance models and mitigation strategies by determining the key factors controlling the process. Irradiation assisted stress corrosion cracking (IASCC) continues to cause failures in key components of both PWRs and BWRs in the US and in international reactor fleets. Identified in the 1960s, IASCC is generic in that all water reactors have exhibited susceptibility over a wide range of alloys and components. The problem will gain increasing importance in the coming years due to two factors; 1) the need for life extension to 60 years and perhaps to 80 years for the existing reactor fleet, and 2) the introduction of advanced reactors that will be subjected to the same types of environments.

The program consists of two major tasks; determination of the factors affecting crack initiation in irradiated alloys strained in high temperature water, and utilizing post-irradiation annealing as a mitigation technique for IASCC and to identify microstructure features controlling IASCC initiation. The first task focused on improving the capability to detect crack initiation and identifying the factors affecting it. This included development of the four-point bend test to determine the stress at initiation and the role of dislocation channels in the initiation process. It also includes the assessment of alloy and environment on IASCC initiation. The second part involves the use of post-irradiation annealing to narrow in on the microstructure features responsible for IASCC initiation. By isolating the PIA conditions that marks the disappearance of IASCC, a comparison of microstructures may provide some insight as to what change is responsible for the elimination of IASCC susceptibility. The outcome of this program is a firmer understanding of parameters and microstructures governing IASCC and a prescription for post-irradiation annealing that mitigates IASCC.

It is challenging to establish the capability of testing neutron-irradiated sample (especially for crack growth rate (CGR) tests) under laboratory conditions. A procedure for loading and spot welding round compact tension (RCT) sample in hot cell has been successfully established and further improved. A new test system (IM5) equipped with a AT5 based direct current potential drop (DCPD) system has been developed at UM for testing neutron-irradiated sample in a hot cell. In order to test the capability to measure crack growth rates in a hot cell, a CGR test on cold worked stainless steel (SS) has been performed according to the procedures for irradiated sample testing. At the same time, another sample from the same heat was tested with an in-house DCPD system based on LabView software to benchmark against the AT5 system. Since then, a 304L SS RCT which has been neutron irradiated to 5.9 dpa and annealed at 550 °C for 20 h (designated as RCT-3) has been tested.

The role of various alloying elements and microstructural features on the crack initiation in austenitic stainless steels in reactor environment under the presence of radiation is not properly understood. A four-point bend test setup was used to evaluate the role of various microstructural features. The system configuration was modified to improve the reliability of the system by aligning the loading fixture with the central loading axis of the system. An improvement in the stability of the cross-head is expected, thus providing more reliable sample displacement results based on the crosshead LVDT deflections. Currently the correlation between the strain and the plastic deflection induced in the material during straining is being established.

3 EXPERIMENT

3.1 Materials and specimens

3.1.1 304L from the Barsebäck 1 BWR

The neutron-irradiated samples were machined from control rod #1690 which was used in the operation of the Barsebäck 1 BWR in Sweden and withdrawn from the core for the entirety of its use. The control rod was manufactured by ASEA-ATOM (now Westinghouse Electric Sweden AB) of 304L SS by Vereinigte Edelstahlwerke AG and was delivered in a solution-annealed condition. No other heat treatments were performed during the fabrication of the control rod. The chemical composition of the material (in wt.%) was determined both by ladle analysis of the un-irradiated material during fabrication [1] and by an APT characterization of the irradiated material [2], as shown in Table 3-1. The two chemical compositions are quite comparable with the exception of impurity traces of both Al and Cu as seen in APT.

Table 3-1: Chemical compositions of the 304L SS in both un-irradiated and irradiated conditions.

Method	C	Si	Mn	Cr	Co	N	Ni	P	S	B	Al	Cu	Fe
Ladle Analysis (Unirradiated)	0.025	0.30	1.09	18.35	0.029	0.024	10.57	0.013	0.003	<0.0005	-	-	Bal.
APT (Irradiated)	0.035	0.40	1.13	18.34	0.15	-	11.6	0.02	0.001	ND	0.02	0.12	68.01

The mechanical properties of the un-irradiated material were determined via tensile experiments at both room temperature and 300°C. Several tests were completed at each temperature for which the results are shown in Table 3-2 [1].

Table 3-2: Results of tensile tests performed on un-irradiated 304L SS at both room temperature and 300 °C [1].

Tensile Test Number	Room Temperature				300°C			
	YS (MPa)	UTS (MPa)	ϵ_{tot} (%)	Z ⁽¹⁾ (%)	YS (MPa)	UTS (MPa)	ϵ_{tot} (%)	Z ⁽¹⁾ (%)
191	215	509	65	80	-	-	-	-
360	215	509	65	82	179	380	35	75
361	208	502	65	82	159	380	49	75
362	209	502	70	82	-	-	-	-
Mean	212	506	66	82	169	380	42	75

(1) Reduction of area.

A summary of the irradiation history of the control rod is shown in Table 3-3 [1]. As it was withdrawn from the core for the entirety of its use, it was exposed to a lower neutron irradiation flux than normal. The total damage accumulated is 5.9 dpa [1].

Table 3-3: Summary of the irradiation damage for control rod #1690 [1].

Control Rod ID#.	Time (hrs.)	Flux (n/cm ² •s) (E> 1 MeV)	Fluence (n/cm ²) (E> 1 MeV)	Dose (dpa)
1690	95608	1.2×10^{13}	4.16×10^{21}	5.9

Two tensile samples machined from this control rod were tensile tested in air at 288 °C. The results from the tensile tests are summarized in Table 3-4 [1]. The results show significant radiation hardening in comparison to the results from the un-irradiated samples (Table 3-2).

Table 3-4: Results of tensile tests performed in air at 288 °C on the irradiated material from control rod# 1690 [1].

Test #	YS (MPa)	UTS (MPa)	ϵ_{tot} (%)	ϵ_u (%)
1	662	662	11.3	0.04
2	683	683	11.2	0.03
Mean	672	672	11.2	0.03

3.1.1.1 Inventory of tensile bars and RCT samples

Studsvik Nuclear AB provided documentation detailing the fabrication of the irradiated specimens. A total of 5 RCT and 12 tensile samples were delivered, as well as an additional 18 smaller blanks which were left over from the fabrication of the other specimens. The samples were machined from the control rod blade as shown in Figure 3-1 [3]. The dimensions as shown in Figure 3-2a were checked for every sample and listed in Table 3-5 [3]. RCT1 has a small notch at the back due to the limited source material and the machining of sample T3. However, this defect is not expected to affect the stress intensity at the crack tip, thus the sample is still considered useable. The tensile samples were machined in accordance with the initial design, shown in Figure 3-2b and the measured dimensions are listed in Table 3-6. Although 10 tensile samples were originally ordered, 12 were included in the shipment, as T-1 and T-3 were machined incorrectly, i.e. they had misaligned flats along the gauge sections. It was also noticed that three samples had different sized flats, resulting from improper centering of the specimen during machining. T-5 was machined such that it only had a single large flat and it was also found to be slightly bent. In summary, of twelve tensile samples that were shipped, six were machined per specifications, three had flats which were not properly centered, two had misaligned flats, and one was slightly bent.

Prior to the annealing treatment of the designated tensile specimens, it was necessary to complete several cutting operations on the heads of the tensile specimens. The goal of these cutting operations was two-fold: first, the cutting removes ~50% of the specimen mass, thereby reducing the total dose rate; second, the cutting operation creates several small specimen slices that can be used to analyze the pre- and post-annealed hardness of the tensile specimens.

Annealing treatments were conducted in an air furnace at varying times and temperatures: 450 – 600°C for 1- 20 hrs. A total of twelve unique annealing conditions were performed, as well as two repeated conditions: 500°C: 1hr and 600°C: 5hr, to verify the consistency of the annealing treatment. The furnace used for the annealing treatments demonstrated a high temperature stability and there was a good agreement between the built-in and secondary thermocouple. The specimens were all removed from the furnace at ± 2 min of their desired timing. Tables 3-5 and 3-6 outline the entire list of irradiated specimens that were utilized for this project as well as their applied annealing treatments. Note that tensile specimen T-2 was not shipped as it was damaged during the production of the specimen, while T-3 was irreparably damaged whilst cutting the tensile heads from the specimens. Specimen T-8 remains as a back-up specimen in case a problem develops with one of the selected specimens.

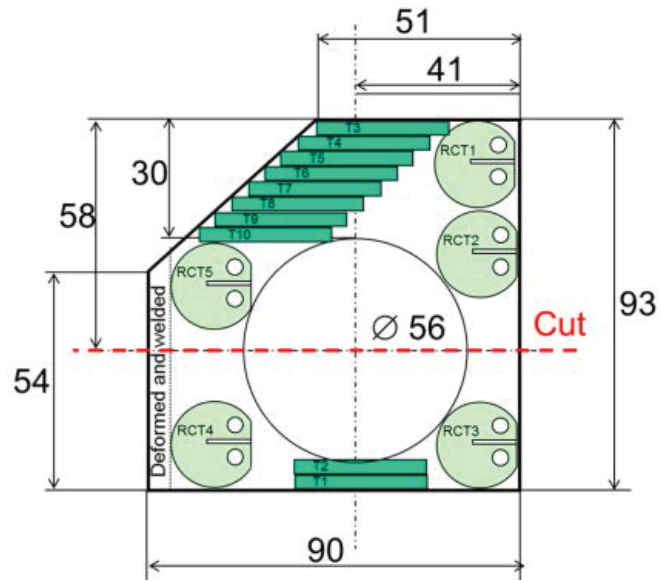
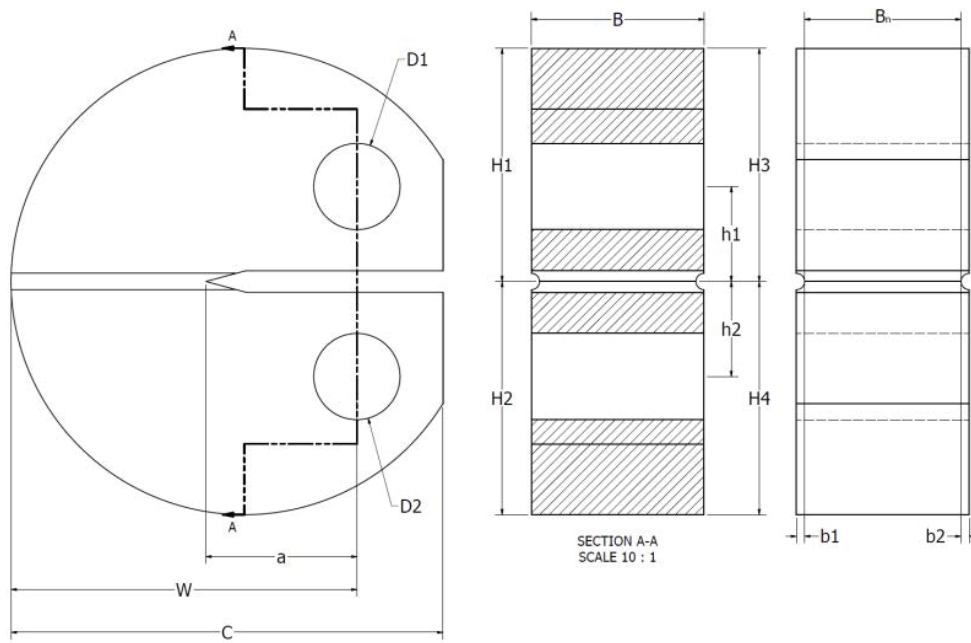
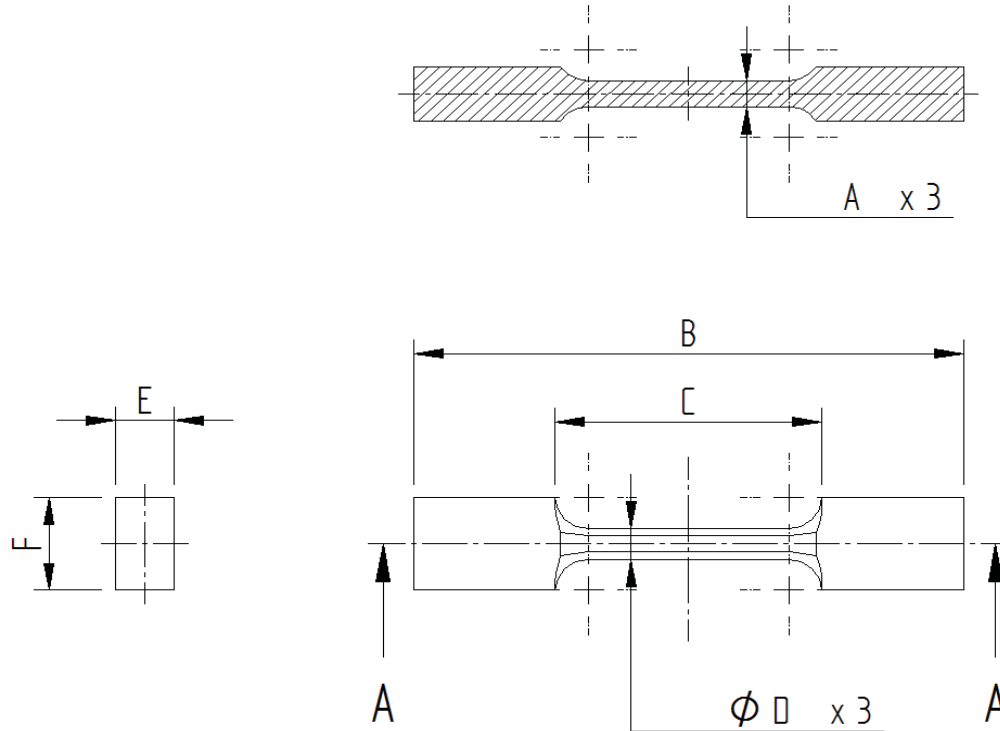


Figure 3-1: Schematic showing the locations of various samples in the control rod blade [3].



(a)



(b)

Figure 3-2: Schematic showing the dimensions checked on (a) the RCT sample and (b) the tensile bar [3].

Table 3-5: Annealing conditions and dimensions of the RCT samples [3].

Sample ID	Annealing condition	W	C	a	B	B _n	b ₁ b ₂	D ₁ D ₂	h ₁ h ₂	H ₁ H ₂	H ₃ H ₄
RCT-1	500°C 1 h	16.30	19.89	6.83	8.08	7.19	0.47 0.35	4.04 4.01	4.51 4.27	10.87 10.81	10.78 10.90
RCT-2	As-irradiated	16.14	20.03	6.43	8.05	7.36	0.43 0.32	4.06 4.13	4.43 4.38	11.19 10.45	11.23 10.42
RCT-3	550°C 20 h	16.10	19.99	6.52	8.06	7.30	0.26 0.55	4.09 4.15	4.39 4.27	10.82 10.79	10.72 10.90
RCT-4	550°C 5 h	16.03	20.02	6.33	8.05	7.31	0.37 0.40	4.15 4.04	4.33 4.47	10.75 10.92	10.72 10.97
RCT-5	550°C 1 h	16.00	19.96	6.44	8.10	7.24	0.55 0.27	4.04 4.15	4.59 4.29	10.74 10.84	10.73 10.85

Table 3-6: Annealing conditions and measured dimensions of the tensile bars and sample blanks [3].

Tensile bars							
Sample ID	Annealing condition	A	B	C	D	E	F
T1	500°C 1 h	1.63	33.07	16.16	2.00	3.51	5.96
T3	Unusable	1.44	33.06	16.11	1.90	3.47	6.01
T4	As-Irradiated	1.68	33.03	15.97	2.02	3.49	5.99
T5	500°C: 1hr	1.66	33.06	16.08	1.99	3.49	6.00
T6	As-Irradiated	1.70	33.16	16.03	2.03	3.49	6.02
T7	550°C: 5hr	1.69	33.16	16.03	2.0	3.51	6.01
T8	As-Irradiated	1.69	33.19	16.08	1.93	3.49	6.01
T9	550°C: 20hr	1.71	33.18	16.4	1.93	3.50	5.99
T10	550°C: 5hr	1.69	33.26	16.06	1.94	3.49	5.99
T11	550°C: 1hr	1.71	33.03	16.11	1.92	3.56	5.96
T12	550°C: 20hr	1.72	33.07	16.09	1.93	3.49	5.99
T13	550°C: 1hr	1.71	33.05	16.07	1.93	3.45	6.01
Sample blanks							
5	500°C: 1hr	-	-	-	-	-	-
5A	450°C: 20hr	-	-	-	-	-	-
9	450°C: 5hr	-	-	-	-	-	-
9A	450°C: 1hr	-	-	-	-	-	-
11	550°C: 1hr	-	-	-	-	-	-
11A	600°C: 20hr	-	-	-	-	-	-
15	550°C: 5hr	-	-	-	-	-	-
15A	550°C: 20hr	-	-	-	-	-	-
16	500°C: 20hr	-	-	-	-	-	-
16A	600°C: 5hr	-	-	-	-	-	-
17	600°C: 1hr	-	-	-	-	-	-
17A	600°C: 5hr	-	-	-	-	-	-
18	500°C: 1hr	-	-	-	-	-	-
18A	500°C: 5hr	-	-	-	-	-	-

3.1.1.2 Specimen Details for CIR Tensile Bars

The specimens for the tensile bars obtained from the CIR materials were in the dog bone type tensile bar specimen geometry shown in Fig 3-3 below. Each sample has a cylindrical gage section with a 2 mm diameter and 12 mm gage length. All the specimens were shoulder loaded during the CERT experiments so the pin hole of 3.1 mm diameter was inconsequential to the testing of the specimen. Shoulder loading was done to avoid premature IASCC crack initiation in the vicinity of these pin holes. Specimens were part of the batch of materials fabricated at Pacific Northwest National Laboratory (PNNL) and irradiations were performed in the BOR-60 reactor at Dimitrograd, Russia.

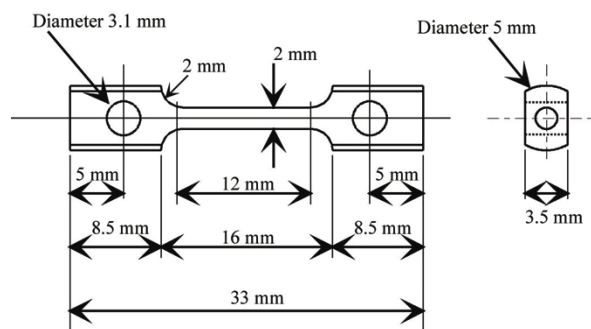


Figure 3-3. Tensile bar sample geometry and dimensions.

Irradiations were performed in liquid sodium fast reactor (BOR-60) at a temperature of 320 °C and were completed between 2001 and 2004. A neutron flux of $\sim 1.8 \times 10^{15}$ n/cm²s ($E > 0.1$ MeV) which corresponds to a damage rate of 9.4×10^{-7} dpa/s using the NRT model was used for the irradiations[4]. Total irradiation doses for the different alloying elements were between 4.4 and 47.5 dpa. Specimen designations along with the damage in the material are shown in Table 3-7 below along with the environment they were CERT tested in the laboratory. Subsequent to the irradiations, the tensile bars were shipped to Oak Ridge National Laboratory (ORNL) where the subsequent specimen preparation was performed.

Each sample was both mechanically and electrochemically polished at the ORNL Low Activity Materials Development and Analysis (LAMDA) Laboratory. Mechanical polishing was performed manually using 320 grit SiC sandpaper to remove sodium and surface contaminants left by the reactor coolant during irradiation. Each sample was then electropolished using the A2 solution supplied by Struers™ (percentages by volume: 73% ethanol, 10% ethylene glycol monobutyl ether, 9% distilled water, and 8% perchloric acid), cooled to below 20°C in an ice-bath. A potential of 30 V was applied for four 15 second periods per sample to achieve a mirror finish. The finished tensile bars were shipped to University of Michigan (UM) for crack initiation testing using the CERT systems.

Table 3-7. Irradiated tensile bar doses and test environments.

Sample ID	Material	Dose, dpa	Water Chemistry	Temperature, °C
AS13	CP304L	5.5	NWC	288
AS14	CP304L	5.5	PW	320
AS17	CP304L	10.2	NWC	288
AS18	CP304L	10.2	HWC	288
AS19	CP304L	10.2	PW	320
AS22	CP304L	47.5	NWC	288
AS23	CP304L	47.5	PW	320
SW36	CP304L	4.4	NWC	288
SW37	CP304L	4.4	PW	320
BS13	CP316	5.5	PW	320
BS16	CP316	10.2	PW	320
B123	CP316	5.4	NWC	288
B126	CP316	25.0	PW	320
BR15	CP316	4.8	PW	320
CR13	CP316+Ti	4.8	PW	320
ES14	HP304L	11.8	PW	320
ES16	HP304L	10.2	HWC	288
ES21	HP304L	10.7	NWC	288
FS13	HP304L-C	9.1	PW	320
GS13	HP304L+Mo	11.8	PW	320
GS14	HP304L+Mo	11.8	NWC	288
HS13	HP304L+Si	7.8	NWC	288
KS13	HP304L+Ni	9.6	NWC	288
LS13	HP304L+Ni,Cr	9.1	NWC	288
PS13	HP316L+Hf	9.6	PW	320
PS14	HP316L+Hf	9.6	HWC	288
PS15	HP316L+Hf	9.6	NWC	288

3.1.2 Neutron irradiated tensile bars from CIR project

In addition to the material mentioned previously, crack initiation studies have also been carried out on the materials shown in Table 3-8 below. A total of eleven different stainless steel alloys were obtained in neutron irradiated condition. Among these alloys four alloys are in commercial purity (CP) standard while seven different alloys were fabricated from high purity (HP) 304L stainless steel alloy with minor modification to the minor alloying elements.

Table 3-8: Chemical compositions of tested alloys in wt %. Alloys FS-PS have the solute addition element indicated in bold.

Heat ID	Material	Fe	Cr	Ni	Mn	Mo	Si	N	C	P	S	Nb	Ti	O	Co	Cu	Hf
AS	SA CP 304L	bal.	19.95	10.80	1.82	0.53	0.56	0.0720	0.023	0.023	0.015	<0.001	0.02	0.011	0.22	0.29	-
BS/BR/B	SA/CW/CW CP 316	bal.	16.84	10.54	1.13	2.25	0.73	0.0210	0.056	0.022	0.022	0.008	0.01	0.009	0.12	0.25	-
CR	CW CP 316+Ti	bal.	16.77	12.78	1.40	2.18	0.56	0.0080	0.070	0.013	0.007	<0.010	0.38	0.004	0.10	0.06	-
SW	SA CP 304L	bal.	18.42	10.45	1.07	0.00	0.24	0.0250	0.022	0.015	<0.002	-	-	-	-	-	-
ES	SA HP 304L	bal.	18.76	12.37	0.94	0.04	0.04	0.0003	0.021	<0.010	0.003	0.005	0.01	0.004	0.01	<0.01	-
FS	SA HP 304L (-C)	bal.	18.17	12.06	0.98	0.02	0.03	0.0005	0.008	<0.010	0.003	0.002	0.01	0.013	0.01	<0.01	-
GS	SA HP 304L (+Mo)	bal.	18.26	12.15	0.97	2.36	0.03	0.0004	0.020	<0.010	0.002	0.003	0.01	0.005	0.01	0.01	-
HS	SA HP 304L (+Si)	bal.	18.17	12.45	1.01	0.02	1.05	0.0005	0.020	<0.010	0.002	<0.001	0.01	0.007	0.01	<0.01	-
KS	SA HP 304L (+Ni)	bal.	18.21	25.08	1.00	0.02	0.03	0.0005	0.020	<0.010	0.002	<0.001	0.01	0.003	0.01	<0.01	-
LS	SA HP 304L (Cr+Ni)	bal.	25.22	25.07	1.02	0.02	0.03	0.0005	0.020	<0.010	0.002	<0.001	0.01	0.009	0.01	0.01	-
PS	SA HP 316L (+Hf)	bal.	17.03	13.60	1.01	2.18	0.10	-	0.028	0.010	0.007	-	-	-	-	-	1.17

The CP alloys have designations A,B,C and SW. Alloy A was a type 304L SS alloy representative of a LWR core shroud material while alloy B was type 316 SS alloy representative of the PWR baffle bolt material. In addition to these material, alloy C is a modified type 316L+Ti material, alloy SW was a Swedish heat of type 304L SS alloy, similar to the alloy A. For the alloy designation scheme shown in Table 3-5, the second letter denotes the condition of the material. The designation S refers to the material being in a solution annealed condition with the solution annealing being done at 1050 °C for 0.5 h followed by water quenching. The designation R represents the material being in a cold worked condition prior to testing. The amount of cold worked was not reported for these materials[5].

In addition to these commercial purity alloys, seven HP alloys with designations E,F, G,H,K,L and P were used for the crack initiation study. Alloy E was used as the model base alloy for the fabrication of alloys G,H,K and L. Alloys G, H,K and L were remelted for the purpose of solute addition of Mo,Si,Ni and Ni+Cr respectively while alloy F was created from a separate starting material to produce an alloy with low carbon content. Alloy P was produced from a type 316 SS base alloy with hafnium (Hf) as the oversized solute addition. All these alloys were given a cold rolling treatment followed by solution annealing between 900 °C and 1200 °C for a duration of 0.5 to 1 h in between cold rolling passes in order to get a final ASTM grain size between 6 and 8 for each condition[5].

3.2 Microstructure Characterization

This section outlines the preparation procedure and analysis techniques used for the microstructural characterization of the material for this research project, including micro-hardness, TEM, and APT analysis.

3.2.1 Micro-Hardness Measurement

Micro-hardness measurements were made to evaluate the bulk changes in the irradiated microstructure. Prior to the measurements, specimens were mechanically polished with a succession of finer grits, ending with a 3 μm diamond polish. The specimens were then electropolished at room temperature for 15 seconds at 30V using a commercial Struers A2 solution at LAMDA at ORNL. After electropolishing, provided no defects or scratches were observed, the specimens were measured using a Vickers micro-hardness indenter at ORNL. Each specimen was measured at a load of 200 gf, with at least 30 independent indents, whereas the average hardness and standard deviation was then recorded.

3.2.2 Transmission Electron Microscopy (TEM) Analysis

TEM foils for dislocation loop analysis were prepared by jet thinning using a commercial Struers A2 solution at LAMDA at ORNL. Dislocation loops were examined with rel-rod technique using JEOL 2100 STEM at ORNL. Dislocation loops were imaged at different locations under the [110] zone axis. The number of loops characterized is dependent on the number density and in the as-irradiated condition, 670 loops were characterized.

Radiation induced segregation (RIS) was assessed in the BWR irradiate 304L SS. Energy-dispersive X-ray Spectroscopy (EDS) maps were taken from random high angle boundary using the FEI F200X S/TEM instrument equipped with ChemiSTEM at the LAMDA laboratory at ORNL using a map size of 1024x1024 pixels with a resolution of ~0.23 nm/pixels with a probe full width half max of ~1.5nm. Each scan had a duration of one hour with more than 100,000 counts/sec with dead times from 1-6%. ChemiSTEM has a much higher counting rate compared to typical STEM/EDS. The qualitative x-rays counts were converted to quantified weight percentages using the Bruker Esprit© 1.9 software package, which uses the Cliff-Lorimer calculations for each pixel. Prior to EDS measurement, grain boundary was tilted to edge-on position. Due to the limitation of available grain boundaries for RIS, only one grain boundary was measured, but multiple EDS maps were obtained from different segments of the grain boundary.

3.2.3 Atom Probe Tomography (APT) Analysis

Precipitates/solute clusters were analyzed using the atom probe tomography. Needle-shaped APT specimens were prepared by the standard lift-out method and focused ion beam milling using Quanta 3D at ORNL. Specimens were prepared from the electropolished 3-mm disk. Prior to the lift-out procedure, Pt was deposited to protect the material from ion beam damage. A final 5 kV clean-up procedure was utilized to minimize the Ga damaged regions and reduce the tip radius to ~50 nm. About 6 APT tips were made for each condition for APT analysis. APT specimens were analyzed using a LEAP-4000XHR microscope at the University of Michigan operated in electrical mode with a voltage pulse fraction of 20%. Specimen temperature was maintained at 50 K and detection rate was kept constant at 0.005 atom/pulse.

Reconstruction of the relative atom positions from the raw data was performed using the commercial software, IVAS 3.6.4 from CAMECA™. Solute clusters were analyzed using the maximum separation method. The nickel-silicon solute clusters were defined by the tenth order spacing of silicon atoms, whereas a suitable d_{\max} (the maximum separation of tenth-order silicon atoms) was selected to best separate the clustered and randomized silicon atoms based on the spacing distribution. The aluminum-copper solute clusters were defined by the fourth order spacing of aluminum atoms, whereas a suitable d_{\max} (the maximum separation of fourth-order aluminum atoms) was selected to best separate the clustered and randomized aluminum atoms based on the spacing distribution. For both cluster types an N_{\min} was chosen equal to the maximum cluster size observed for a randomized experimental APT data.

3.3 Test systems and procedures

3.3.1 Test Systems

The CERT and CGR testing systems, IM1 and IM5 are located in the Irradiated Materials Testing Laboratory (IMTL) at the University of Michigan. Both systems have similar water loops and a schematic diagram is shown in Figure 3-4. The water loop consists of two sub loops, loop 1 for conditioning the water at room temperature and low pressure, and loop 2 for simulating light water reactor environment at high temperature and high pressure. The environmental parameters such as conductivity and dissolved gas concentration can be well controlled and maintained in loop 1. The water then is pressured and heated in loop 2, reaching the desired temperature and pressure in the autoclave and recirculates to the primary water column. The conductivity and dissolved oxygen (DO) are monitored at both inlet and outlet.

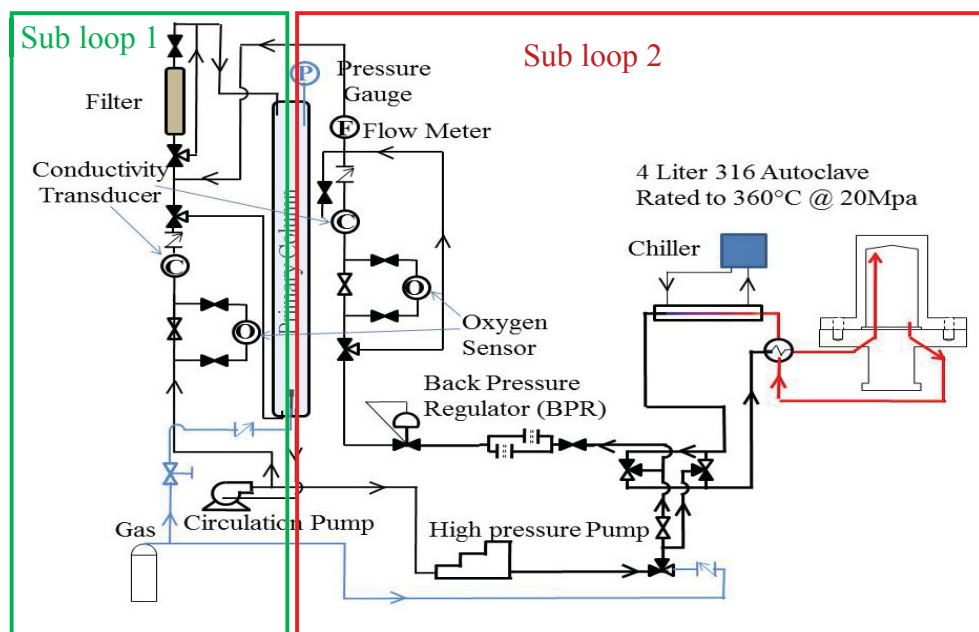


Figure 3-4: Schematic of the water loop used in the IM systems in the Irradiated Materials Testing Laboratory.

The CERT experiments were performed using the IM1 autoclave system. The IM1 loading system can strain up to four tensile bar samples simultaneously, while the application of load is controlled using a 50 kN servo motor. A linear variable differential transformer (LVDT) is mounted on the crosshead to measure displacement. Four pull rods connect each sample to the crosshead, and load is measured on each by a load cell. Each of the pull rods are sealed at the feed-through into the autoclave with a self-energizing graphite seal with an internal spring that expands under pressure. Each pull rod is connected to an Inconel 625 sample loading fixture. Electrical insulation is provided by zirconia washers located in the loading fixtures. After loading the specimen, the autoclave body is sealed to the autoclave head, thus preventing any leakage during the experiment.

CGR tests were performed on IM5. A AT5 based DCPD system was used to control K value and monitor the crack growth rate. Due to the planned testing of highly irradiated specimens, IM5 was

designed as a mobile system, such that an experiment could be performed in a hot cell, thus significantly reducing the dose to the operator. The wires were spot welded onto the neutron-irradiated RCT sample in hot cell #1 using manipulator arms. Then the whole autoclave containing the RCT sample was moved to hot cell #2 for testing. The water loops and controls remained outside of the hot cell for easy access during the experiment. Figure 3-5 shows the images of test system.

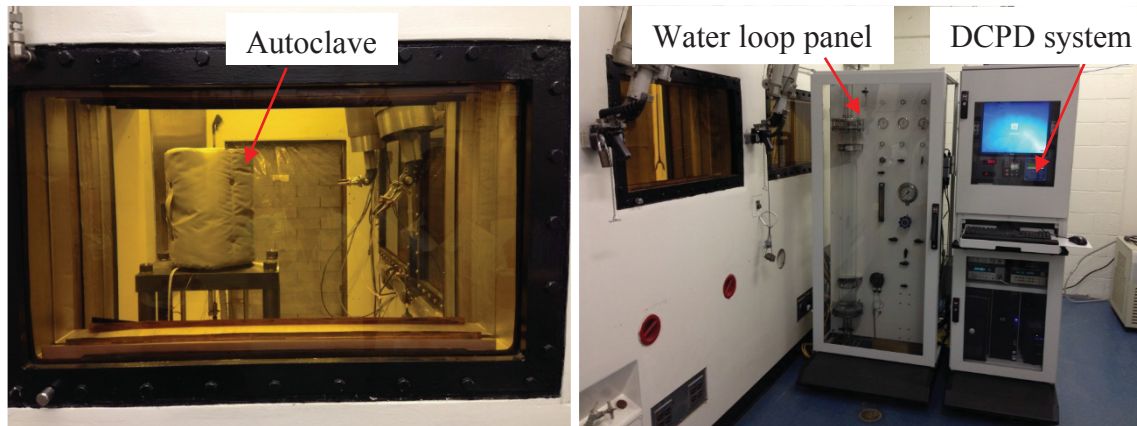


Figure 3-5: Images of CGR test system IM5 in hot cell #2.

Crack growth is measured using the reversed Direct Current Potential Drop (DCPD) technique, which has long been used for crack growth monitoring in high temperature water environments. As shown in Figure 3-6 (a), the DCPD monitoring system consists of a DC power supply, relays, nano-volt meter, data acquisition unit, servomotor and Skala controller. DC power supply provides stable current (2.5 A for 0.5 T RCT) to a specimen. The current is reversed once per second through solid-state relays to correct for thermocouple effects. The current and potential probe leads were spot-welded to the RCT specimen as shown in Figure 3-6 (b). The potential drop resulting from crack propagation in the specimen is measured by a nano-volt meter. Data acquisition and instrument control are all integrated in a DOS program called AT5, which was developed by Dr. Peter Andresen at GE Global Research Center. A data processing program called HIKO, based on DotNetFX2.0 was also used in this study.

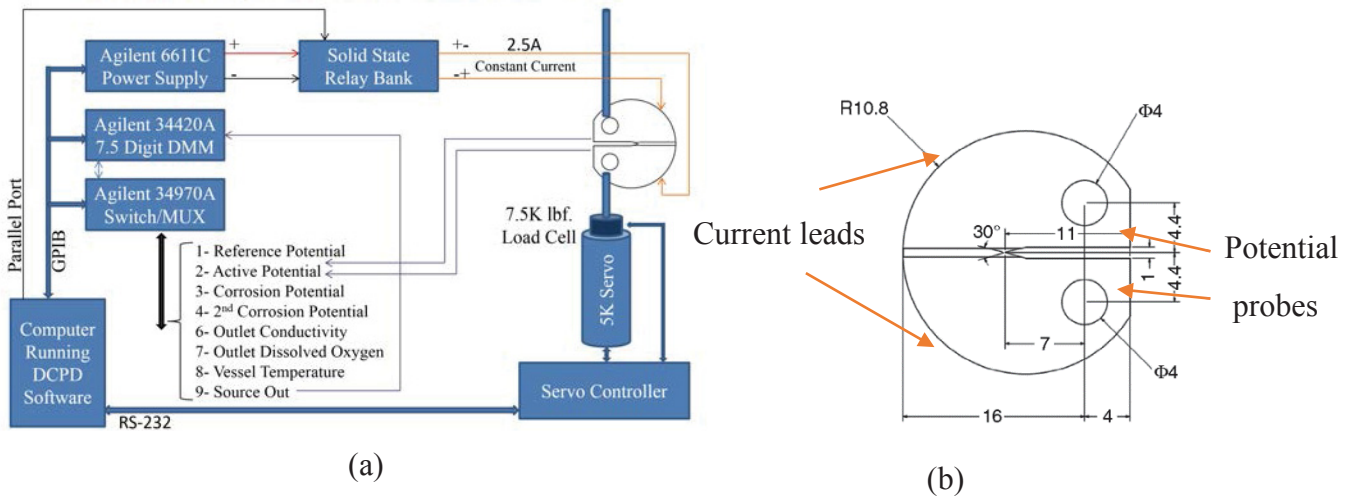


Figure 3-6: DCPD monitoring system. (a) a schematic of DCPD instrumentation and circuit diagram, and (b) arrangement of current and potential probe leads on the 0.5T RCT specimen.

3.3.2 Procedures for CERT test

Tensile Bar Annealing and Surface Preparation

During the original machining of the tensile specimens, EDM was used to cut the parallel flats on opposing sides of the gauge section. EDM leaves a thin, amorphous recast layer, which may affect the cracking behavior in a simulated-BWR environment. As such, the gauge section flats were mechanically polished to remove the EDM recast layer. However, due to the high radioactivity of the specimens this procedure was completed in a hot cell facility using manipulators. First both tensile heads were clamped in a small vice, to prevent any possible bending, and a polishing paper was carefully moved across the gauge flats. An 800-grit sandpaper was first utilized to remove the EDM layer, while a 1200 grit paper was utilized to prepare the surface for a later electropolishing. The removal of the EDM layer was monitored by the usage of an in-cell camera.

Electropolishing was completed on the tensile specimens to create a final surface finish satisfactory for CERT testing in both simulated BWR environment and an inert argon environment. The electropolishing operation was completed in the hot cell fume hood. The electropolishing setup was quite simple, consisting of a power supply, a grid cathode, and a beaker of electrolyte. The electropolishing will be completed at 30V and consist of four 15 sec polishing increments, between each of which the specimen was flipped. This procedure was expected to remove roughly 20 μ m of material based on prior experiments. After electropolishing, each specimen was observed via an optical microscope to verify a good surface quality.

CERT Test: PIA conditions

All CERT tests were conducted in a simulated boiling water reactor normal water chemistry (BWR-NWC) environment. During each straining, system water pressure was maintained at 9.7

MPa and temperature at 288°C, while the outlet dissolved oxygen and conductivity were maintained at 200 ppb and 0.2 μS/cm, respectively.

After pressurizing and achieving the desired temperature for the intended environment, the system was allowed to stabilize until conductivity had dropped below 0.2 μS/cm. Upon stabilization, dissolved gas was added to the primary water column and the target concentration was achieved by regulating the main column pressure with a back-pressure regulator. Following gas addition, the environment stabilized for an additional period of 12 hr before commencing straining. During this 12 hr period, conductivity control was initiated once the vessel outlet water conductivity again dropped below 0.2 μS/cm. Conductivity was controlled by adding small amounts of dilute H₂SO₄ to the primary water column with the peristaltic pump, which was set to operate whenever conductivity would decrease below the target.

Due to the crosshead arrangement, non-irradiated type 304L SS tensile bar specimens were used to occupy empty loading locations and provide load balance at the system crosshead. These specimens were selected such that they would have a similar yield stress as the irradiated specimen currently being examined.

Prior to straining, a preload of 20 MPa was applied to each specimen. At the end of the 12 h stabilization period, straining was initiated by moving the crosshead with the servo motor. This displacement was completed at a rate of 1.65×10^{-7} in/s for all experiments, which corresponds to a strain rate of $3.0 \times 10^{-7} \text{ s}^{-1}$. During the stabilization period and straining, all environmental and stress-strain data were recorded every 30 sec using the LabView data acquisition program. Recorded data includes inlet and outlet water pressure, inlet and outlet water conductivity, outlet dissolved oxygen, vessel internal and preheater temperature, LVDT displacement, and load cell readings.

Dislocation Channeling Characterization

As previously discussed, increased localized deformation has been closely correlated with increasing cracking susceptibility [6], however, the change in localized deformation following PIA treatments has never been measured for a neutron-irradiated material. Furthermore, more recent studies [7-9], have indicated that dislocation channels that are discontinuous at grain boundaries have a higher propensity of crack initiation.

For this study, the straining experiments were completed in small increments, such that we could more precisely identify point of crack initiation and study the development of the localized deformation. After each stress/strain increment the specimen was removed from the autoclave were examined using a JEOL JSM-6480 SEM. This examination measured any plastic strain through fiducial markings, as well as recording any sites of crack initiation and changes in the localized deformation.

The dislocation channel characterization focused on the changes in both the total dislocation channel density and the density of channel-grain boundary interaction sites, which were characterized as either continuous or discontinuous, with increasing plastic strain. A continuous channel-grain boundary site refers to a location where a dislocation channel intersects a grain boundary, but is then able to transmit across said boundary, i.e. a continuous channel. Whereas a discontinuous interaction site is one where the channel was unable to transmit across the grain boundary. By comparing the densities of both continuous and discontinuous interaction sites, one can measure the relative propensity that a condition has for forming continuous channels, and how this may change with both PIA treatments and additional strain.

Following each stress/strain increment both the gage flats were fully imaged at 500x magnification. This magnification allowed for a balance of both image quality and imaging time, while the large-scale imaging also allows for a spatial correlation of images following each additional strain increment.

Post-Failure Fractography

Fractography was performed on each fractured tensile bar following the CERT test using a JEOL JSM-6480 SEM. Low magnification images of the gage surface were taken to indicate the locations of IG fracture and secondary cracking, while the edges of the main IG crack and secondary cracks were imaged at higher magnification.

The fracture surfaces of each specimen were also examined in detail to characterize the nature of failure by viewing the fracture surface perpendicular to the tensile axis. Regions of intergranular (IG), transgranular (TG), mixed (IG+TG), and ductile type failure were identified. Higher magnification imaging (>500x) was performed in regions of note.

Reduction of area (RA) was calculated using the total area of the fracture surface viewed perpendicular to the tensile axis. The area of the fracture surface was determined using the Image J™ imaging software program particle analysis feature. The RA is determined by dividing the difference in area between the fractured surface (viewed parallel to the tensile direction) and the original cross-sectional area by the original cross sectional area.

Regions of IG, mixed, TG, and ductile fracture were characterized by area and expressed as an area-based percentage of the total fracture surface. Using the Image J™ program, each fracture regions were cut from the overall view of the fracture surface and area was determined using the particle analysis feature of the software. This area divided by the total fracture surface area yielded the percentage of fracture type.

CERT Testing: BOR-60 Neutron Irradiated Conditions

To evaluate the crack initiation behavior in the four commercial purity (CP) and seven high purity (HP) alloys irradiated in BOR-60 reactor, CERT tests were performed in IM1 system described previously using a strain rate of 3.5×10^{-7} /s until fracture. Specimens were tested in one of three following environmental conditions: Normal water chemistry (NWC), hydrogen water chemistry (HWC) or Primary water chemistry (PWR). Details of the specimen tensile bar and the corresponding testing environment are described in Table 3-8 above. Test conditions and the strain rates were similar to those used in the CIR program previously [10-11]. Environmental conditions were stabilized for durations up to 72 hours prior to start of straining. The water conductivity in the NWC and the HWC environments were maintained by addition of dilute H₂SO₄ solution to the main column water reservoir. The PWR conditions were maintained by addition of Boric acid and lithium hydroxide to the solution. Pressure, dissolved oxygen and water conductivity were monitored at the autoclave inlet and outlet, and the internal autoclave temperature was monitored by thermocouple. The summary of the three different environmental conditions are shown in Table 3-9 below.

The loading system was capable of straining up to 4 samples simultaneously. Load was applied by a computer driven 50 kN servo motor connected to the loading crosshead. Crosshead displacement was measured by a Solartron linear voltage displacement transducer (LVDT). Interactive Instruments load cells mounted on each pull rod monitored load on each sample. Load, displacement, and environmental data were collected and recorded by computer data acquisition once every sixty seconds.

Table 3-9: Environmental parameters for CERT tests.

Parameter	NWC	HWC	PW
Temperature	288°C	288°C	320°C
Autoclave Pressure	10.3 MPa	10.3 MPa	12.4 MPa
Conductivity	0.2 μS/cm	0.2 μS/cm	20.5 μS/cm
O ₂ concentration	2000 ppb	0 ppb	0 ppb
H ₂ concentration	-	100 ppb	3000 ppb
B concentration	-	-	1000 ppm
Li concentration	-	-	2 ppm
ECP (SHE)	215 mV _{SHE}	-570 mV _{SHE}	-860 mV _{SHE}

Data Analysis and Fractography

Plots of stress versus strain were created using load and displacement data, to compare mechanical properties such as the yield stress, max stress, uniform elongation, and total elongation. Before analysis, stress and strain data were adjusted to account for system pressure and pull rod compliance, respectively.

A tare pressure was added to each load cell measurement, by calculating the load due to the autoclave pressure acting on the cross sectional area of each 3/16" diameter pull rod. Tare pressure on the 2 mm diameter tensile bar samples was 58.6 in NWC and HWC and 70.3 MPa in PW.

To adjust for system compliance, each raw stress-strain curve was plotted to determine the apparent modulus, generally ~10,000 MPa. LVDT displacement values were converted to strain by dividing the change in displacement by the original specimen gage section length (12 mm). The apparent modulus was determined by fitting a linear trend line to the linear elastic region of the curve. A good linear fit (i.e., R^2 value minimum of 0.999) was created by eliminating data points at low stress. The apparent modulus was then divided by the reported modulus for type 304 SS at 300°C, 166.3 GPa [12]. The resulting ratio was multiplied by the apparent strain up to the yield point. This method is consistent with the general definition of compliance: the change in length due to compliance is equal to the observed change in length less the force divided by the system stiffness, where stiffness is equal to either: force divided by the change in length, or system modulus multiplied by cross sectional area divided by original length [13]. Plastic strain data was resumed from the yield point by subtracting the difference in strain between the original strain and the compliance corrected strain. No compliance correction was made during plastic elongation.

After correcting for system pressure and compliance, several measures were determined from the stress-strain curve. Yield stress (YS) was recorded as the 0.2% offset YS if strain hardening was observed. In the case of strain softening, YS was taken to be the maximum stress. Uniform elongation (UE) was recorded as the strain at which the maximum stress occurred. Total elongation (TE) was recorded as the strain at the point of fracture.

Fractography was performed on each tensile bar following CERT testing using a JEOL JSM-6480 SEM. Gage and fracture surfaces were examined in detail, and the nature of failure was characterized. Samples showed combinations of ductile, intergranular (IG), transgranular (TG), or mixed mode IG/TG type cracking. Reduction of area (RA) was calculated by dividing the difference between the fracture surface area and the original cross-sectional area by the original cross-sectional area. Areas of IG, TG or mixed IG/TG fracture were determined and expressed as an area-based percentage of the total fracture surface.

3.3.3 Procedures for CGR Test

Test steps

RCT-3 and RCT-2 were tested first. The main purpose of the CGR test is to determine the K dependence of the CGR rate and the effect of environment change on CGR. The post-irradiation heat treatment of sample RCT-3 was expected to have removed much of the damage due to irradiation in reactor. To compare the SCC susceptibility between different samples, it is desirable to test them at same K levels. Meanwhile, the applied K values should meet the validity criterion. Based on the K validity check of as-irradiated sample and RCT3, three K values were selected: 18, 14, 11 MPa.m^{1/2}. The test steps are listed in Table 3-10.

Table 3-10: Test steps on RCT-3.

Step	Water chemistry	Stress intensity (MPa.m ^{1/2})	Waveform	Intended crack growth (mm)
1	BWR NWC	18	Precracking Haversine to Trapezoid	~0.8
2	BWR NWC	18	Constant K	0.09
3	BWR HWC	18	Constant K	0.09
4	BWR NWC	18	Constant K	0.09
5	BWR NWC	18 to 14	K transition Trapezoid	0.128
6	BWR NWC	14	Constant K	0.09
7	BWR NWC	14 to 11	K transition Trapezoid	0.128
8	BWR NWC	11	Constant K	0.09
9	BWR HWC	11	Constant K	0.09

Sample welding

Due to the annealing treatment, a slight oxide layer was developed on the RCT specimens that underwent PIA treatments, despite the stainless steel oxygen getter that was utilized. This oxide layer would be detrimental to the later spot welding of the DCPD probes, thus it had to be removed via mechanical polishing. In the hot cells at ORNL the RCT specimens were mounted onto a specially designed apparatus, which slowly rotated the specimens while an 800-grit sandpaper was applied to the specimen; special attention was also given to front flats of the RCT specimens. After completing the annealing and mechanical polishing the RCT specimens were shipped to the University of Michigan.

The current leads and potential probes need to be spot welded onto the RCT sample for DCPD measurement. Due to the radioactivity of neutron-irradiated samples, the welding should be implemented in hot cell via manipulator. A set of tools and welding procedure have been successfully developed in this lab and described in ref. [14]. Figure 3-7 shows the RCT sample after spot welding.

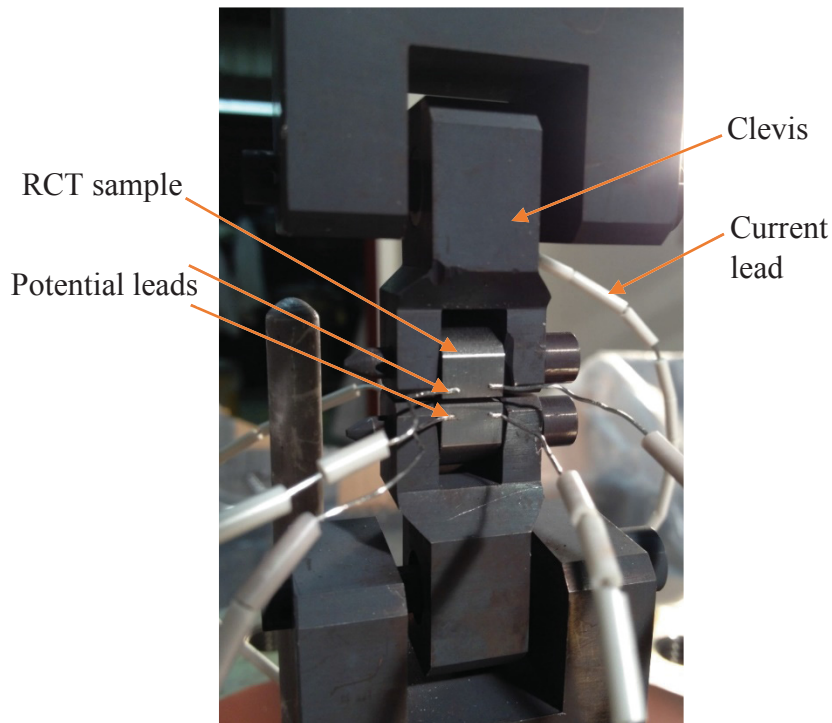


Figure 3-7: RCT sample spot welded in a hot cell.

Water Chemistry

The CGR tests were conducted in a BWR environment, including both NWC and HWC. Temperature and pressure of the testing system are 288°C and 10.3 MPa, respectively. The water in a 3.5 L autoclave was refreshed three times per hour with a flow rate of 180 mL/min. The conductivity of inlet DI water was 0.056 $\mu\text{S}/\text{cm}$. Inlet dissolved oxygen (DO) was controlled at 2

ppm for NWC and dissolved hydrogen (DH) was controlled at about 100 ppb for HWC. DO and conductivity of inlet/outlet water were continuously monitored using a Thornton 770Max DO/conductivity meter.

ECP Measurement

The electrochemical potential (ECP) of the RCT specimen was continuously monitored during the CGR test with a Cu/Cu₂O reference electrode. At the same time, the redox potential between a 1.0 cm² Pt flag and the reference electrode was also recorded. The measured potentials were converted to potentials vs standard hydrogen electrode (SHE) by subtracting 0.273 V.

Reset Initial a/W in High Temperature Water

During heating, the DCPD potential reading increases with temperature because of the increasing resistivity of metallic materials. So the initial a/W from direct measurement should be recorded and reset in AT5 once the DCPD potentials stabilize at the target temperature. If a fatigue pre-crack was introduced in air at room temperature before beginning a stress corrosion cracking test in high temperature water. In this case, the final a/W in air needs to be set as the initial a/W prior to growing the crack at the testing temperature.

Crack Growth Procedures

After loading the sample in the autoclave, the current and potential wires for DCPD measurement were spot-welded to the CT specimen as described above. The wires in each pair were twisted in order to cancel magnetic fields. A thermocouple for temperature control was placed at the crack plane in the autoclave. Once the temperature, conductivity and DO reached the target levels, the a/W for DCPD readings was reset to the initial value. After that, the system was ready for controlling K on sample. The tare load from high pressure water was accounted for in AT5. The temperature inside the autoclave, inlet and outlet pressure, inlet and outlet conductivity, and inlet and outlet DO were continuously recorded during the entire test period.

The crack was introduced by fatigue in the testing environment, i.e. in-situ fatigue pre-cracking. The maximum K was slightly lower than that being used for the next constant K step. At the beginning of the test, low loading ratio R and high frequency ν , were used to introduce cracking at a fast rate and to leave beach marks as evidence on the fracture surface for tracking the crack growth in each step. Later, in order to initiate the transition from TG to IG, R was decreased to 0.6 and the frequency was lowered from 0.1 to 0.01, and then to 0.001 Hz. After 0.001 Hz fatigue, a trapezoidal load form was applied with a holding time of 9000 seconds. Subsequently, constant K control was started.

Auto K control

The CGR is highly dependent on the K value applied on the sample. In order to reliably measure the CGR at a certain K value, the value of K should be kept constant during the test. According to ASME standard E-399 [15], K is a function of a/W and load:

$$K = \frac{P}{B\sqrt{W}} f\left(\frac{a}{W}\right) \quad (3-1)$$

where:

$$f\left(\frac{a}{W}\right) = \frac{\left(2 + \frac{a}{W}\right)\left[0.76 + 4.8\frac{a}{W} - 11.58\left(\frac{a}{W}\right)^2 + 11.43\left(\frac{a}{W}\right)^3 - 4.08\left(\frac{a}{W}\right)^4\right]}{\left(1 - \frac{a}{W}\right)^{3/2}} \quad (3-2)$$

For which: P is applied force, B is the sample thickness, W is the sample width and a is the crack length. So, the applied force should be adjusted as the crack grows to maintain a constant K . AT5 has a built-in auto K control function which can adjust the applied load automatically once a minimum increase (set to 0.01 in the program) in a/W is achieved.

dK/da Control

It is very important to sustain the crack growth when changing the K value. dK/da control was used for a smooth transition for both increasing and decreasing K . Trapezoid waveform was normally used during K transition. AT5 also has this built-in function. The rate of change of K with a (dK/da) is calculated based on the a/W and K values from two neighboring steps. As the crack grows, a change in K will be made based on the dK/da value. The threshold for the K change was set to $0.02 \text{ ksi} \cdot \text{in}^{1/2}$.

Post-test Cracking

When the test was finished in high temperature water, the autoclave system was cooled to room temperature. The sample was cracked open at room temperature using fatigue loading. To minimize the plastic deformation at the crack front formed in the environment, K was controlled at the same (or lower) level as that last used in high temperature water. After growing a post-test crack about 0.5 mm, the RCT specimen was fatigued at high frequency until it broke.

Fractography

The fracture surfaces of the RCT specimen were observed using both optical microscopy and scanning electron microscopy (SEM). The fracture surface was examined for evidence of each step change during the crack growth rate test. The morphology of the fracture surface, especially transgranular (TG) or intergranular (IG) features and the transition between TG and IG, was confirmed. The area of intergranular fracture surface was measured and divided by the width of sample to calculate the average length of IGSCC growth. The measured crack length from fracture surface analysis was compared with that from DCPD measurement. The ratio between them was used as correction factor for crack length and crack growth rate.

K correction after CGR test

As the real crack length is not exactly the same as the DCPD measured value, neither is the real K the same as the nominal K . After CGR test, the crack length was corrected using the correction factor mentioned above. K was recalculated using the corrected crack length and the applied load which was recorded in the data file.

3.3.4 Crack Initiation Studies using Four Point Bend Test

In order to study the crack initiation behavior of the neutron irradiated alloys, a modified four point bend test was used as the testing method. The specimens used for the testing was obtained from cutting a slice from the neutron irradiated tensile bars previously tested in the CERT system. The schematic of the specimen dimensions and the location from which the slices were made are shown in Figure 3-8 below.

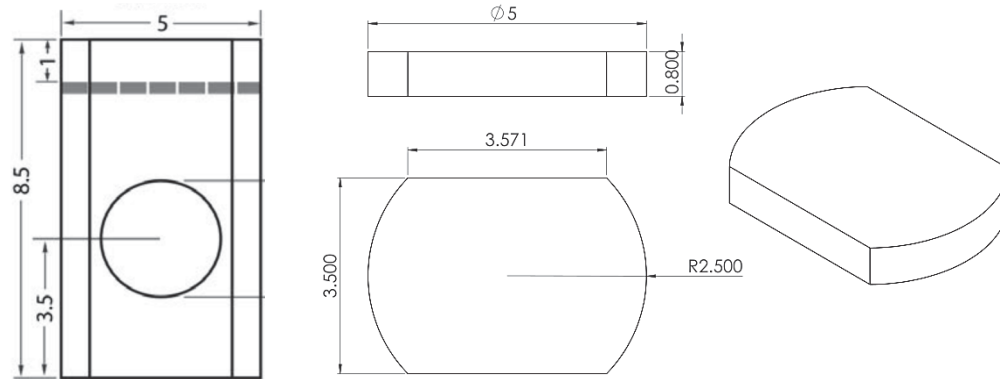


Figure 3-8 Schematic showing a) tensile bar head dimensions and location of the cut (gray dashed line), and b) bend test sample dimensions.

Prior to testing, the four point bend test specimens were created from the shoulder region of the tensile bar in the LAMDA facility at ORNL. 1 mm thick slices were generated and were mechanically polished on both sides to create a specimen with a thickness of about 800 μm . A Buehler Mini-met 1000™ sample grinding and polishing tool performed sample thinning to a target thickness of 800 μm ; using 120, 180, and 340 grit SiC grinding paper on each side to achieve a uniform thickness (final thickness variations were less than 10 μm). One side of each sample was then polished with a 3 μm nylon pad in the Mini-met™ and subsequently electrochemically polished in a Struers LectroPol-5™ system. One electrochemical polishing step was applied for 15 sec at a potential of 30 V at 20 °C in the A2 solution. Parent tensile bar ID, bend sample ID, dose, and thickness are presented in Table 3-11.

Table 3-11. Samples for four-point bend SCC testing.

Parent Tensile Sample ID	Bend Sample ID	Dose, dpa	Thickness, μm
AS14	AS01	5.5	825
AS14	AS02	5.5	797
AS14	AS03	5.5	839
AS14	AS04	5.5	801
AS18	AS05	10.2	813
AS19	AS06	10.2	796
AS17	AS07	10.2	791
AS19	AS08	10.2	777
AS22	AS09	47.5	807
AS23	AS10	47.5	729
ES16	ES01	10.2	770
ES21	ES02	10.7	804
KS13	KS01	9.6	806
KS13	KS02	9.6	815

Finite element analysis

Prior to testing of the neutron irradiated specimens, finite element analysis was done to optimize the loading fixture and estimate the stress strain distribution on the specimen surface. The details of the FEA analysis have been reported previously [16]

To benchmark the four point bend test setup and evaluate the strain profile on the specimen surface, two different material conditions were used in the simulation, based on the tensile stress-strain behavior of the unirradiated AR and 16.9% cold worked 304L SS alloy. The modulus of these two conditions was that for type 304 SS at 300 °C, 166.3 GPa, the Poisson's ratio was 0.3, and the density was 7.8 g/cm³. The respective yield strength and strain hardening coefficients were determined from tensile tests performed in the IM1 test system. The AR and 16.9% CW conditions had yield strength of 445 and 783 MPa, and strain hardening coefficients 4.5 and 7.5 GPa, respectively. The final loading geometry was designed to create a uniaxial tensile strain of up to 3%, which is sufficient to induce cracking in neutron irradiated austenitic stainless steels.

The four point bend test fixture used for the study was fabricated from Inconel 718 alloy in accordance with the schematic shown in Figure 3-9 below. Heat treatment of fixture parts, post-fabrication, created a hardened state to prevent deformation during bend testing. Four round posts called centering pins, elevated from the bottom support surface by 0.4 mm, surrounded the sample to prevent rotational misalignment. Set screws to the left and right of the sample centered the sample laterally, and were retracted after pre-loading the sample to prevent constraint during bending. The loading fixture was electrically insulated with zirconia washers in the connection between the pull rod and the loading fixture.

Bend samples were loaded into the fixture with tweezers, centered by tightening the set screws on either side, and held in place by bringing the loading points into contact with the sample and lightly tightening the connection of the pull rod at the system crosshead.

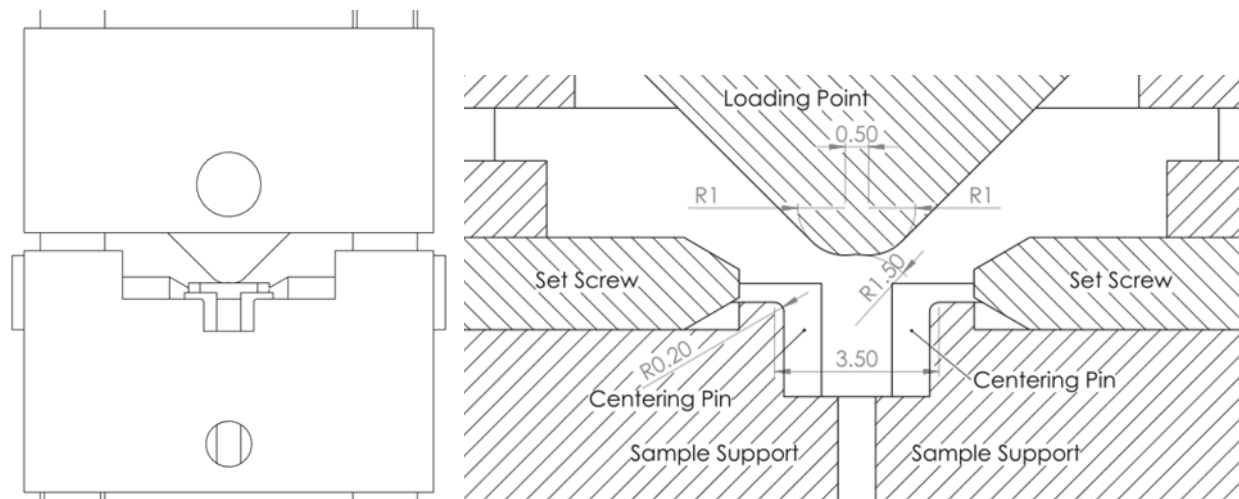


Figure 3-1. Schematic of the four-point bend loading fixture. Left: front view including bend sample, right: cross sectional view excluding the bend sample, showing dimensions in mm.

Four point bend tests were carried out using the IM1 system in the Irradiated materials Testing laboratory (IMTL) at University of Michigan. The details of the closed water loop and the autoclave environment have been described previously in this report for the CERT testing.

Tests were performed in room temperature (RT) air, 288°C Ar gas, or 288°C normal water chemistry (NWC) conditions. Sample bending was performed by moving the crosshead at a constant rate of 1.3×10^{-7} in/s, until achieving the desired amount of stress or sample deflection. The crosshead movement rate created a sample strain rate of $3.5 \times 10^{-7} \text{ s}^{-1}$, identical to the strain rate used in the previous CERT experiments.

The testing was performed in increments to create crack initiation sites and limit the amount of crack propagation in the material. Increments are reported in this report as either the fraction of yield stress or the amount of plastic strain added to the material post yielding. Samples surfaces were imaged between increments using scanning electron microscopy.

To evaluate the dependence of the strain in the material on the deflection, four point bend tests were carried out on unirradiated material in as-received and cold-worked conditions. The strain after a particular amount of deflection was measured using digital image correlation technique. Digital image correlation (DIC) was used to map strain on the surface of bend samples after tests in RT air and 288°C Ar gas, to compare with FEA model results. The DIC technique determines displacement by comparing images of a speckle pattern with high contrast difference recorded pre- and post-deformation. Speckle patterns were applied by using high-temperature RustOleum™ spray paint (10), and images were recorded with a Fujifilm Finepix S7000 digital camera used together with a 4x magnification stereoscope. A Matlab script created by McMurtrey

et al. 17] was used to make strain maps from the images, and the surface strain for each test was recorded as the average strain determined from the DIC analysis in the center 500 x 500 μm region of the bend sample. DIC results were confirmed by comparison with measurements taken from an array of microhardness indentations made on the sample surface prior to deformation. The specimen deflections were measured using a microminiature LVDT for the tests carried out at room temperature. For the high temperature tests, direct sample measurements were not possible in high temperature environments due to limitations of the electronics in the device, therefore estimations of sample deflection were made by subtracting a load dependent compliance correction factor from the crosshead deflection measurement. This compliance correction factor was determined in both 288°C Ar and 288°C NWC, by loading a ~10 mm thick ‘bend’ sample with the same cross-sectional geometry as the actual bend test samples. Due to its large thickness, it was assumed that no sample deflection occurred during loading, and compliance correction curves were determined by fitting a second order polynomial to the resulting load vs. crosshead displacement curves. Compliance correction curve determination was repeated three times for accuracy, and averaged to determine the following correction factors in 288°C Ar:

$$CF_{Ar} = -6.57 \times 10^{-5} P^2 + 0.493 P - 15.8 \quad (3-3)$$

and in 288°C NWC:

$$CF_{NWC} = -5.56 \times 10^{-5} P^2 + 0.460 P - 24.6 \quad (3-4)$$

where P is the applied load in N and CF_i is the correction factor in environment i in μm . The correction factor subtracted from the crosshead deflection yields the sample deflection.

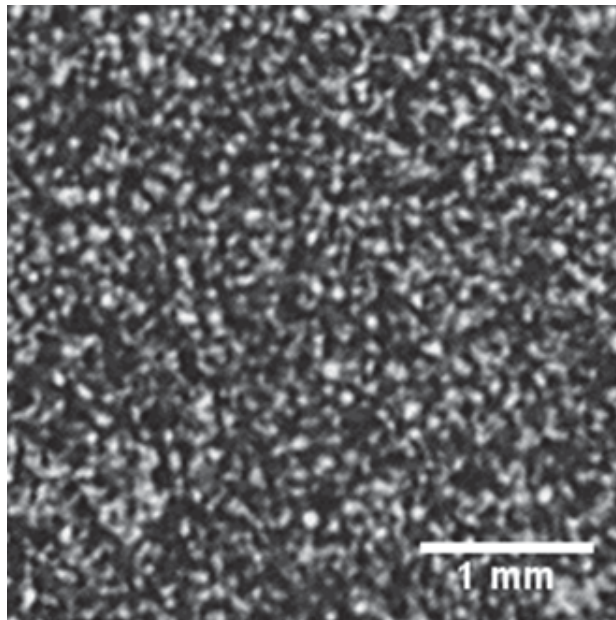


Figure 3-10. DIC speckle pattern applied with Rustoleum spray paint. Particle size ranges between 50 and 100 μm .

Oxide removal

The oxide from exposure during SCC testing was removed to allow for better observation of IGSCC initiation sites and electron backscatter diffraction imaging in these regions. A chemical

procedure is effective at removing surface oxidation, which consists of soaks in two water-based solutions. The first is 100 g/L sodium hydroxide with 30 g/L potassium permanganate, the second is 100 g/L ammonium oxalate. Both solutions are heated to ~ 95 °C, and samples are soaked for 5 minutes in each, rinsing between soaks and ultrasonically cleaning after each iteration. The process is repeated until all oxide was visibly removed, and was found to have a minor effect on surface feature height, causing only a 17 ± 33 nm variation for features in the height range 300 — 3000 nm.

4 RESULTS

This section presents a summary of the results of the microstructural analysis, completed on the as-irradiated condition and multiple annealing conditions. The microstructural analysis includes the effect of annealing on the Vickers micro-hardness, dislocation loop size and density, solute cluster size and density, and the grain boundary segregation.

4.1 *Hardness*

Vickers micro-hardness has been often used in literature as a simple method to evaluate the bulk changes in the irradiated microstructure following PIA treatments, as a greater reduction in hardening has been observed to correspond to a greater removal of irradiation defects such as dislocation loops and solute clusters. As such, to select the specific annealing conditions for microstructure analysis, CERT and CGR experiments, a wide range of annealing temperatures and times were applied to the as-received sample blanks listed in Table 3-6.

Four different temperatures: 450°C, 500°C, 550°C, and 600°C with times: 1, 5, and 20 hr at each temperature were utilized, as these time/temperature combinations were expected to fully bound the complete removal of irradiation hardening. The hardness measurements for the selected temperatures are shown in Table 4-1 and Figure 4-1, where the irradiation hardening is plotted as a percentage of the as-irradiated condition.

Table 4-1: Change in the as-irradiated hardness due to specific annealing treatments. Overall the hardness was seen to decrease with increasing temperature and time. The irradiation hardening was determined using a value of $H_v = 157.95$ for the hardness of 304L stainless steel.

Sample Blank ID	PIA treatment	As-Irradiated Hardness (H_v)	PIA Hardness (H_v)	Irradiation Hardening (H_v)	Post-PIA Irradiation Hardening (H_v)	% of As-Irradiated Hardening
17	600C: 1hr	371.67	228.95	213.71	71.00	33.22
17A	600C: 5hr	371.67	185.38	213.71	27.43	12.83
9	450C: 5hr	341.50	310.42	183.55	152.48	83.07
9A	450C: 1hr	341.50	329.09	183.55	171.14	93.24
18	500C: 1hr	334.75	321.00	176.80	163.05	92.22
18A	500C: 5hr	334.75	304.81	176.80	146.86	83.07
16	500C: 20hr	350.33	286.61	192.38	128.67	66.88
16A	600C: 5hr	350.33	184.61	192.38	26.67	13.86
11	550C: 1hr	341.67	287.28	183.71	129.33	70.40
11A	600C: 20hr	341.67	150.42	183.71	-7.52	-4.10
5	500C: 1hr	339.75	308.05	181.80	150.10	82.56
5A	450C: 20hr	339.75	309.81	181.80	151.86	83.53
15	550C: 5hr	337.42	248.28	179.46	90.33	50.33
15A	550C: 20hr	337.42	220.19	179.46	62.24	34.68

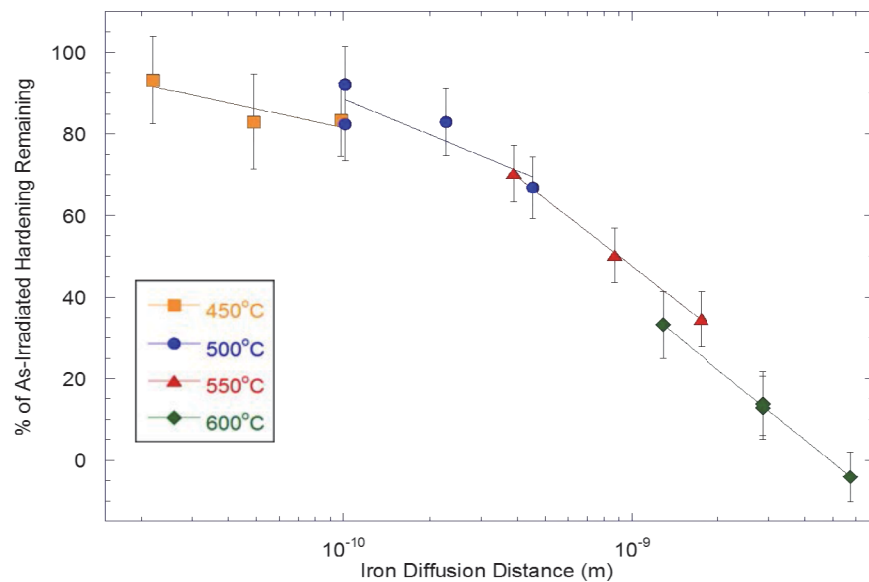


Figure 4-1: Change in the residual irradiation hardening due to specific annealing treatments.

The irradiation hardening ($\Delta H_{v, Irr}$), is the increase in the hardness due to the presence of irradiation defects and is calculated from the following expression:

$$\Delta H_{v, Irr} = H_{v, Irr} - H_{v, Unirr} , \quad (4.1)$$

where $H_{v, Irr}$ is the measured hardness of the as-irradiated 304L stainless steel, and $H_{v, Unirr}$ is the measured hardness of the unirradiated 304L stainless steel. As no archive material is available, the hardness of the unirradiated materials was assumed to be that of a 304L stainless steel, which was measured as 157.95 H_v . Similarly, the post-PIA irradiation hardening ($\Delta H_{v, PIA}$) can be calculated via equation 3.1, where the measured hardness after PIA ($H_{v, PIA}$) is substituted for the as-irradiated measurement ($H_{v, Irr}$). The percentage of as-irradiated hardening remaining is then calculated as:

$$\% \text{ of as - irradiated hardening} = \frac{\Delta H_{v, PIA}}{\Delta H_{v, Irr}} . \quad (4.2)$$

Overall, it was observed that annealing at 450°C, had a negligible impact on the irradiation hardening up to times of 20 hours. However, temperatures of 500°C, 550°C, and 600°C showed successively greater removal of hardening with increasing time and temperature. Following annealing at 600°C: 20hr, the measured hardness had returned to a value expected for an unirradiated 304L stainless steel.

Using the criterion outlined in previous quarterly reports, annealing conditions of 500°C: 1hr, 550°C: 1hr, 550°C: 5hr, and 550°C: 20hr, were selected for both more detailed microstructural analysis and application to the CERT and CGR specimens. Following the annealing of the CERT and CGR samples, small slices from the tensile heads were used to confirm the removal of hardness. The results for these slices was outlined in previous reports, but there was a good agreement in hardening removal as compared to the data in Table 4-1.

4.2 Irradiated Microstructure

4.2.1 Dislocation Loops

The faulted dislocation loops in the as-irradiated 304L SS and after PIA at 500°C: 1hr, 550°C:1 and 5hr were characterized using rel-rod dark field technique. TEM images of dislocation loops are shown in Figure 4-2. Dislocation loops were confirmed in all examined PIA conditions. However, annealing at 550°C: 20hr resulted in a low density of large loops. Because of the very low density of loops, rel-rods were not observed and dislocation loops were imaged bright field under a two-beam condition. In addition to loops, stacking fault tetrahedral were observed as shown in the insert of Figure 4-3.

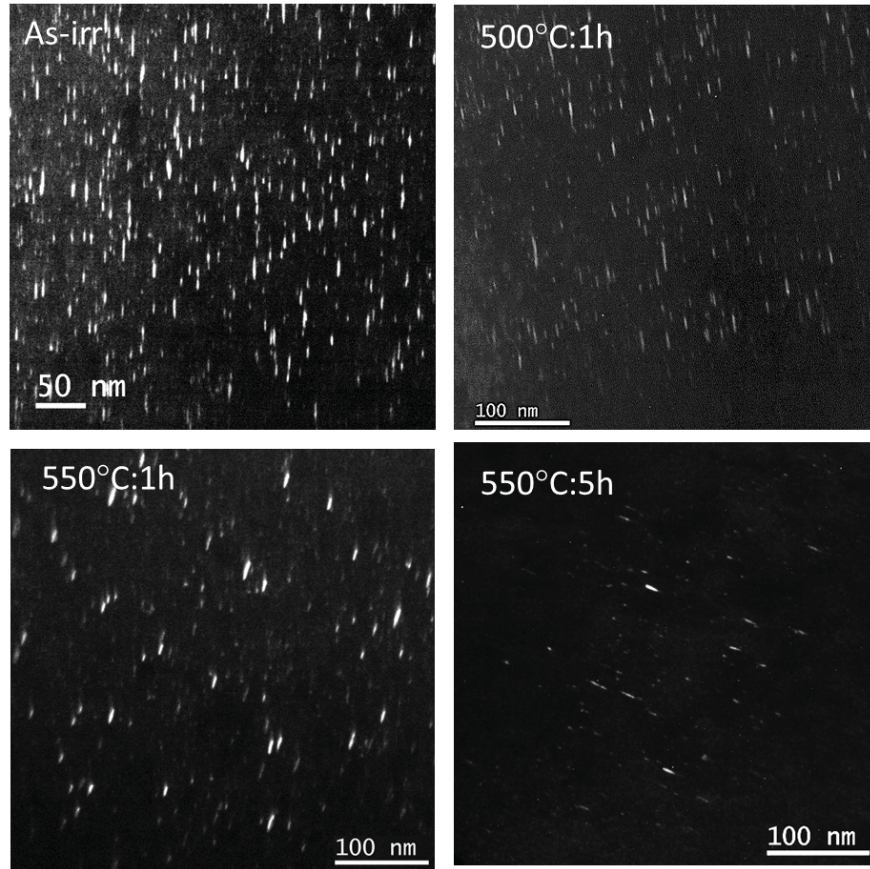


Figure 4-2: Rel-rod dark field TEM image showing the faulted dislocation loops in 304L SS irradiated to 5.9 dpa in BWR after various post-irradiation annealing conditions.

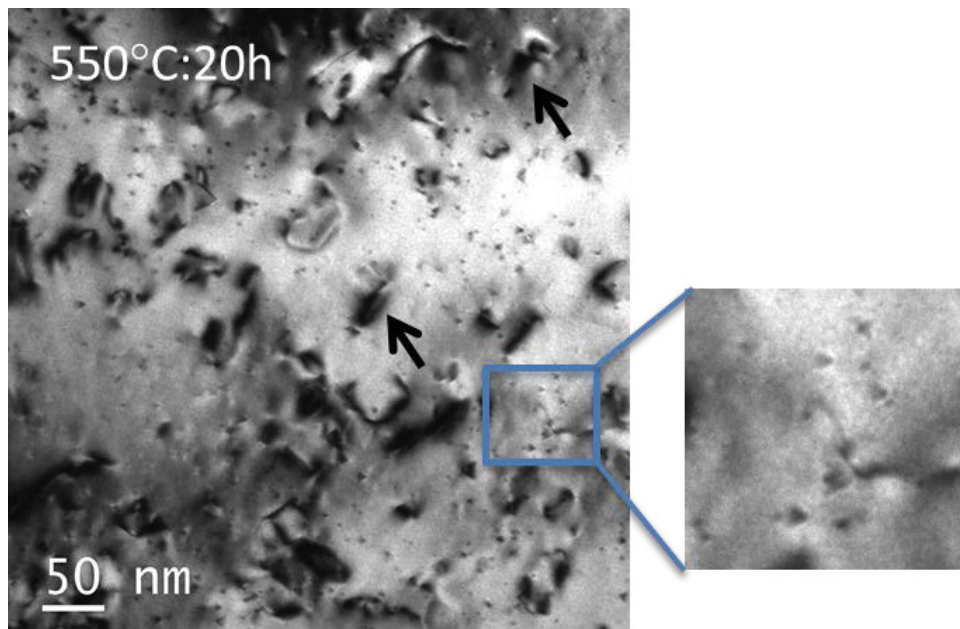


Figure 4-3: Dislocation loops (as indicated by arrows) and small stacking fault tetrahedral (as shown in the insert) in 304L SS after post-irradiation annealing at 550°C for 20 hours.

The average dislocation loop diameter and density in 304L SS irradiated to 5.9 dpa in BWR and after various post-irradiation annealing conditions are given in Table 4-2. The initial average dislocation loop diameter after irradiation was 8.3 nm and it was 9.6 nm after PIA at 500°C: 1hr. The average loop size was 8.9 nm and 8.0 nm after PIA at 550°C: 1hr and 5hr, respectively. The comparison of loop size at different annealing conditions is shown in Figure -4. The dislocation loop sizes are virtually the same after annealing at 550°C for 5h. Significant increase in dislocation loop size was observed after 550°C for 20h, in which the loop size was about 26 nm. The loop density reduced significantly from $1.1 \times 10^{23} \text{ m}^{-3}$ at the as-irradiated condition to $8.2 \times 10^{22} \text{ m}^{-3}$ after annealing at 500°C: 1hr, to $3.2 \times 10^{22} \text{ m}^{-3}$ at 550°C: 1hr, to $1.3 \times 10^{22} \text{ m}^{-3}$ at 550°C: 5hr. The number density became negligible after annealing at 550°C: 20hr compared to the number density in the as-irradiated condition. In other words, the loop density reduced to $\sim 3/4$ of its initial density after PIA at 500°C: 1hr and to $\sim 1/10$ after PIA at 550°C: 5hr. Post-irradiation annealing at 550°C for 20 hours resulted in low number density of large loops.

The size distribution of dislocation loops in 304L SS irradiated to 5.9 dpa at in BWR environment and after various post-irradiation annealing conditions is shown in Figure b. It appears that smaller dislocation loops are preferably annealed out at the 500°C for 1hr condition and in fact, the density of large loops ($>13\text{nm}$) shows increase in population compared to the as-irradiated condition. However, this is not observed for annealing at 550°C condition, in which the dislocation density decreases across the whole size spectrum.

Table 4-2: Dislocation loop size, number density in 304L SS irradiated to 5.9 dpa in BWR and after various post-irradiation annealing conditions.

Condition	Density (m^{-3})	% of As-Irradiated Density	Average Diameter (nm)
As-Irradiated	11.1×10^{22}	100%	8.3
500°C: 1 hr	8.21×10^{22}	74.0%	9.6
550°C: 1 hr	3.25×10^{22}	29.3%	8.9
550°C: 5 hr	1.27×10^{22}	11.4%	8.0
550°C: 20 hr	0.05×10^{22}	0.4%	26

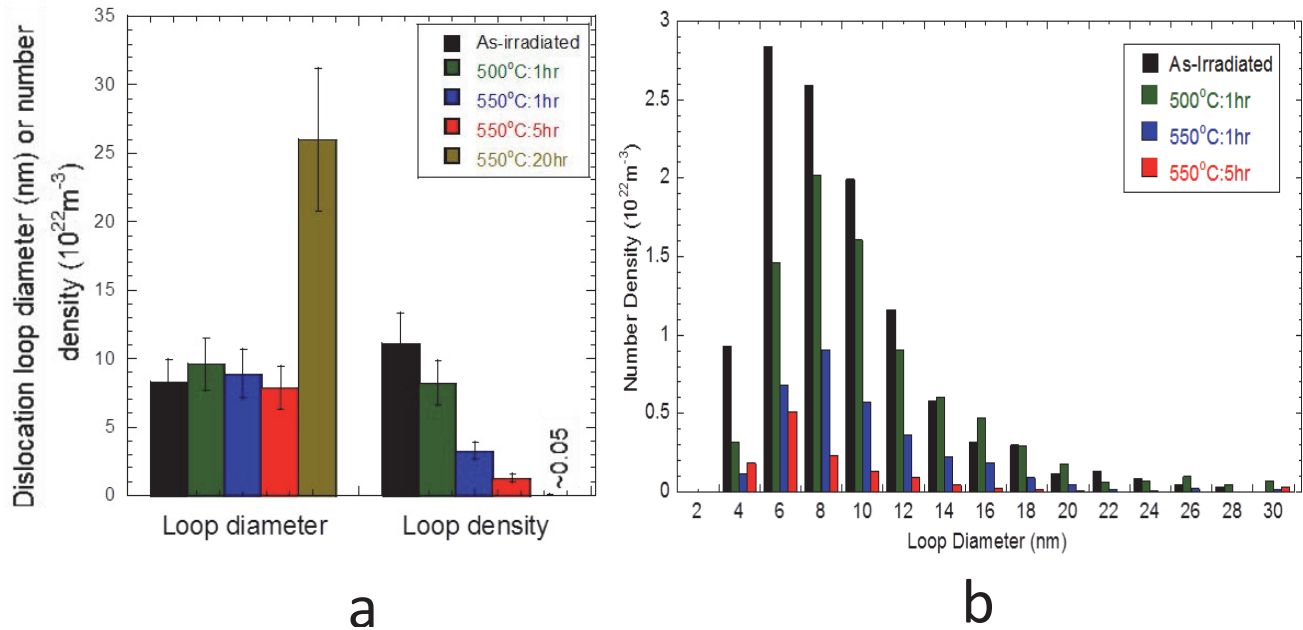


Figure 4-4: Comparison of (a) size and number density, and (b) size distribution of faulted dislocation loops in 304L SS irradiated to 5.9 dpa in BWR and after various post-irradiation annealing conditions.

4.2.2 Solute Clusters

Two types of solute clusters were observed in 304L SS irradiated to 5.9 dpa in BWR; Ni-Si rich clusters and Al-Cu rich clusters. They are shown in Figure 4-5 for the as-irradiated condition. Evolution of the Ni-Si and Al-Cu clusters after PIA is shown in Figure 4-6 and the number density, diameter, Ni-Si or Al-Cu ratio and volume fraction for Ni-Si and Al-Cu clusters are given in Table 4-3 and Table 4-4, respectively. The Ni-Si clusters are mainly precursors of the γ' or G phase as the concentrations of Ni and Si have not reached that for phase formation (75% Ni and 25%Si). The number density in the as-irradiated condition is $3.88 \times 10^{23} \text{m}^{-3}$ and decreases to $2.83 \times 10^{23} \text{m}^{-3}$ after PIA at 500°C: 1hr and to $2.35 \times 10^{23} \text{m}^{-3}$ after PIA at 550°C:1hr. The density further decreases to $0.7 \times 10^{23} \text{m}^{-3}$ after annealing at 550°C: 20hr, which is only ~18% of the as-irradiated number density. The cluster size, however, increases from ~9 nm at the as-irradiated condition to 20 nm after annealing at 550°C: 20hr. Due to the increase in the cluster size the volume fraction does not change significantly after annealing: ~2.5% in the as-irradiated condition and ~3.0% after annealing at 550°C: 20hr.

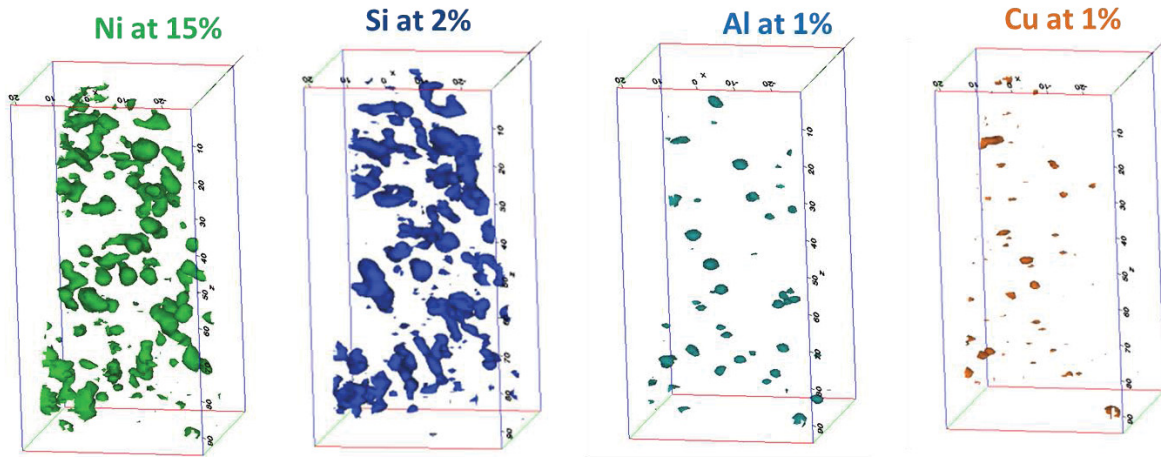


Figure 4-5. Ni-Si and Al-Cu clusters as observed in 304L SS irradiated to 5.9 dpa in BWR. Clusters are shown using isoconcentration surface plots from APT atom maps.

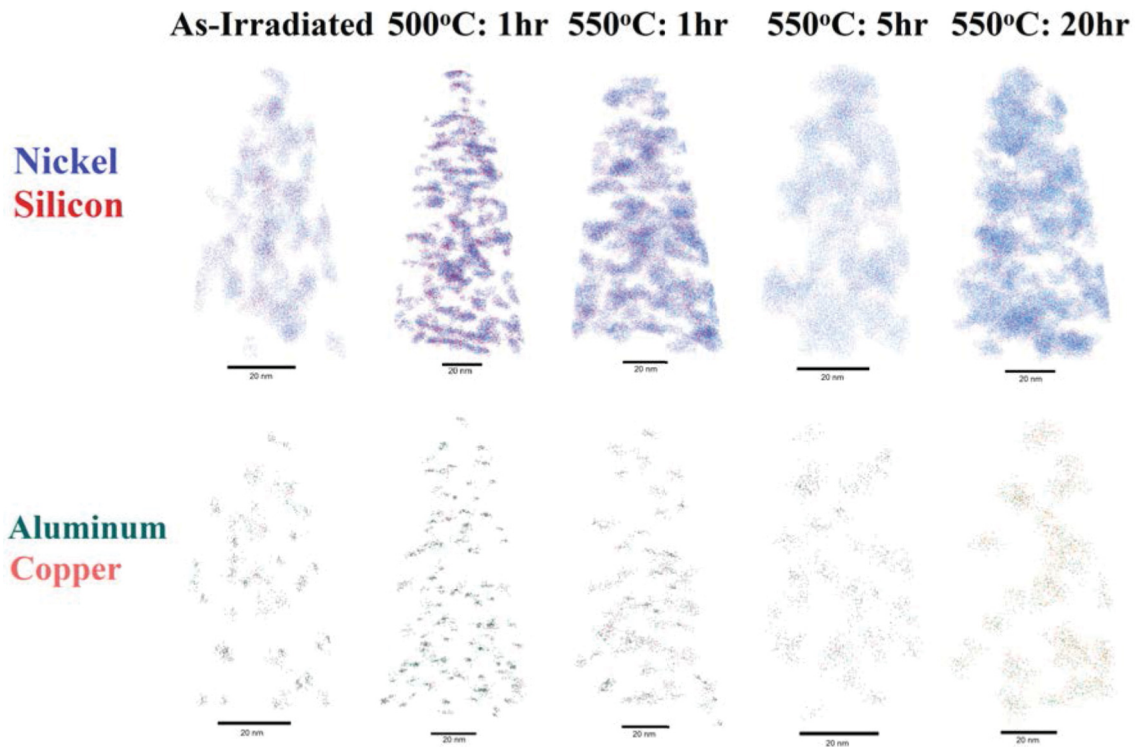


Figure 4-6. Evolution of Ni-Si and Al-Cu clusters in 304L SS irradiated to 5.9 dpa in BWR after various post-irradiation annealing conditions.

Table 4-3: Ni-Si cluster size, density and volume fraction in 304L SS irradiated to 5.9 dpa in BWR and after various post-irradiation annealing conditions.

Condition	Density ($10^{23} / \text{m}^3$)	% of As-Irradiated	Diameter (nm)	Ni-Si Ratio	Volume Fraction (%)
As-Irradiated	3.88 ± 0.59	100%	9.24 ± 0.71	5.13 ± 0.13	2.47 ± 0.08
500°C: 1 hr	2.83 ± 0.46	72.8%	10.79 ± 0.90	5.12 ± 0.48	2.44 ± 0.21
550°C: 1 hr	2.35 ± 0.11	60.5%	12.34 ± 0.09	5.43 ± 0.27	2.97 ± 0.05
550°C: 5 hr	1.21 ± 0.47	31.2%	18.09 ± 4.68	7.61 ± 0.59	3.59 ± 0.91
550°C: 20 hr	0.70 ± 0.07	18.0%	20.23 ± 1.43	7.95 ± 0.03	2.96 ± 0.16

Table 4-4: Al-Cu cluster size, density and volume fraction in 304L SS irradiated to 5.9 dpa in BWR and after various post-irradiation annealing conditions.

Condition	Density ($10^{23} / \text{m}^3$)	% of As-irradiated	Diameter (nm)	Al-Cu Ratio	Volume Fraction of Al (%)
As-Irradiated	2.92 ± 0.20	100%	5.68 ± 0.56	2.13 ± 0.60	0.035 ± 0.006
500°C: 1 hr	2.46 ± 0.10	84.4%	6.19 ± 0.36	2.20 ± 0.06	0.027 ± 0.003
550°C: 1 hr	1.57 ± 0.11	53.8%	9.54 ± 0.66	1.19 ± 0.05	0.021 ± 0.002
550°C: 5 hr	2.61 ± 0.47	89.4%	11.00 ± 0.63	1.14 ± 0.08	0.041 ± 0.006
550°C: 20 hr	0.93 ± 0.26	31.8%	13.15 ± 1.16	0.69 ± 0.02	0.016 ± 0.001

The Al-Cu shows the same trend as Ni-Si cluster after annealing. The number density in the as-irradiated condition is $2.92 \times 10^{23} \text{ m}^{-3}$ and it decreases to $2.46 \times 10^{23} \text{ m}^{-3}$ after PIA at 500°C: 1hr and to $1.57 \times 10^{23} \text{ m}^{-3}$ after PIA at 550°C: 1hr. The density further decreases to $0.9 \times 10^{23} \text{ m}^{-3}$ after annealing at 550°C:20hr, which is ~32% of the as-irradiated number density. The cluster size, increases from ~6 nm at the as-irradiated condition to ~13 nm after annealing at 550°C: 20hr. The volume fraction of Al-Cu clusters is rather small, ~0.035% in the as-irradiated condition and ~0.016% after annealing at 550°C: 20hr.

4.2.3 Grain Boundary Segregation

Radiation-induced segregation was examined in 304L SS in the as-irradiated condition and PIA at 550°C for 5 and 20 hours. Figure 4-7 and Figure 4-8 are the ChemiSTEM elemental images showing segregation of Cr, Ni and Si at the grain boundary in the as-irradiated and PIA at 550°C:20h condition, respectively. Depletion of Cr, Fe and enrichment of Ni, Si and P are evident from composition profile across the grain boundary in the as-irradiated condition (Figure 4-7).

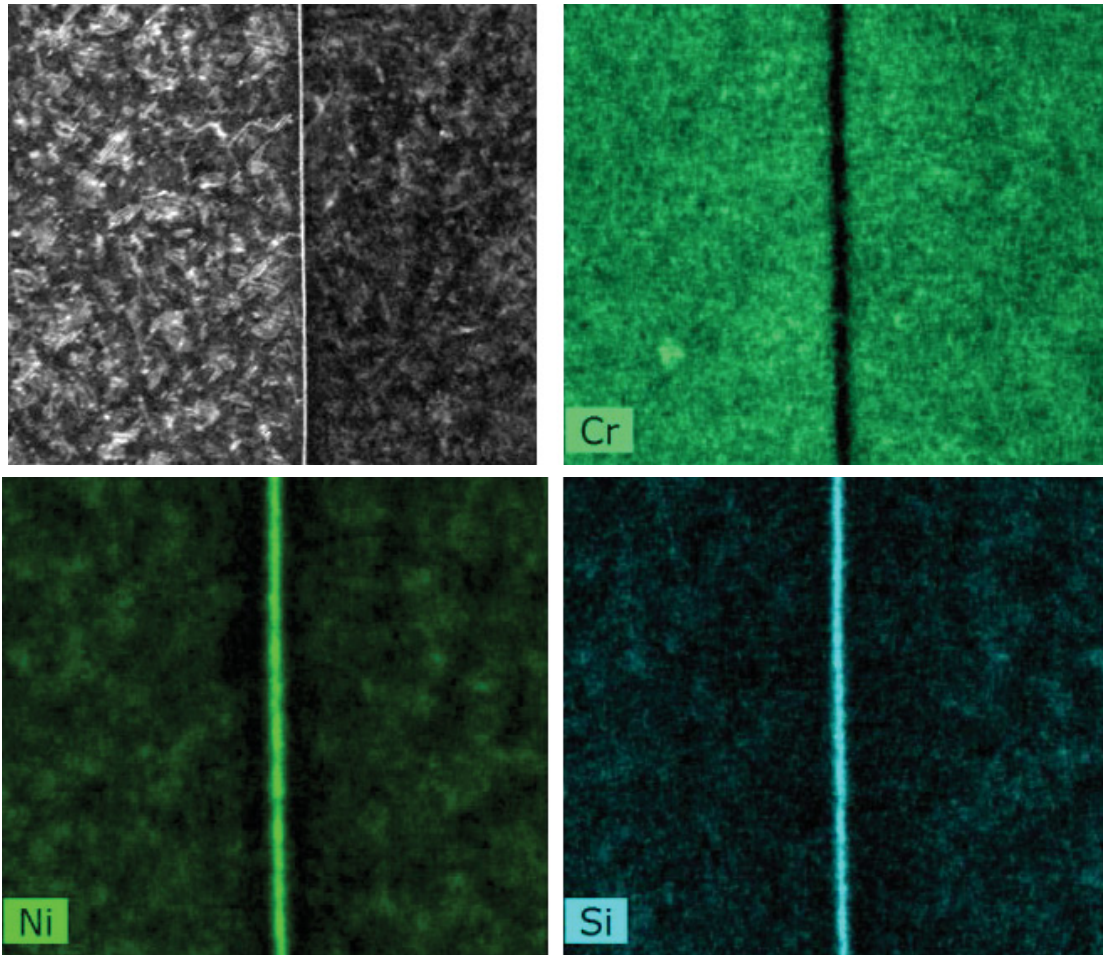


Figure 4-7: Radiation-induced segregation in 304L SS irradiated to 5.9 dpa in BWR as revealed by ChemiSTEM image. Depletion of Cr and enrichment of Ni and Si are evident.

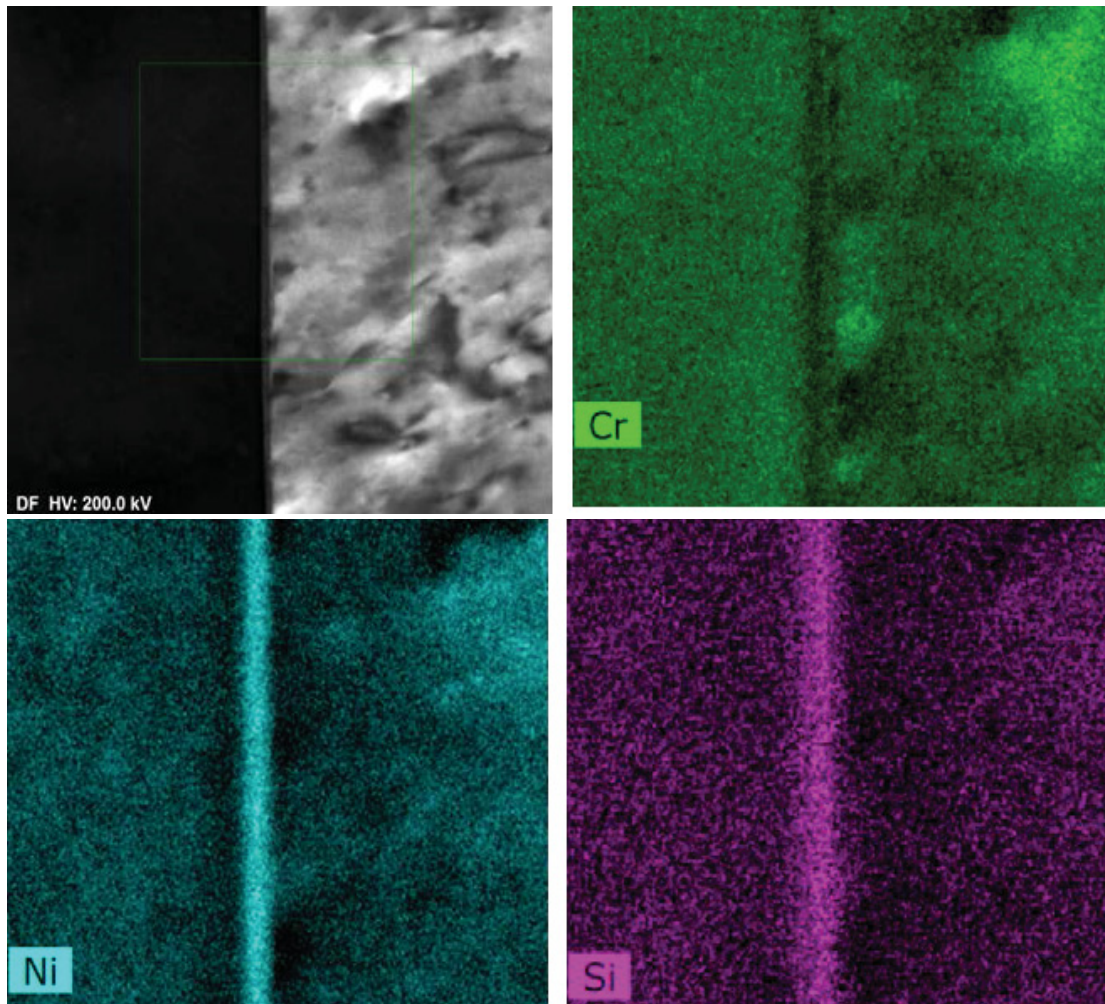


Figure 4-8: Radiation-induced segregation in 304L SS irradiated to 5.9 dpa in BWR and post-irradiation annealing at 550°C for 20 hr as revealed by ChemiSTEM image. Depletion of Cr and enrichment of Ni and Si are still evident.

In the as-irradiated condition, the depletion of Cr is ~5.5% and enrichment of Ni and Si are 13.4% and 2.2% respectively (Table 4-5). The depletion of Cr decreases to 3.2% after annealing at 550°C:5h and 1.4% after annealing at 550°C:20h. Enrichment of Ni drops to ~3.2% after annealing at 550°C:20h, which corresponds to 23% of Ni enrichment in the as-irradiated condition. There is only 0.13% of enrichment of Si at the grain boundary after annealing at 550°C:20h, which is ~6% of Si enrichment at the as-irradiated condition.

Composition profiles for Cr, Ni, Si and P for the as-irradiated and 550°C PIA conditions are shown in Figure 4-9 and 4-10. Besides the recovery of the segregated elements by thermal annealing, the segregated peaks appear to broader after annealing.

Table 4-5. Grain boundary concentrations in the as-irradiated condition and after PIA at 550°C for 5 and 20 hours.

	GB Cr (wt%)	Δ Cr (wt%)	% of As-Irr Δ Cr	GB Ni (wt%)	Δ Ni (wt%)	% of As-Irr Δ Ni	GB Si (wt%)	Δ Si (wt%)	% of As-Irr Δ Si
As-Irradiated	12.83	-5.52	100	23.98	13.41	100	2.48	2.18	100
550°C:5h	15.11	-3.24	59	14.7	4.13	31	0.65	0.35	16
550°C:20h	16.92	-1.43	26	13.72	3.15	23	0.43	0.13	6

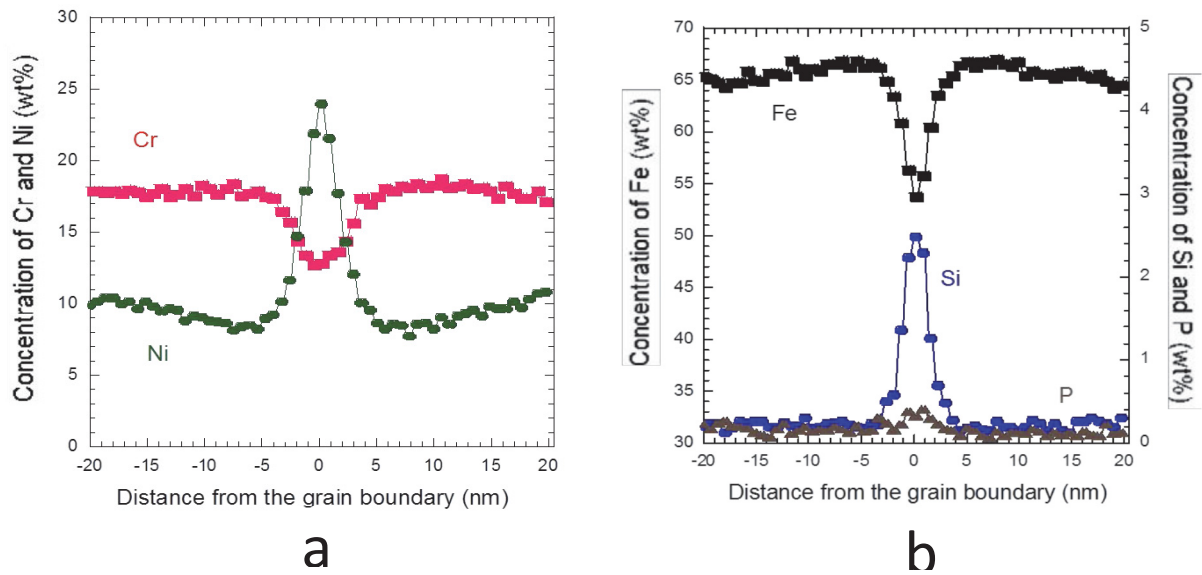


Figure 4-9. Composition profile across the grain boundary in 304L SS irradiated to 5.9 dpa in BWR for (a) Ni and Cr, and (b) Fe, Si and P.

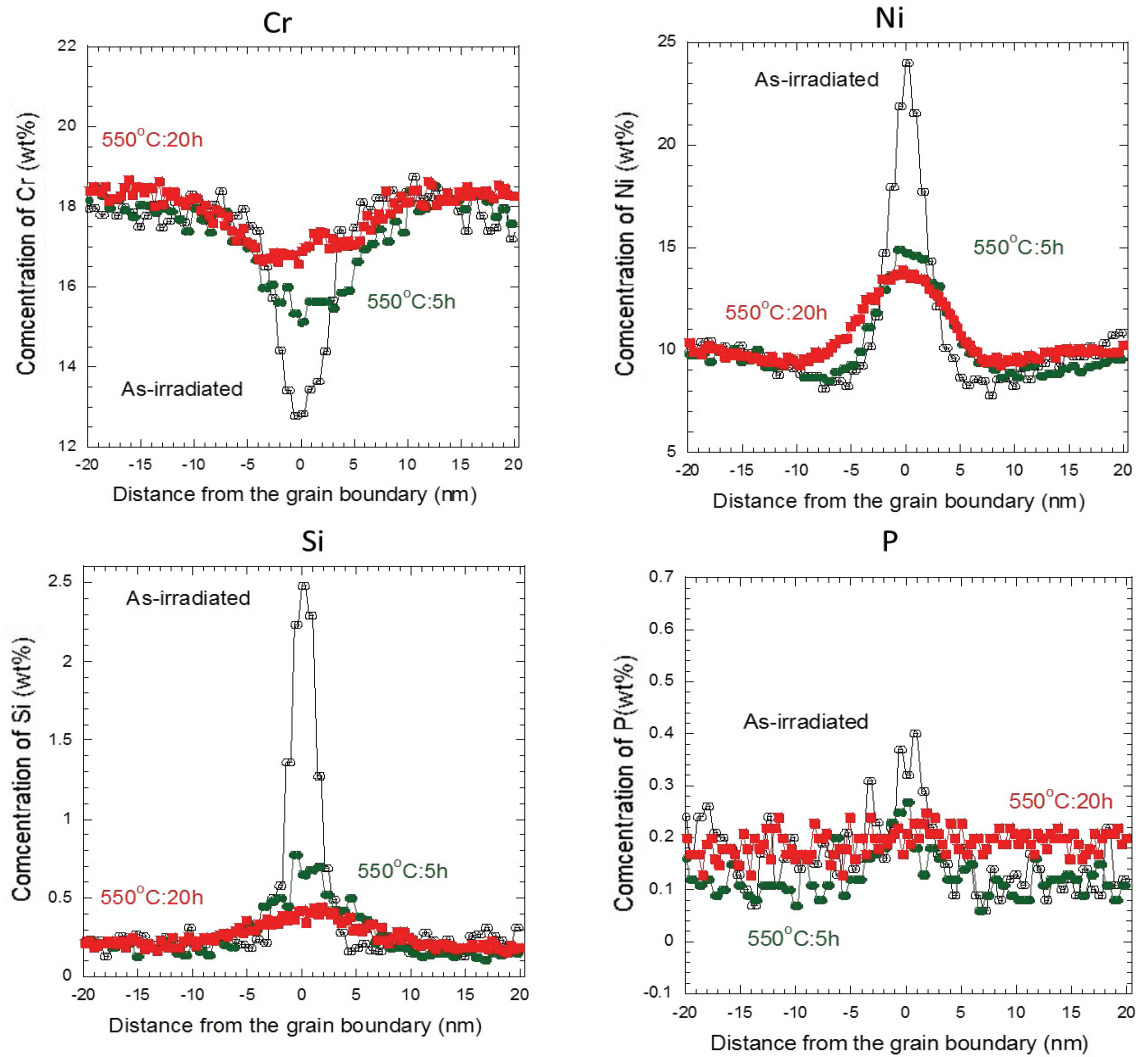


Figure 4-10. Effect of post-irradiation annealing on the segregation profiles of Cr, Ni Si and P in 304L SS irradiated to 5.9 dpa in BWR.

4.2.4 Overview of Microstructure of CIR Alloys

The microstructural characterization for the eleven alloys (four commercial purity and seven high purity alloys) was not carried out as part of this work. However, the microstructure for these materials has been previously characterized by different groups as part of the Cooperative IASCC Research (CIR) program. The microstructural details are reproduced here for reference purposes.

A summary of the franks loops and the precipitates observed in alloys A, B, C, E and H are summarized in Table 4-6 below. Frank Loops were observed in all the alloys and at the damage levels of the tensile bars used for the CERT studies (>5.5 dpa), the loop microstructure was observed to have reached saturation as shown by minimal change in the loop size and the loop density in these conditions. The loop size distribution for the different materials in the study are reproduced from the CIR report and are shown in Fig 4-11 below. The similar distribution profiles for the different alloys indicate that the loop microstructure for these alloys is similar in nature.

Table 4-6: Summary of Frank Loop and Precipitate Data in Alloy AS, B, C, ES and HS [18,19]

Alloy	Dose (dpa)	Frank Loops		Precipitates	
		Average Size (nm)	Density (10^{23} m^{-3})	Average Size (nm)	Density (10^{23} m^{-3})
Alloy AS	5.4	6	4.4		
	10.3	8	2	7.6	0.14
Alloy B	5.4	8	1.2		
	25	7.8	1.4	5.7	0.1
Alloy C	5.4	8.5	1.8		
Heat ES	10.3	6.1	4	5.8	0.14
Heat HS	10.3	8.9	1.4	5.2	0.24
Heat KS	9.9	9.2	0.8	5.2	0.12
Heat LS	9.9	11.4	0.47	5.9	0.09

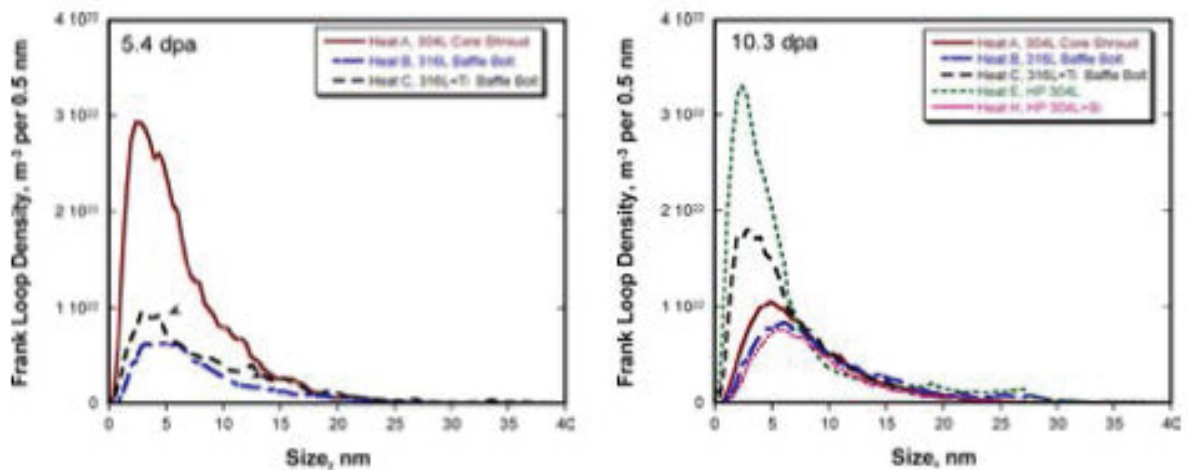


Figure 4-11: Frank loop size distributions for different heats at two damage levels of 5.4 dpa and 10.3 dpa. [18]

Precipitates were observed for alloy AS at a damage level of 10.3 dpa while they were absent at a damage level of 5.4 dpa. For alloy B, the precipitates were only found to be present at a damage level of 25 dpa and the size and number density was similar to those observed for alloy AS at 10.3 dpa. This indicates that the precipitation of γ' precipitates requires higher damage level for type 316 stainless steels compared to type 304 stainless steels. For alloy C (type 316 + Ti), no precipitates were observed in the CIR program for a damage level as high as 25 dpa.

For the high purity(HP) alloys, precipitates were observed in both the model alloy ES as well as the alloy modified using Si addition. The precipitates size were similar for both alloys at similar

damage level of 10.3 dpa while the number density was higher for alloy HS which indicates that the addition of Si leads to preferential precipitation of γ' precipitates in the material.

4.3 Crack initiation in CERT tests on PIA conditions

This section presents a description of the incremental CERT test results obtained from the irradiated and PIA tensile bars. CERT tests were conducted at 288°C under simulated BWR-NWC conditions. The stress-strain behavior of each specimen is first presented with comments about the observed mechanical behavior. A summary of the mechanical properties is then presented followed by images of each fracture surface after completion of the CERT tests. More detailed information regarding each of the strain increments was outlined in previous quarterly reports.

4.3.1 Prediction of Yield Stress and Incremental Straining Experiments

Prior to beginning the incremental straining experiments, it was necessary to first predict the yield stress of both the as-irradiated condition and following each annealing treatment. Previous studies have shown that the change in irradiation hardening is linearly related to the change in the yield stress as seen in Equation 3.3 [19].

$$\Delta\sigma_y = X * \Delta H_v \quad (4.3)$$

Where X is the linear correlation factor; prior work by Busby *et al.* found that for irradiated austenitic stainless steels, an average correlation factor of $X = 3.03$, best fits the available data. However, the correlation factors for individual data sets had more variability, ranging from 2.63 to 3.83 [19]. Using the average correlation factor of 3.03 and the unirradiated hardness and yield stress of 157.95 H_v and 211.5 MPa, respectively, an initial prediction of the yield stress for our initial two specimens, T-4 (as-irradiated) and T-9 (550°C: 20 hr) were calculated based on the hardness measurements taken after PIA treatments, as shown in Table 4-7. Based on these predictions straining increments to 40, 60, 80, and 100% were made for these two specimens as listed in Table 4-8. The as-irradiated specimen T-4 showed a very close agreement between the predicted and measured yield stress. However, for the T-9 specimen, the actual hardness was originally incorrectly measured, thus resulting in a miscalculated yield stress prediction. As such, an additional stress increment was required to reach the correct yield stress. After remeasuring the hardness of the T-9 specimen, it was found to be much higher than originally expected, but the new value correctly matched the measured yield stress.

Utilizing these measured yield stresses a new correlation factor was fit to our specific material, which was then used to predict the yield stresses of the T-5 (500°C: 1hr), T-13 (550°C: 1hr), T-7 (550°C: 5hr), and T9 (550°C: 20hr) specimens. As shown in Figure 4-12, the final correlation factor for these five specimens was calculated as $X = 2.47$. While this correlation factor is lower than those previously seen in literature, the difference is likely an effect of the PIA treatments, as the previous correlation factors only examined as-irradiated microstructures [19]. Figure 4-13

shows a near perfect 1-to-1 comparison between the final predicted and measured yield stresses utilizing this fitted correlation factor.

Table 4-7: Prediction of the yield stress following PIA based on change in hardness.

Specimen	Measured Hardness (H_v)	Predicted Yield Stress (MPa)	Measured Yield Stress (MPa)
T-4 (As-Irradiated)	348.0 ± 9.6	681.3 ± 23.8	685.5
T-5 (500°C: 1 hr)	326.9 ± 9.5	629.1 ± 23.5	633.4
T-13 (550°C: 1 hr)	298.5 ± 13.9	558.9 ± 34.4	553.2
T-7 (550°C: 5 hr)	266.7 ± 10.2	480.2 ± 25.2	483.6
T-9 (550°C: 20 hr)	247.9 ± 14.2	433.7 ± 35.1	421.3

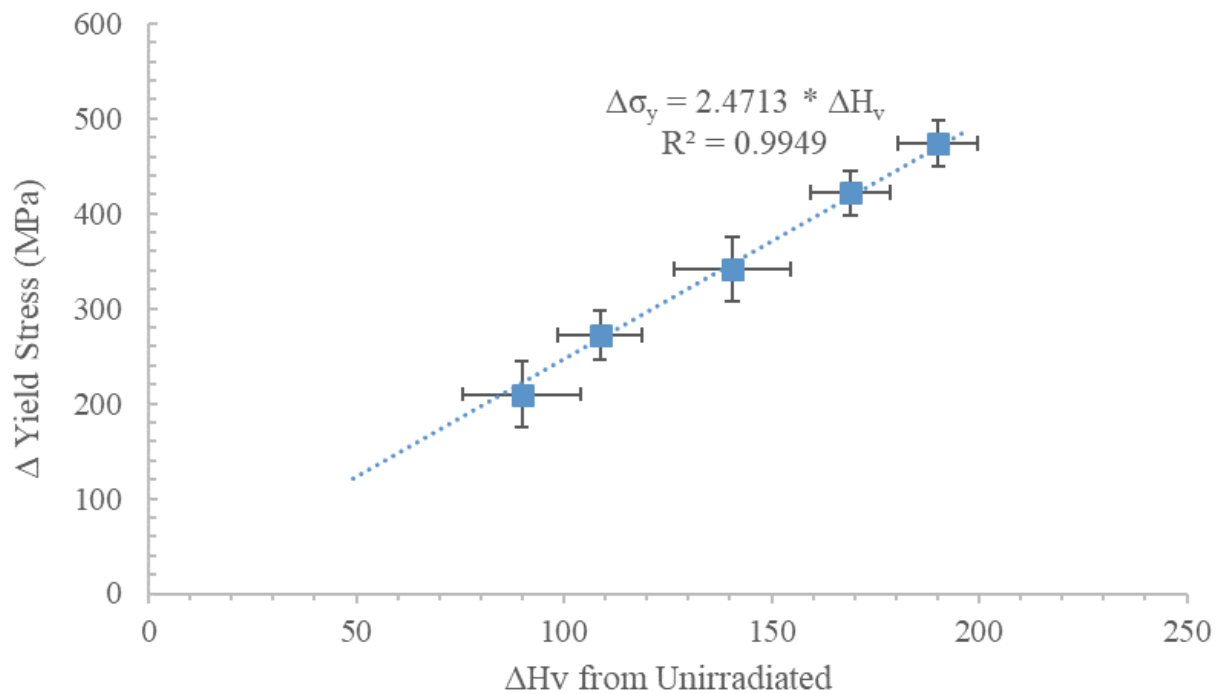


Figure 4-12: Comparison between the measured change in yield stress and the change in hardness from the base unirradiated condition ($\sigma_y = 211.5$ MPa, $H_v = 157.95$) used to predict the yield stress.

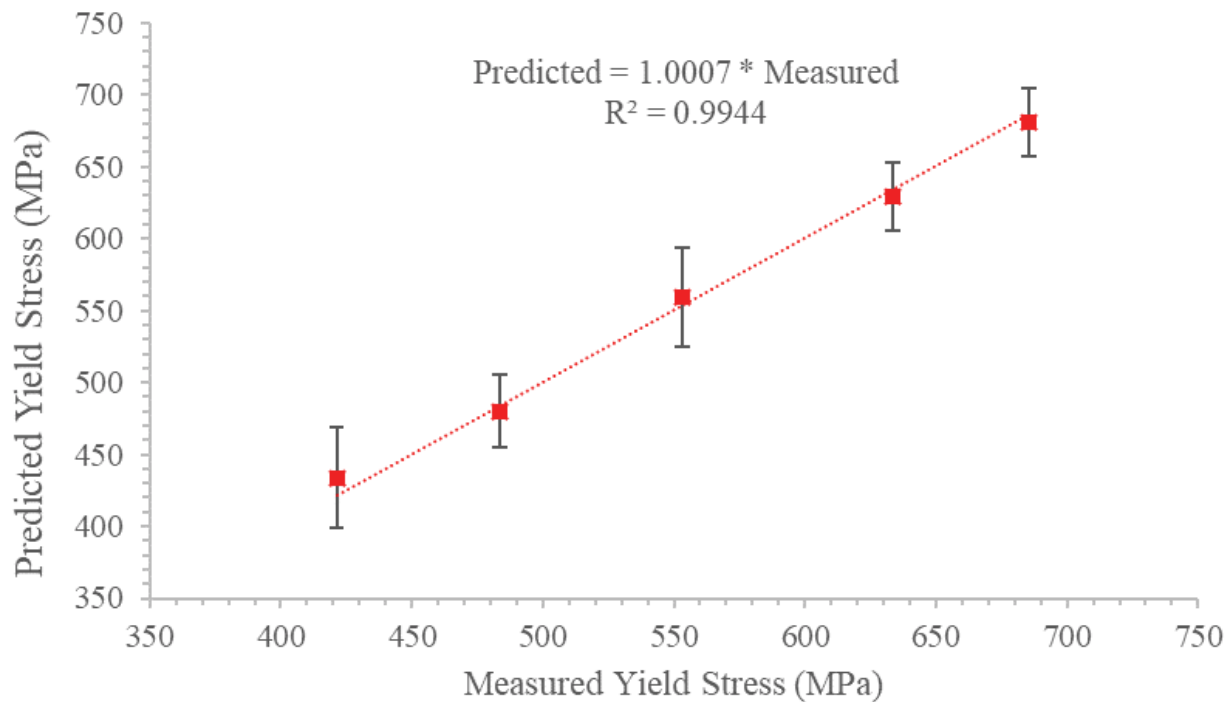


Figure 4-13: Comparison between the predicted yield stress values using a correlation factor of 2.47 and the measured values experimentally.

Table 4-8 shows a complete list of the incremental straining experiments that were applied to each of the examined specimens, including the target stress/strain for each increment as well as the final value. Overall, there was a good agreement between the target and achieved stress/strain, excluding the yield stress for specimen T-9 specimen and the 0.5% ϵ_p increment for the as-irradiated T-4 specimen. The T-7 increment to 10% ϵ_p was purposely halted early for examination due to the occurrence of significant necking.

Table 4-8: List of the tensile straining increments that were applied to the four tensile specimens tested for this project, including the maximum stress and plastic strain following each increment.

Specimen	Target value of stress or strain	Maximum Stress (MPa)	Fraction of Yield Stress (%)	Total Plastic Strain (%)
T-4 (As-Irradiated)	40% σ_y	270.6	39.5	0.00
	60% σ_y	401.5	58.6	0.00
	80% σ_y	539.5	78.7	0.00
	100% σ_y	685.5	100.0	0.13
	0.5% ϵ_p	682.8	99.6	0.74
	1.5% ϵ_p	657.2	95.9	1.51
	ϵ_f	328.5	47.9	2.00

T-5 (500°C: 1 hr)	80% σ_v	497.7	78.6	0.00
	100% σ_v	633.4	100.0	0.12
	1.0% ϵ_p	642.4	101.4	1.10
	1.5% ϵ_p	636.7	100.5	1.63
	2.0% ϵ_p	607.8	96.0	2.11
	ϵ_f	471.0	74.4	2.58
T-13 (550°C: 1 hr)	80% σ_v	446.0	80.6	0.00
	100% σ_v	553.2	100.0	0.08
	1.0% ϵ_p	555.1	100.3	1.07
	2.0% ϵ_p	558.6	101.0	2.05
	3.0% ϵ_p	564.0	102.0	3.11
	4.0% ϵ_p	565.8	102.3	4.07
	5.0% ϵ_p	571.1	103.2	5.10
	7.0% ϵ_p	569.2	102.9	7.19
	9.0% ϵ_p	557.7	100.8	9.35
	ϵ_f	515.8	93.2	11.61
T-7 (550°C: 5 hr)	80% σ_v	377.4	78.0	0.00
	100% σ_v	483.7	100.0	0.13
	2.0% ϵ_p	502.0	103.8	2.06
	4.0% ϵ_p	510.5	105.6	4.04
	6.0% ϵ_p	512.0	105.9	6.08
	10.0% ϵ_p	512.6	106.0	9.47
	ϵ_f	470.1	97.2	12.12
T-9 (550°C: 20 hr)	40% σ_v	130.8	31.0	0.00
	60% σ_v	196.2	46.6	0.00
	80% σ_v	261.6	62.1	0.00
	100% σ_v	341.1	81.0	0.00
	100% σ_v	415.6	98.6	0.08
	1.0% ϵ_p	437.7	103.9	1.06
	2.0% ϵ_p	454.4	107.9	2.02
	3.0% ϵ_p	463.6	110.0	3.00
	5.0% ϵ_p	478.6	113.6	5.00
	ϵ_f	491.5	116.7	11.43

4.3.2 Stress-Strain Behavior

Stress-strain curves for each of the tested conditions: as-irradiated, 500°C: 1 hr, 550°C: 1 hr, 550°C: 5 hr, and 550°C: 20 hr are shown in Figure 4-14. The elastic deformation portion of each curve has been corrected to subtract system compliance by normalizing to the expected Young's modulus for an austenitic stainless steel.

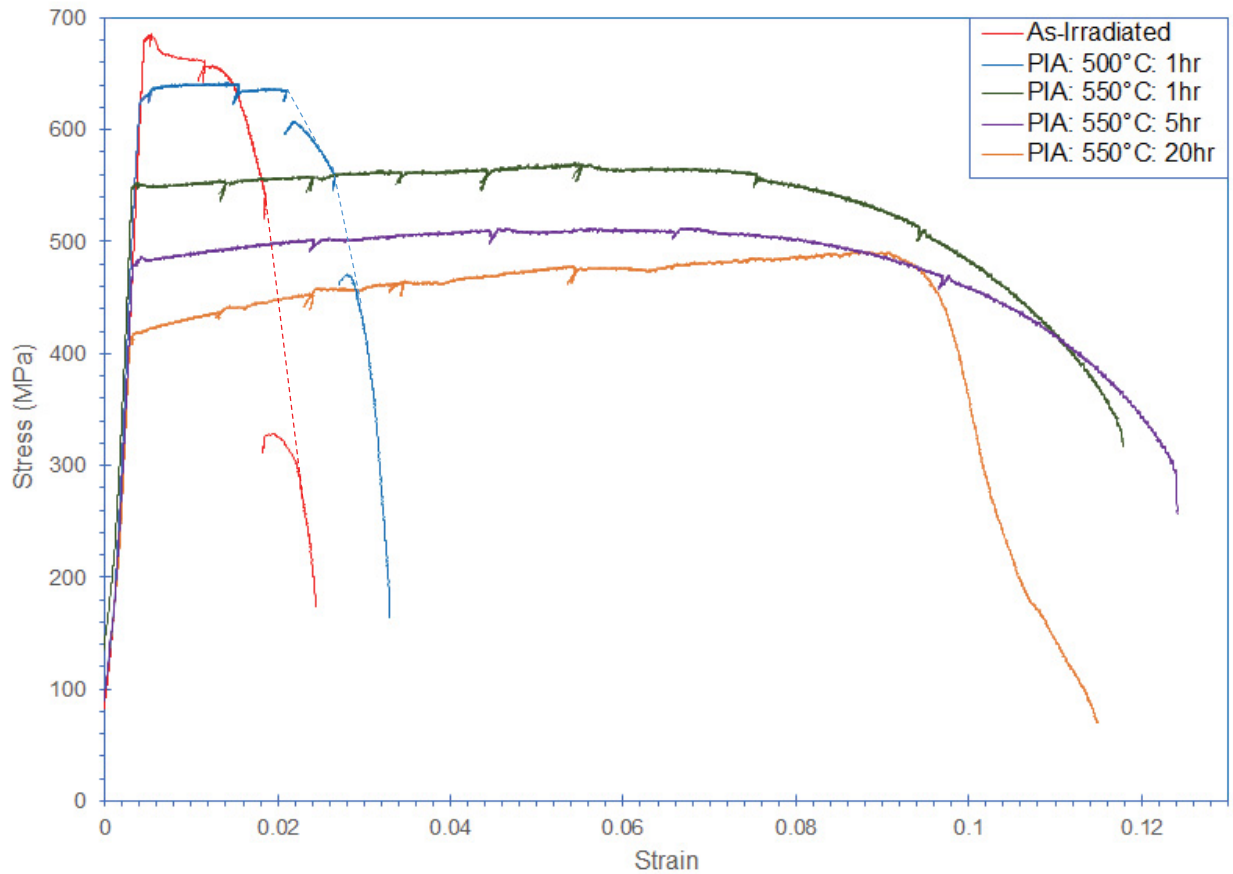


Figure 4-14: Change in the stress strain behavior because of specific annealing treatments: 500°C: 1hr, 550°C: 1hr, 550°C: 5hr, and 550°C: 20 hr.

The as-irradiated condition displayed a yield stress drop and subsequent strain softening, as typical of the irradiated condition of a solution-annealed stainless steel. Annealing at 500°C:1 hr slightly reduced the yield stress, while completely removing the yield stress drop and strain softening behavior, instead displaying an extremely slight strain hardening behavior. Annealing at 550°C: 1 and 5hr, further reduced the yield stress and resulted in a slight strain hardening behavior, with higher elongations and onset of necking at ~7% plastic strain. Annealing at 550°C:20 hr caused a further drop in the yield stress and an increase in the elongation. Furthermore, this condition displayed a clear strain hardening behavior, up to about 8.5% plastic strain.

4.3.3 Mechanical Properties

Mechanical properties of the varying annealing treatments, as determined from the CERT tests, are summarized in Table 4-9. The table is organized by annealing condition, showing the yield stress, maximum stress, uniform and total elongation. For the as-irradiated condition, uniform elongation and maximum stress both occurred at the yield point (~0.5%) because of strain softening.

Table 4-9: Change in the mechanical properties due to specific annealing treatments: 500°C: 1hr and 550°C: 20 hr.

Specimen	Yield Stress (MPa)	Maximum Stress (MPa)	Uniform Elongation (%)	Total Elongation (%)	Reduction in Area (%)
T-4 (As-Irradiated)	685.5	685.5	0.00	2.00	28.6
T-5 (500°C: 1 hr)	633.4	642.4	0.95	2.58	46.8
T-13 (550°C: 1 hr)	553.2	559.2	6.52	11.61	83.8
T-7 (550°C: 5 hr)	483.6	512.6	6.27	12.12	84.0
T-9 (550°C: 20 hr)	421.3	491.5	8.71	11.43	73.0

Overall, it was observed that the yield stress was reduced in proportion to the residual hardening following the annealing treatments. Furthermore, it was seen that the annealing treatments also lead to a change in the mechanical behavior, from a strain softening to strain hardening, and an increase in the ductility of the specimen.

4.3.4 SCC Behavior

Following CERT testing, the fracture and gage surfaces were fully examined by SEM. Areas of IG or TG cracking were quantified to determine %IG, %TG, %mixed IG/TG, and %ductile failure. Examples of each type of fracture can be seen in Figure 4-15. Reduction in area was also determined based on the full area of the fracture surface. Each sample fracture surface is shown in Figure 4-16.

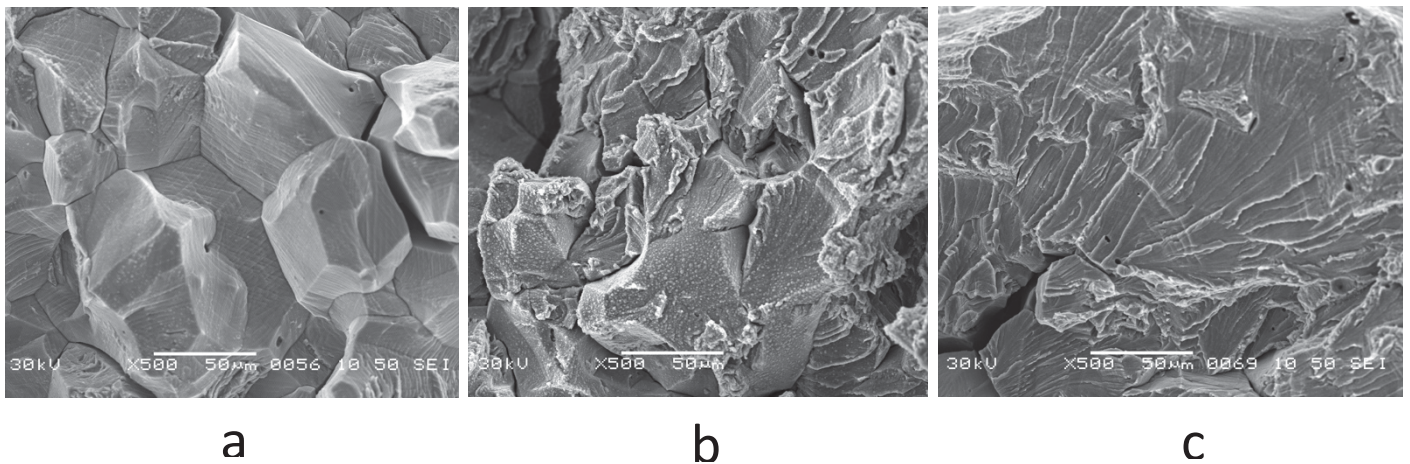
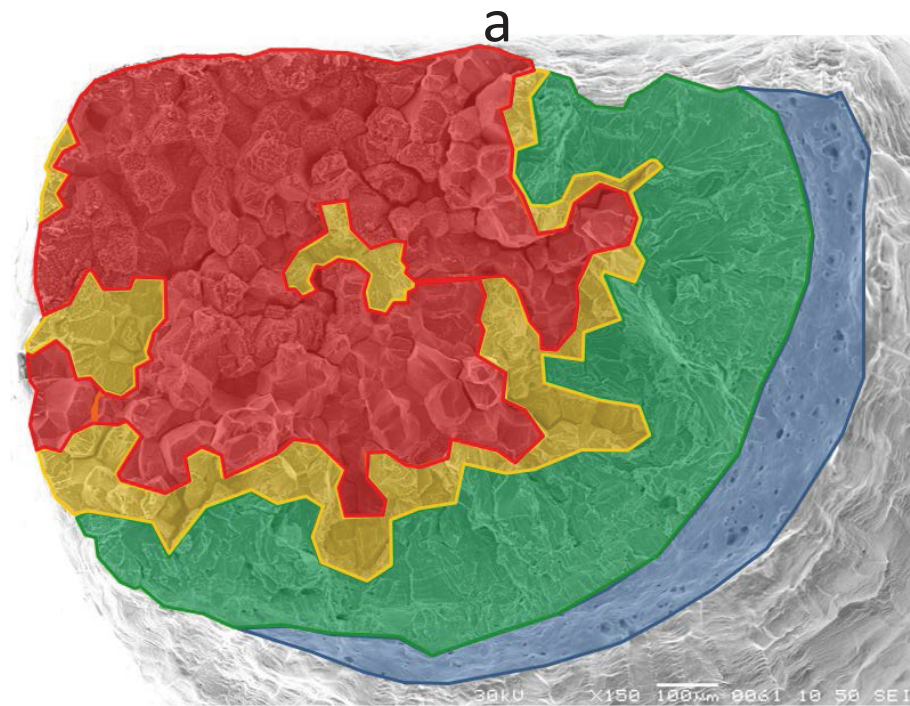
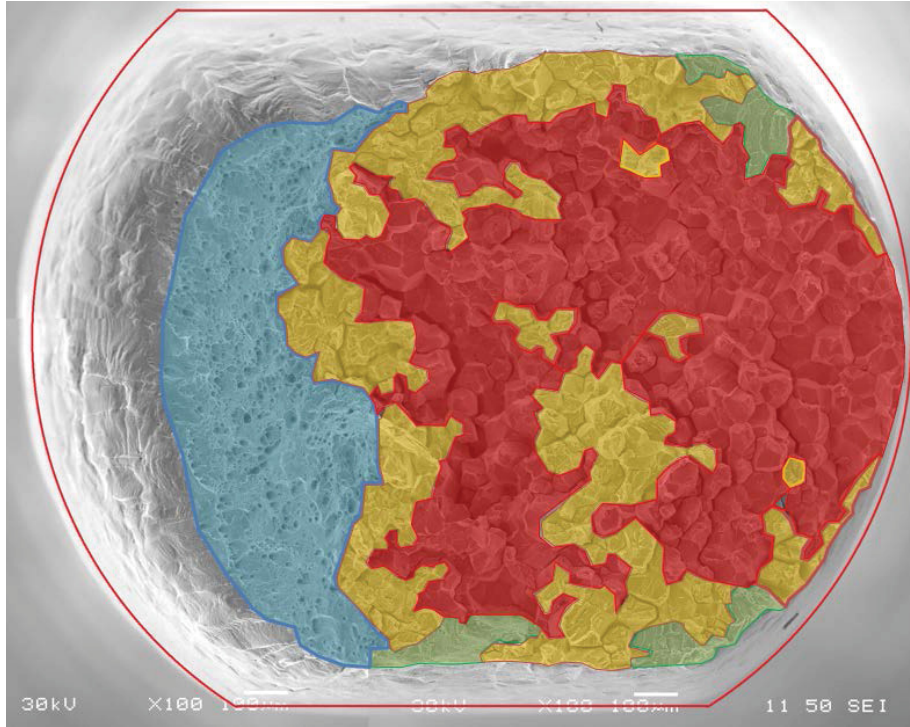
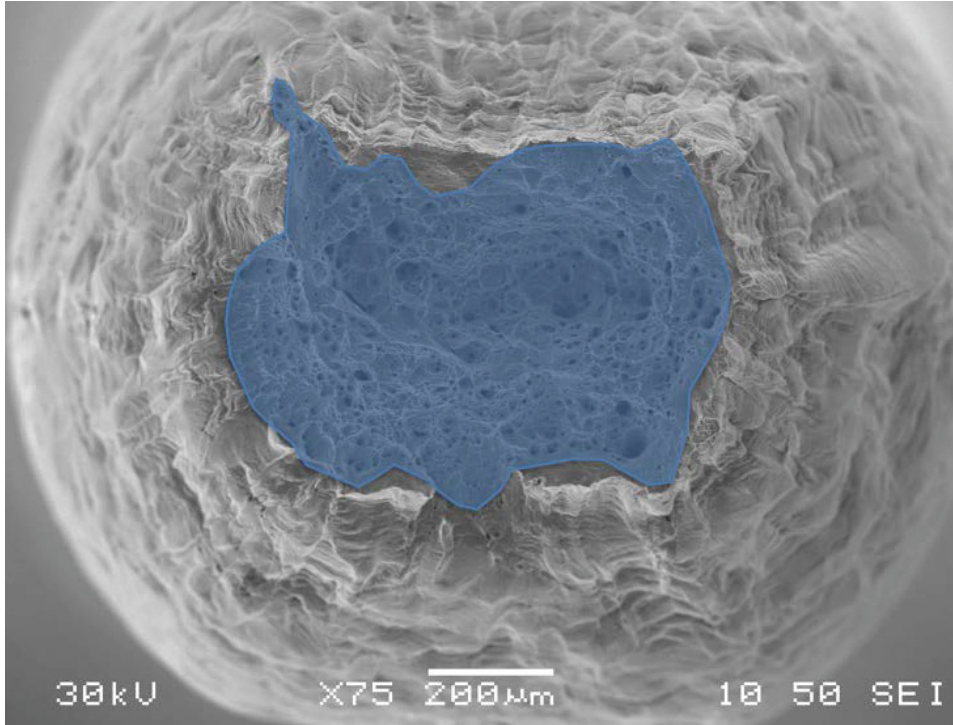


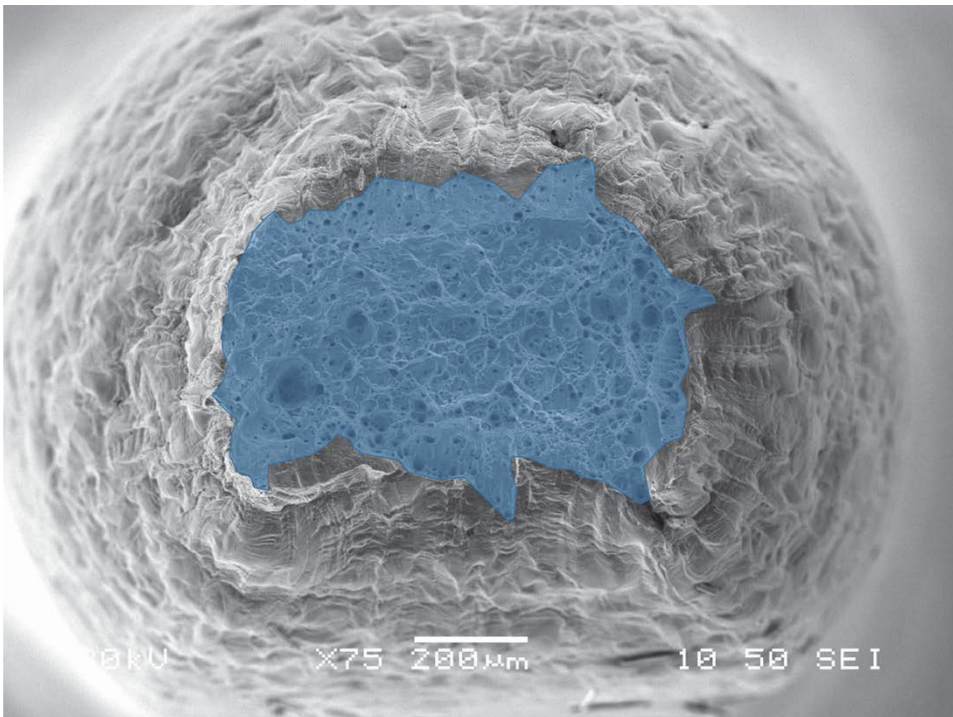
Figure 4-15: Example images from the T-5 (PIA 500°C: 1hr) specimen of the three primary modes of failure that were observed during the crack growth of the as-irradiated and PIA specimens: a) intergranular, b) mixed intergranular-transgranular, c) transgranular.



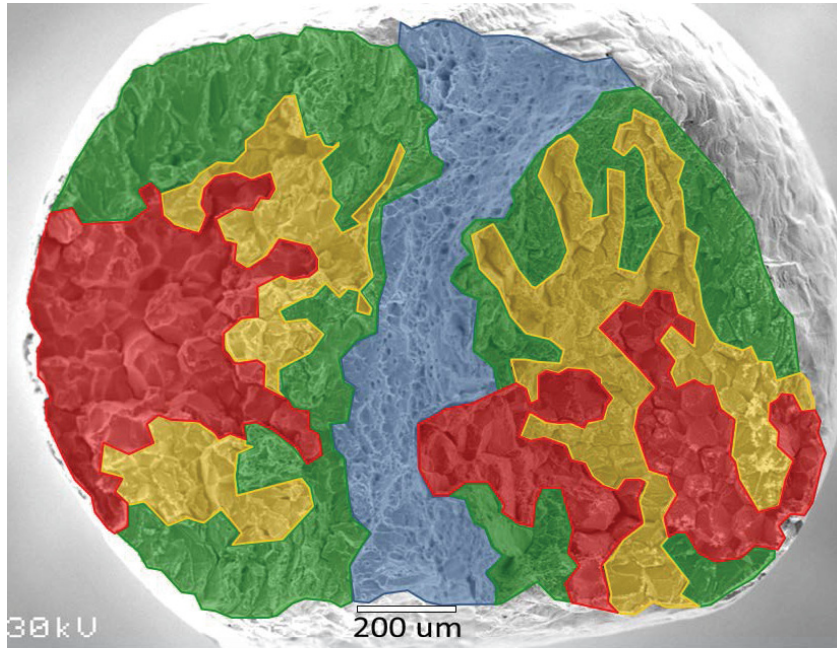
b



c



d



e

Figure 4-16: Comparison of the final fracture surface of the CERT tensile specimens: a) as-irradiated, b) PIA: 500°C: 1hr, c) PIA 550°C: 1hr, d) PIA 550°C: 5hr and e) PIA: 550°C: 20hr. Red

The results of the fractography classification and reduction of area analysis after CERT are summarized in Table 4-10 and Figure 4-17. The %IG varied between 48.4% and 0%, and was observed to largely decrease in response to increasing annealing time and temperature. However, while the IASCC susceptibility was fully removed for the 550°C: 1 and 5 hr annealed specimens, as evidenced by the fully ductile fracture surfaces, the 550°C: 20 hr specimen still displayed some residual IASCC susceptibility. Reduction in area followed a similar trend as total elongation in that it increased with annealing, however, specimen T-5 (PIA 500°C) showed a much higher reduction in area as compared to T-4 (as-irradiated), despite an only slightly higher final elongation.

Table 4-10: Change in the relative areas of fracture mode for the as-irradiated and PIA treatments: 500°C: 1hr and 550°C: 20 hr.

Specimen	%IG fracture	%Mixed fracture	%TG fracture	%Ductile fracture
T-4 (As-Irradiated)	48.40	28.62	3.62	19.41
T-5 (500°C: 1 hr)	34.86	12.82	36.58	15.74
T-13 (550°C: 1 hr)	0.00	0.00	0.00	100.00
T-7 (550°C: 5 hr)	0.00	0.00	0.00	100.00
T-9 (550°C: 20 hr)	20.60	25.04	35.28	19.08

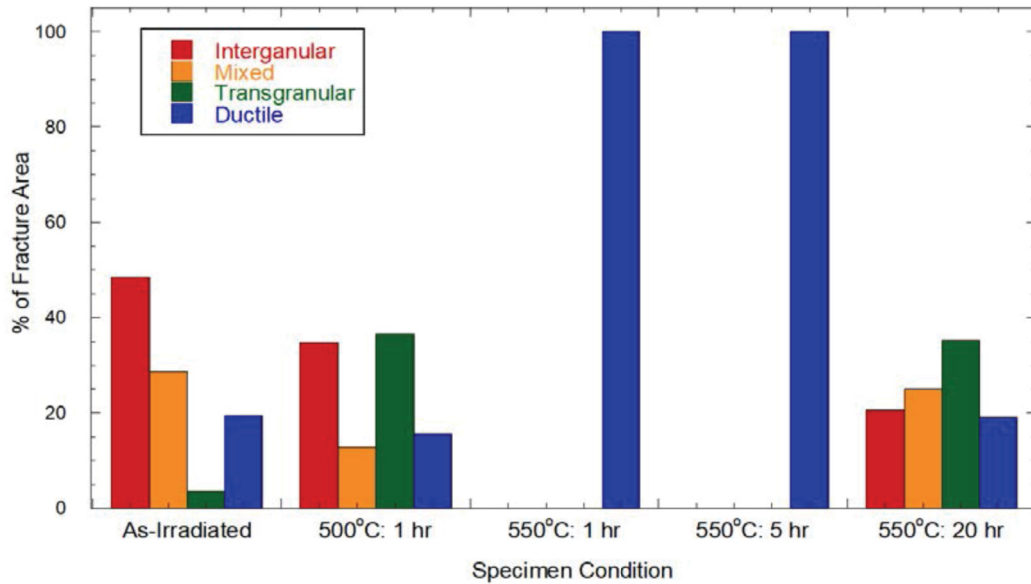


Figure 4-17: Change in the fracture morphology of the as-irradiated, PIA: 500°C: 1hr, PIA: 550°C: 1, 5, and 20 hr conditions.

4.3.5 Dislocation Channeling

As previously mentioned, the degree of localized deformation was measured via SEM following each stress/strain increment. However, due to the highly strained region that occurs during the advancement of a crack tip, measurements of the dislocation channel density were only taken for increments which displayed no significant crack growth. Furthermore, the as-irradiated material displayed an unexpected resistance to localized deformation as significant populations of dislocation channels were only observed post-yield, following the addition of bulk plastic strain. Previous literature studies for irradiated material have observed significant localized deformation below the specimen yield stress [20-22].

The measured dislocation channel densities, grain boundary interaction site densities, and ratio of discontinuous to continuous interaction sites for each strain increment are shown in Table 4-11. As multiple specimens exhibited regions of enhanced strain, where the eventual crack initiation later occurred, the measured channel densities are averaged over this region (~1mm of gage length) where later crack initiation was observed, rather than over the entire gage length. Comparison of the dislocation channel density with strain for the examined conditions: as-irradiated, 500°C: 1hr, 550°C: 1, 5, and 20hr, is shown in Figure 4-18. The density of dislocation channel-grain boundary interaction sites is given in Figure 4-19, while the ratio of discontinuous to continuous interaction sites is presented in Figure 4-20.

It is observed that the as-irradiated, PIA: 500°C: 1hr, and PIA: 550°C: 1 hr conditions have very similar dislocation channel densities, while the 550°C: 5 hr sample exhibited a decrease in the density. The 550°C: 20hr had a significantly reduced density regardless of the plastic strain. The interaction site density seems to change in proportion to the total dislocation channel density regardless of the annealing condition.

Table 4-11: Change in the dislocation channel and interaction site densities in the failure region. Also compared is the percentage of discontinuous sites. A plastic strain of 0.00 corresponds to the density observed at the yield stress.

Specimen	Plastic Strain (%)	Channel Density (#/mm ²)	Continuous Site Density (#/mm ²)	Discontinuous Site Density (#/mm ²)	Discontinuous/Continuous site ratio
T-4 (As-Irradiated)	0.00	121 ± 19	21.2 ± 7.3	139 ± 22	6.5
	0.74	1337 ± 169	332 ± 67	1747 ± 229	5.3
T-5 (500°C: 1 hr)	0.00	3.5 ± 2.9	0.0 ± 0.0	6.9 ± 5.8	-
	1.00	1903 ± 246	239 ± 99	3019 ± 424	12.7
	1.50	2462 ± 232	307 ± 78	3454 ± 553	11.2
	2.00	2718 ± 297	325 ± 116	4239 ± 553	13.1
T-13 (550°C: 1 hr)	0.00	180 ± 48	42 ± 18	230 ± 66	5.5
	1.00	2448 ± 228	398 ± 68	3540 ± 520	8.9
	2.00	2602 ± 153	451 ± 51	3618 ± 225	8.0
	3.00	2737 ± 274	417 ± 48	4010 ± 446	9.6
	4.00	2869 ± 206	427 ± 95	4201 ± 306	9.8
	5.00	2856 ± 192	405 ± 65	4105 ± 296	10.1
	7.00*	5099 ± 344	765 ± 115	7365 ± 498	9.6
T-7 (550°C: 5 hr)	0.00	99 ± 38	14 ± 12	151 ± 58	10.9
	2.00	2137 ± 220	262 ± 69	3289 ± 332	12.6
	4.00	2617 ± 376	405 ± 132	3891 ± 579	9.6
	6.00*	3657 ± 494	438 ± 79	5588 ± 736	12.8
T-9 (550°C: 20 hr)	0.00	8.8 ± 5.5	0.0 ± 0.0	16 ± 10	-
	1.00	132 ± 46	21 ± 13	190 ± 61	9.1
	2.00	174 ± 76	44 ± 38	260 ± 81	5.9
	3.00	499 ± 115	100 ± 49	694 ± 132	6.9
	5.00	585 ± 136	107 ± 56	835 ± 156	7.8

*Specimens T-7 and T-13 were observed to begin to neck at $\sim 6\% \epsilon_p$, thus the sharp increase in dislocation channel density is believed to be a direct result of the necking, and does not accurately represent the changing localized deformation with strain.

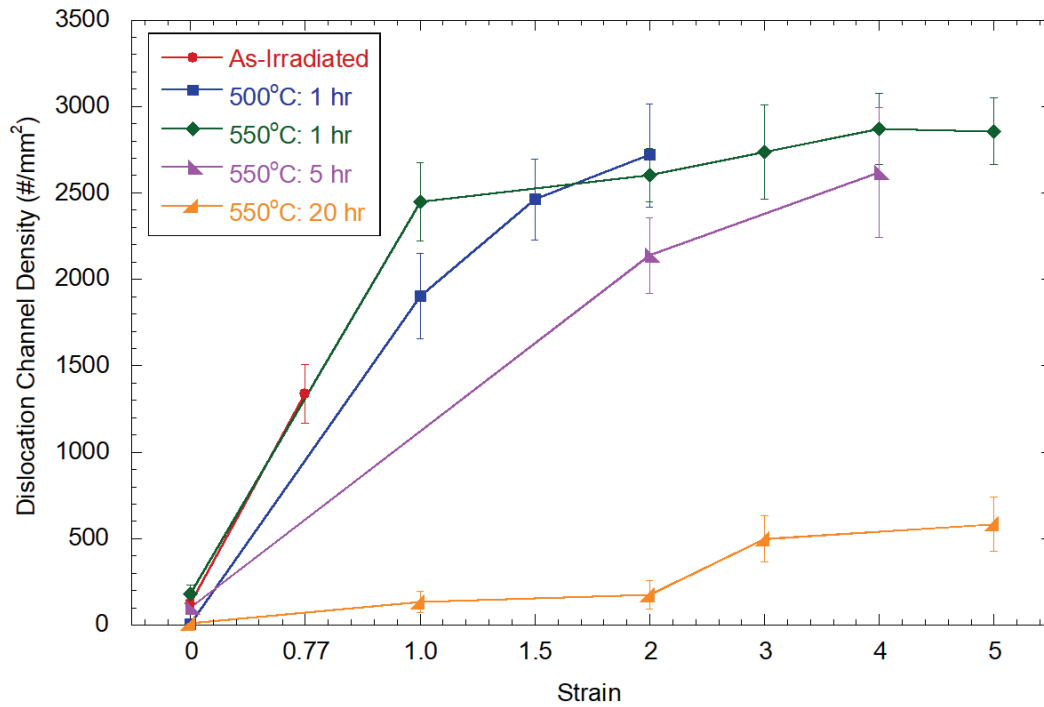


Figure 4-18: Change in the dislocation channel density of the failure region in response to increasing plastic strain, prior to crack growth or specimen necking.

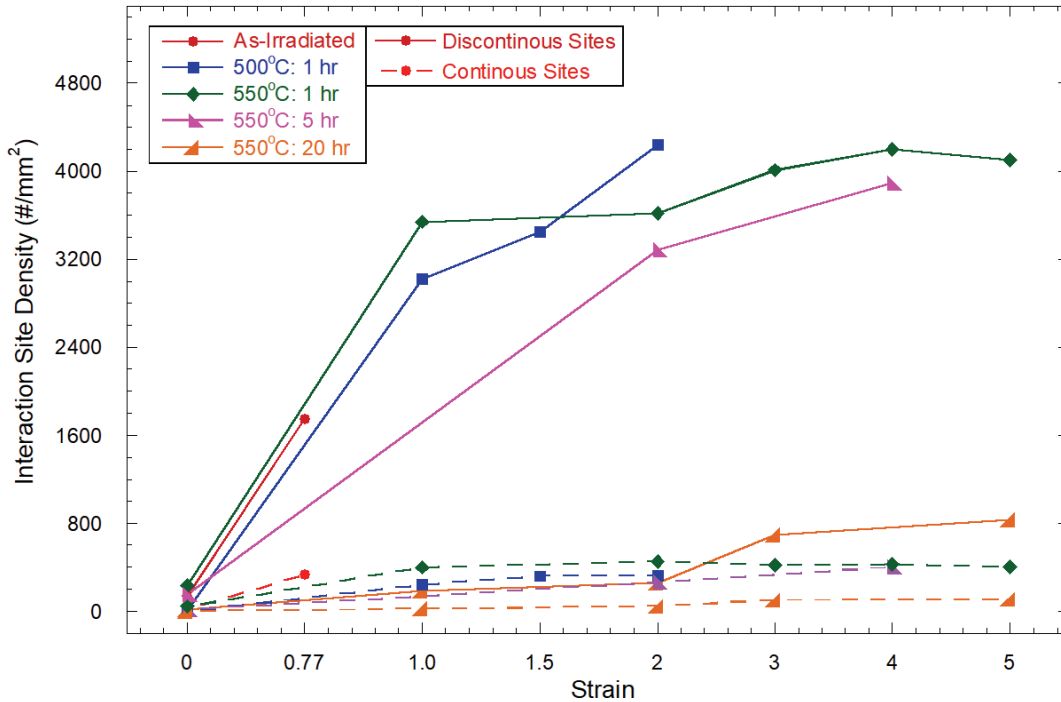


Figure 4-19: Change in the grain boundary-dislocation channel interaction site density of the failure region in response to increasing plastic strain.

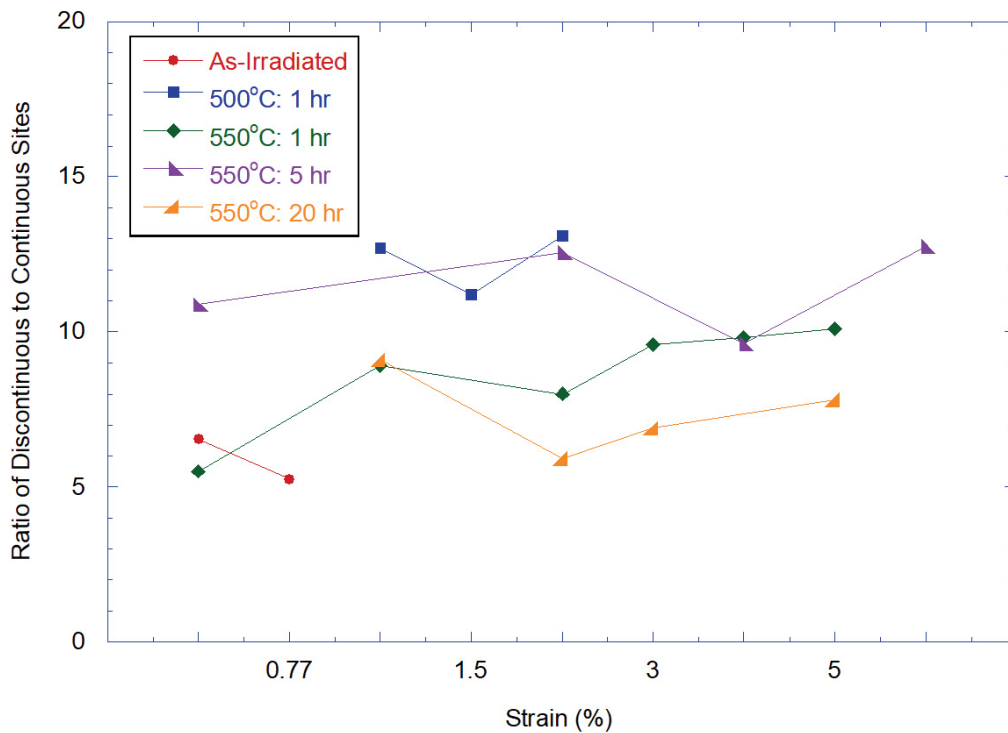


Figure 4-20: Change in the ratio of discontinuous to continuous grain boundary-dislocation channel interaction sites.

4.4 Crack initiation in CERT tests on neutron-irradiated CIR alloys

This section presents a summary of the CERT test results obtained from the BOR-60 irradiated tensile bars. CERT tests were conducted in one of three environments, 288°C BWR NWC, 288°C BWR HWC, or 320°C PWR PW, straining each sample until complete failure. The stress-strain curves of each sample are first presented with comments about the observed mechanical behavior. A summary of the mechanical properties is then presented followed by images of each fracture surface after completion of the CERT tests. More detailed information from each of the fracture surfaces and higher magnification images are presented in the appendices of previous semi-annual reports (January 2012, July 2012, January 2013, and July 2013). Finally, a summary of the fractographic analysis is presented.

4.4.1 Stress-Strain Curves

Stress-strain curves for each alloy are shown in Figure 4-21, Figure 4-22 and Figure 4-23 for samples tested in 288°C BWR NWC, 288°C BWR HWC, and 320°C PWR PW, respectively. The elastic deformation portion of each curve has been corrected to subtract system compliance, using the technique described in section 3.3.2.

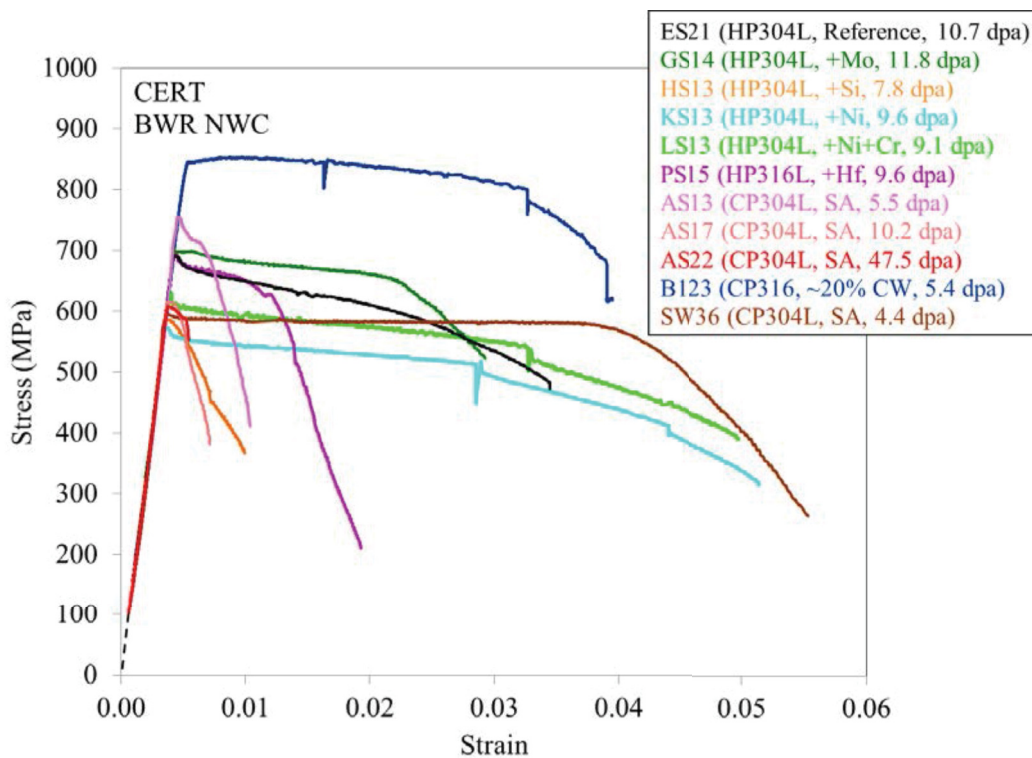


Figure 4-21 Stress-strain curves of neutron irradiated specimens tested in BWR NWC.

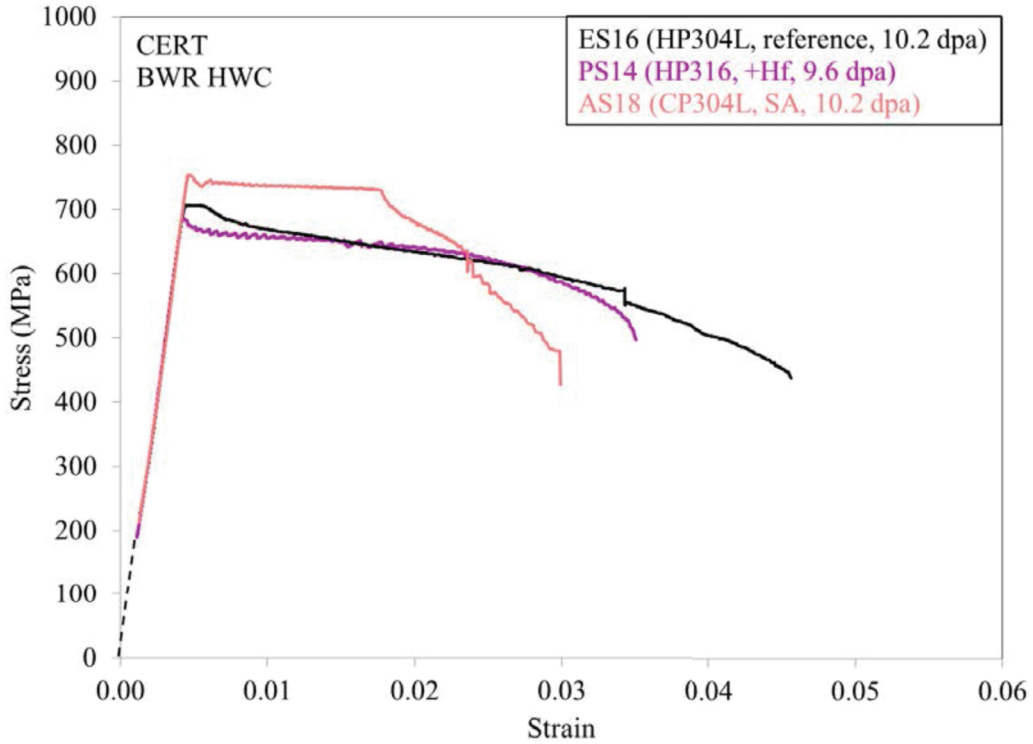


Figure 4-22 Stress-strain curves of neutron irradiated specimens tested in BWR HWC.

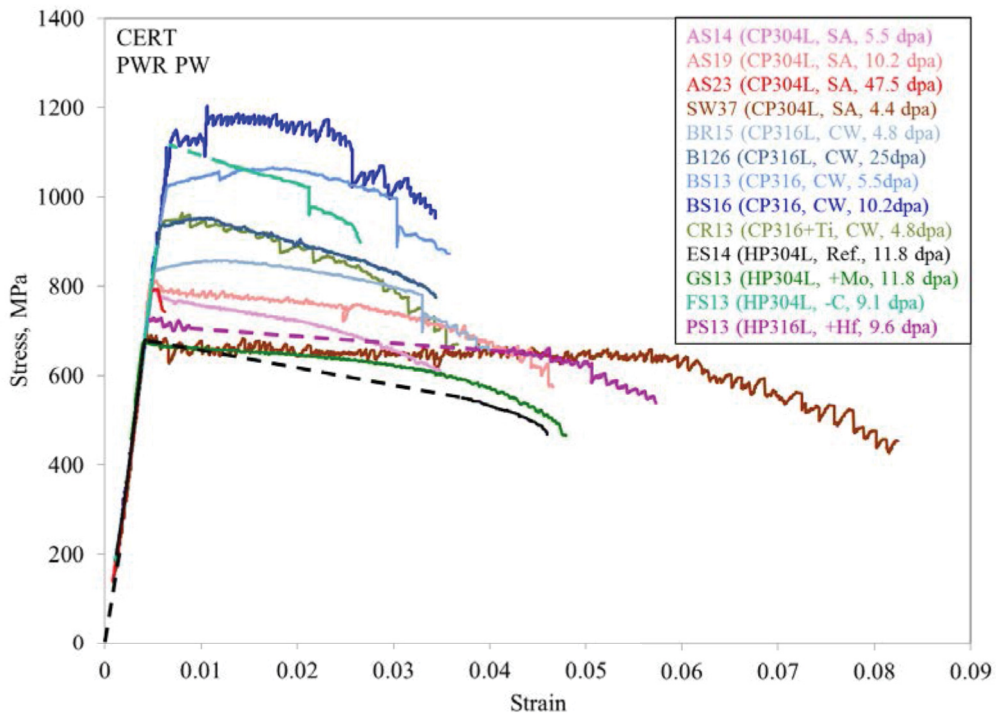


Figure 4-23 Stress-strain curves of neutron irradiated specimens tested in PWR PW.

All alloys in the solution annealed condition prior to irradiation exhibited strain softening, therefore the yield stress equaled the maximum stress, and uniform elongation consisted of only elastic deformation. Also common to solution annealed alloys was the observation of a sharp drop in stress upon yielding, likely caused by dislocation channeling [23]. When a large quantity of dislocation channels form at the yield point, the local strain rate of the sample exceeds the system strain rate and a drop in stress occurs. In general, increasing irradiation dose caused an increase in YS for solution annealed alloys, with the exception of those with very high SCC susceptibility, which failed during elastic deformation and did not achieve their full expected YS (i.e., samples AS17, AS22, AS23, HS13).

All alloys in the cold worked condition prior to irradiation exhibited a small degree of strain hardening, even up to 25 dpa. None of the CW alloys exhibited a stress drop at the yield point, likely because of the dislocation barriers remaining from the cold work. Alloy BS had a higher degree of cold work than alloy BR or B, as the increased YS suggests. Increasing irradiation dose also caused an increase in YS for the cold worked alloys.

Solute addition alloys exhibited similar YS between ~600 and ~700 MPa, as expected due to the similar microstructures created prior to irradiation. Sample FS13 was the only exception, having a much higher yield stress than the other solute addition alloys. The YS and maximum stress of this sample were not recorded due to an error in data collection, but were estimated to be 1140 MPa by extrapolating the stress-strain curve pre- and post- yield point. Regions where the gap in data exists due to the collection error for samples FS13, ES14, and PS13 are indicated by dashed lines in Figure 4-23. The high YS of sample FS13 implies that either the alloy was cold worked prior to irradiation or the irradiation dose was higher than the reported 9.1 dpa.

Substantial variations were observed in the elongation of the irradiated samples, but several trends were observed. In general, elongation was greater in environments with lower corrosion potential (samples had greater elongation in PW than in HWC, and both were greater than in NWC). The high purity solute addition alloys had generally greater elongation than the commercial purity alloys, although those with cold work had consistently intermediate elongations and little variation as a result of varying degree of cold work or irradiation dose. The only exception was alloy SW, which had very high elongation in both NWC and PW environments. The cause of the high elongation of alloy SW is unknown due to the lack of processing history for this alloy.

4.4.2 Mechanical Properties

Mechanical properties determined from the CERT tests are summarized in Table 4-12. The table is organized by experiment, showing the test environment, samples tested (including alloy and dose) and results from each including the YS, maximum stress, uniform and total elongation.

Table 4-12 Mechanical properties from CERT tests.

Test ID	Environment	Sample ID (alloy, dpa)	Yield Stress, MPa	Yield Stress in 330°C Air, MPa ^a	Maximum Stress, MPa	Uniform elongation, % ^b	Total Elongation, % ^b
CI-01	BWR NWC (288° C)	K-4 (CP304L, 0)	687	NM	722	1.50	6.10
		ES21 (HP304L, 10.7)	691	712	691	0.42	3.45
CI-02	BWR NWC (288° C)	K-5 (CP304L, 0)	805	NM	810	0.65	2.78
		GS14 (HP304L+Mo, 11.8)	705	745	705	0.43	2.93
		PS15 (HP316L+Hf, 9.6)	696	688	696	0.43	1.93
		HS13 (HP304L+Si, 7.8)	587	829	587	0.36	0.99
CI-03	BWR NWC (288° C)	K-6 (CP304L, 0)	867	NM	888	1.15	1.43
		KS13 (HP304L+Ni, 9.6)	574	606	574	0.35	5.14
		LS13 (HP304L+Ni+Cr, 9.1)	633	NM	633	0.39	4.98
		B123 (CW CP316L, 5.4)	843	861	855	0.97	3.94
CI-04	BWR NWC (288° C)	SW36 (CP304L, 4.4)	602	623	602	0.36	5.53
		AS13 (CP304L, 5.5)	755	784	755	0.45	1.04
		AS17 (CP304L, 10.2)	616	825	616	0.37	0.72
		AS22 (CP304L, 47.5)	608	907	608	0.37	0.54
CI-05	BWR HWC (288° C)	PS14 (HP316L, 9.6)	687	688	687	0.42	3.50
		AS18 (CP304L, 10.2)	754	825	754	0.46	2.99
		K-4 (CP304L, 0)	665	NM	682	1.08	5.37
		ES16 (HP304L, 10.2)	707	712	707	0.45	4.56
CI-06	PWR PW (320° C)	SW37 (CP304L, 4.4)	695	623	695	0.42	8.24
		AS14 (CP304L, 5.5)	794	784	794	0.48	3.53
		AS19 (CP304L, 10.2)	813	825	813	0.49	4.65
		AS23 (CP304L, 47.5)	793	907	793	0.48	0.62
CI-07	PWR PW (320° C)	B126 (CW CP316L, 25)	916	947	953	1.07	3.44
		BR15 (CW CP316L, 4.8)	840	861	858	1.18	3.99
		K-5 (CP304L, 0)	889	NM	935	1.83	4.82
CI-08	PWR PW (320° C)	BS13 (CW CP316L, 5.5)	1023	1056	1065	1.76	3.58
		BS16 (CW CP316L, 10.2)	1126	1087	1204	1.07	3.43
		CR13 (CW CP316L, 4.8)	945	949	963	0.80	3.67
		K-5 (CP304L, 0)	967	NM	996	1.18	3.61
CI-09	PWR PW (320° C)	ES14 (HP304L, 11.8)	681	712	681	0.41	4.60
		GS13 (HP304L+Mo, 11.8)	676	745	676	0.41	4.79
		FS13 (HP304L-C, 9.1)	1140 ^c	594	1140 ^c	NA ^c	2.65
		PS13 (HP316L+Hf, 9.6)	730	688	730	0.44	5.74

^aData reproduced from [22]

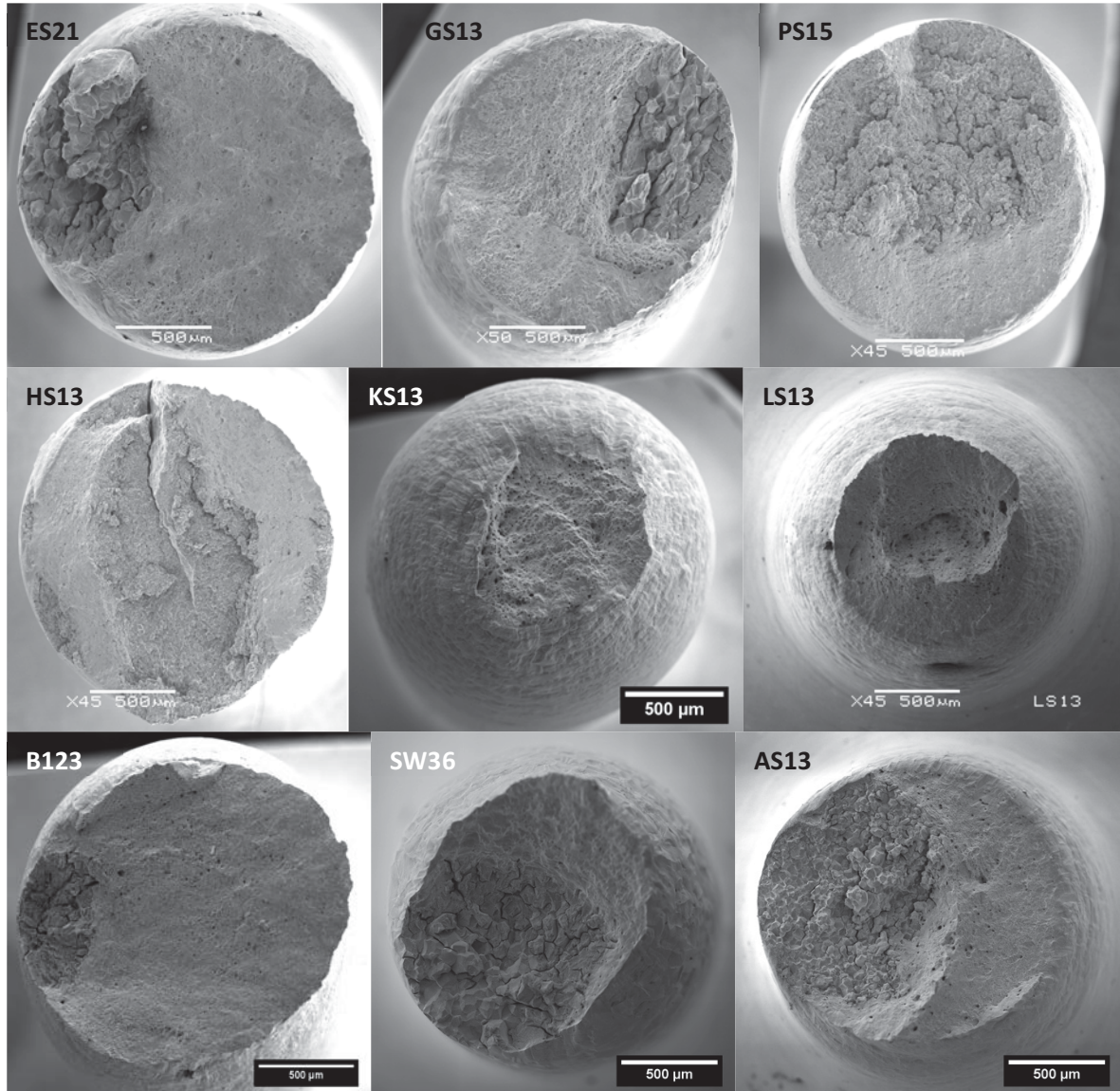
^bBased on the original gage length of 12 mm.

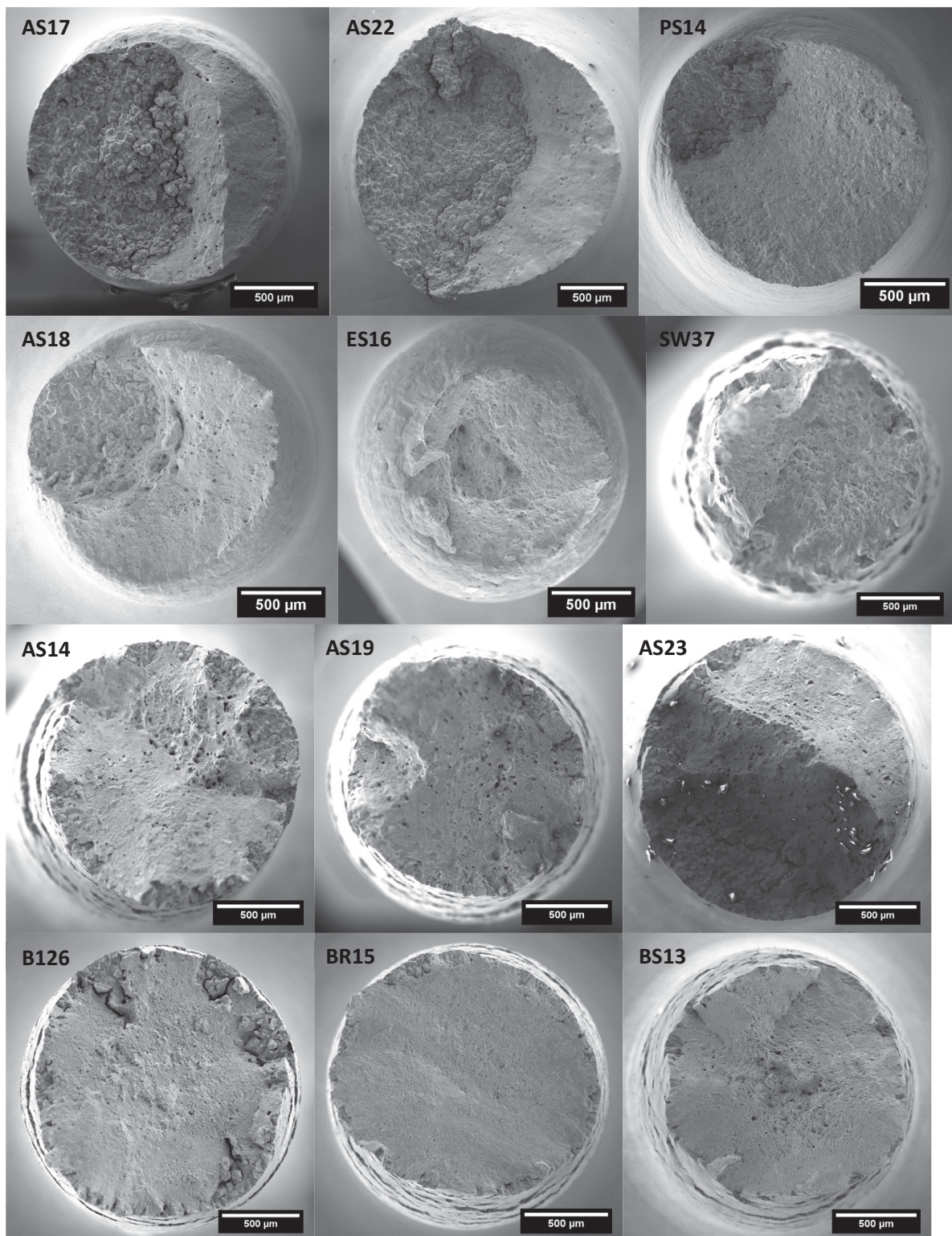
^cData not collected, values were not available or estimated by extrapolation of pre- and post-yield stress-strain data.

For all irradiated samples in the solution annealed condition, uniform elongation was the strain at the yield point (~0.5%) because of strain softening. Irradiated samples with prior cold work all displayed a small amount of strain hardening and limited amounts of uniform elongation, never exceeding 2%. Despite the small uniform elongation in most cases, a wide range in TE (0.54% to 8.24%) was observed. In general, TE increased in HWC and PW environments compared to NWC. TE decreased with increasing dose, although CW materials showed more consistent TE with variations in dose.

4.4.3 SCC Behavior

Following CERT testing, the fracture and gage surfaces were fully examined by SEM. Areas of IG or TG cracking were quantified to determine %IG, %TG, or % mixed IG/TG. Reduction in area was also determined based on the full area of the fracture surface. Each sample fracture surface is shown in Figure 4-24. Although not shown, gage surfaces were fully investigated for the existence of secondary cracks and other defects such as pitting. Previous semi-annual reports (January 2012, July 2012, January 2013, July 2013) contain all gage surface images as well as higher magnification investigation of each fracture surface.





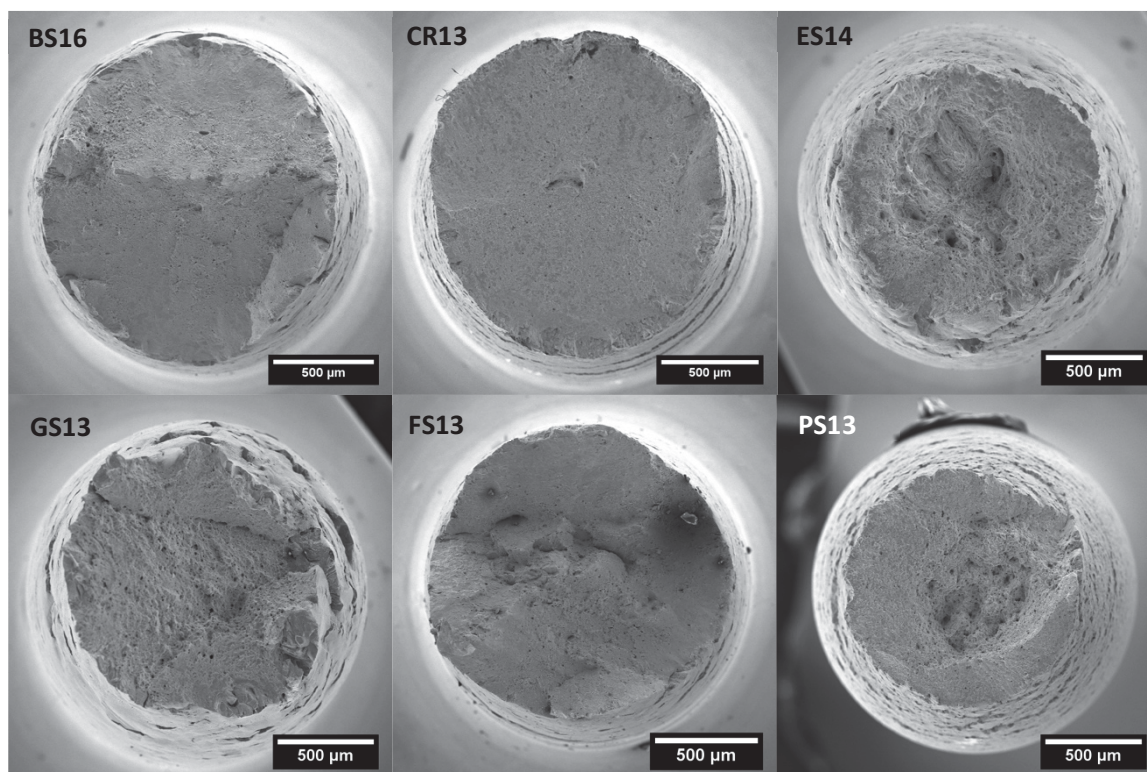


Figure 4-24 Fracture surfaces of samples strained to failure during CERT tests.

The results of the fractography and reduction of area analysis after CERT are summarized in Table 4-13. Commercial purity alloy AS samples were the most susceptible to IASCC in all test environments. High purity solute addition alloys were less susceptible than the CP alloys, and not susceptible to IGSCC in the PW environment. Similar to the results from the CERT tests in NWC, alloy PS was the most susceptible solute addition alloy in the PW environment. Cold work apparently reduced the amount of IG cracking in both NWC and PW. BS alloys with presumably the highest amount of cold work were the least susceptible of the cold worked alloys.

%IG varied between 0% and 57.5% for all irradiated samples. In general, %IG was higher in NWC than in HWC or PW for a given alloy. %IG was accepted as the best indicator of IASCC susceptibility, and is used to describe relative differences in cracking susceptibility throughout the discussion chapter.

Reduction of area followed a similar trend as total elongation, but could not be calculated for sample AS22 due to failure at the sample shoulder where the initial cross sectional area was unknown. Typically, either measure of ductility (TE or %RA), followed the inverse trend of %IG.

Table 4-13 Reduction in area and percentage of crack type on the fracture surface.

Test ID	Environment (°C)	Sample (Alloy, dpa)	RA (%)	%IG	%Mixed IG/TG	%Ductile
CI-01	BWR NWC (288)	K-4 (CP304L, 0)	49.7	0.0	12.8 ^a	87.2
		ES21 (HP304L, 10.7)	39.2	16.8	7.4	75.8
CI-02	BWR NWC (288)	K-5 (CP304L, 0)	36.8	0.0	13.4 ^a	86.6
		GS14 (HP304L+Mo, 11.8)	38.6	16.9	9.1	74.1
		PS15 (HP316L+Hf, 9.6)	31.8	50.5	8.4	41.1
		HS13 (HP304L+Si, 7.8)	13.3	42.8	14.2	43.1
CI-03	BWR NWC (288)	K-6 (CP304L, 0)	20.8	0.0	12.9 ^a	87.1
		KS13 (HP304L+Ni, 9.6)	78.5	0.0	0.0	100.0
		LS13 (HP304L+Ni+Cr, 9.1)	64.5	0.0	0.0	100.0
CI-04	BWR NWC (288)	B123 (CW CP316L, 5.4)	40.7	4.2	10.5	89.5
		SW36 (CP304L, 4.4)	58.8	29.2	32.5	38.3
		AS13 (CP304L, 5.5)	45.3	40.7	0.0	59.3
		AS17 (CP304L, 10.2)	39.9	55.9	0.0	44.1
CI-05	BWR HWC (288)	AS22 (CP304L, 47.5)	NA	57.5	0.0	42.5
		PS14 (HP316L, 9.6)	37.9	16.9	0.0	83.1
		AS18 (CP304L, 10.2)	41.6	23.4	0.0	76.6
		K-4 (CP304L, 0)	65.6	0.0	0.0	100.0
CI-06	PWR PW (320)	ES16 (HP304L, 10.2)	64.1	0.0	0.0	100.0
		SW37 (CP304L, 4.4)	52.2	5.2	0.0	94.8
		AS14 (CP304L, 5.5)	38.7	20.4	0.0	79.6
		AS19 (CP304L, 10.2)	46.7	14.0	0.0	86.0
CI-07	PWR PW (320)	AS23 (CP304L, 47.5)	20.2	28.4	0.0	71.6
		B126 (CW CP316L, 25)	41.4	22.4	0.0	77.6
		BR15 (CW CP316L, 4.8)	53.8	10.9	0.0	89.1
CI-08	PWR PW (320)	K-5 (CP304L, 0)	51.7	0.0	1.4	98.6
		BS13 (CW CP316L, 5.5)	44.5	3.7	0.0	96.3
		BS16 (CW CP316L, 10.2)	42.1	3.9	0.0	96.1
		CR13 (CW CP316L, 4.8)	36.2	9.6	0.0	90.4
CI-09	PWR PW (320)	K-5 (CP304L, 0)	48.0	0.0	0.6 ^a	99.4
		ES14 (HP304L, 11.8)	57.4	0.0	1.6 ^a	98.4
		GS13 (HP304L+Mo, 11.8)	50.4	0.0	11.9	88.1
		FS13 (HP304L-C, 9.1)	42.5	0.0	1.3	98.7
		PS13 (HP316L+Hf, 9.6)	61.8	1.8	0.0	98.2

^aOnly TG type cracking was observed.

4.4.4 Step Height Measurement

Dislocation channel step heights were measured on several select samples after failure during CERT testing. Measurements were taken from replicas made of the gage surfaces after oxide removal had been performed. Randomly selected grains near the IG fracture edge were measured,

because these regions were believed to be IG crack initiation sites. A summary of the dislocation channel height measurement is shown in 14, along with select CERT test results for comparison. Error values for the weighted average channel height were calculated based on the standard deviations calculated from each measured channel height.

Table 4-14 Dislocation channel height analysis from sample replicas, with CERT test results for comparison.

Sample ID	Dose, dpa	TE, % (air)	YS, MPa	TE, %	%IG	Grains Analyzed	Channels Analyzed	Weighted Avg. Channel Height, nm
AS13	5.5	8.4	755	1.04	40.7	16	130	291 ± 57
AS17	10.2	8.0	616	0.72	55.9	9	82	364 ± 67
AS18	10.2	8.0	754	2.99	23.4	11	130	507 ± 93
AS22	47.5	7.8	608	0.54	57.5	7	25	1342 ± 497
ES21	10.7	7.6	691	3.45	16.8	10	136	265 ± 34
KS13	9.6	10.7	574	5.14	0.0	11	146	541 ± 127

Dislocation channel height varied substantially among the six samples. Due to the low elongation during CERT, samples AS17 and AS22 had very few visible channels, limiting the number available for analysis. The gage surfaces of samples AS13 and AS22 are compared in Figure 4-25 as an example. Weighted average channel height decreased in the order: AS22, AS18, AS17, AS13 for commercial purity alloys. The high purity ES21 sample had the lowest channel height of all samples, and the KS13 sample with no IG cracking had a step height of nearly double that of sample ES21. Because of the lack of IG cracking, step height measurements were taken in the necked region of the gage surface on KS13, which likely caused the increased channel height.

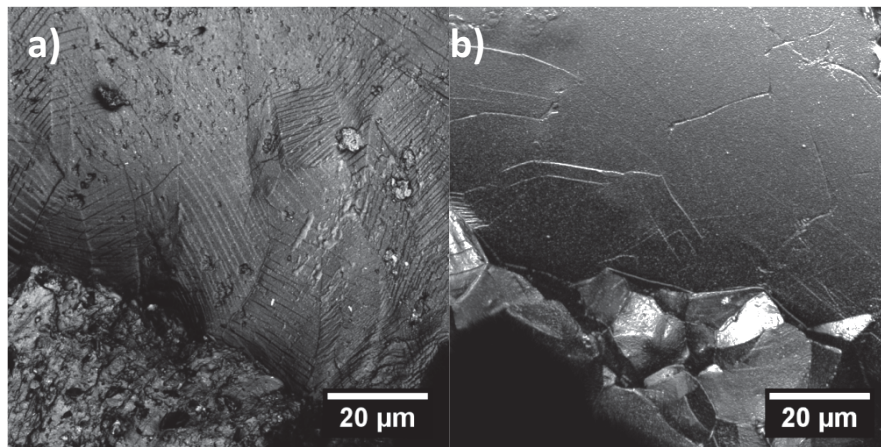


Figure 4-25 Surface topography of the gage surfaces of a) AS13 and b) AS22. The IG fracture edge is visible at the bottom of each image.

4.5 Four-Point Bend Test Results

To describe and qualify the strain state created during the four point bend test, results of the FEA model are first presented and compared with unirradiated bend test results in RT air, 288°C Ar gas, and 288°C NWC. Results from irradiated samples are then summarized, while discussing the capability of the bend test to create IASCC initiation with limited crack propagation, and how bend test results compare to more traditional SCC initiation test techniques. Results will focus on the convex surface of the bend sample, where the tensile stress/strain state exists. Images are presented with the sample oriented as shown in Figure 4-26, with the tensile (ϵ_{xx}) direction left to right.

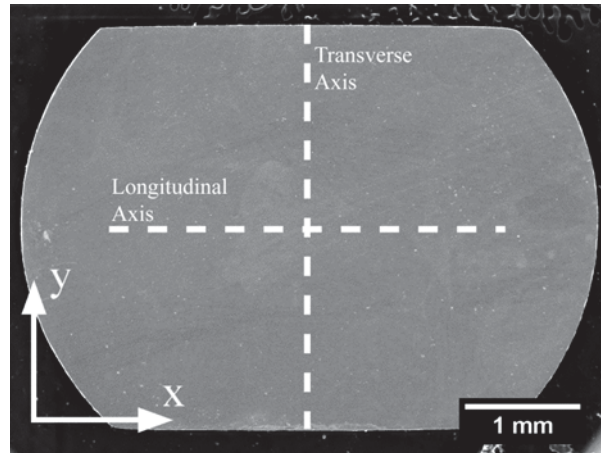


Figure 4-26. Sample orientation and coordinate system. Dashed lines indicate axes for strain profiles.

4.5.1 FEA results

A full description of the in-plane surface strain for the 16.9% CW condition at a sample deflection of 58.3 μm is shown in Figure 4-27. This amount of deflection was chosen as it gives a description of the strain state after a significant amount of plastic deformation. The xx component of the strain tensor accounted for the majority of the surface strain, however, strain in the yy component was observed near the edges of the sample. Very little shear strain (xy component) was created on the sample surface. A more quantified view of the surface strain as a function of deflection for the 16.9% CW condition is shown in Figure 4-28. The main difference observed by changing material condition was in the response of the xx component of strain, which is shown for the AR condition in Figure 4-29. Because of the lower yield stress, strain peaks under the loading points become larger, and are likely a result of additional tensile stress formed by the frictional constraint at the lower sample supports due to the small sample size. This likelihood was confirmed by performing FEA simulations with an increased coefficient of friction between the sample and supports. With an increased coefficient of friction, the formation of strain peaks was observed at the same locations but at smaller sample deflection.

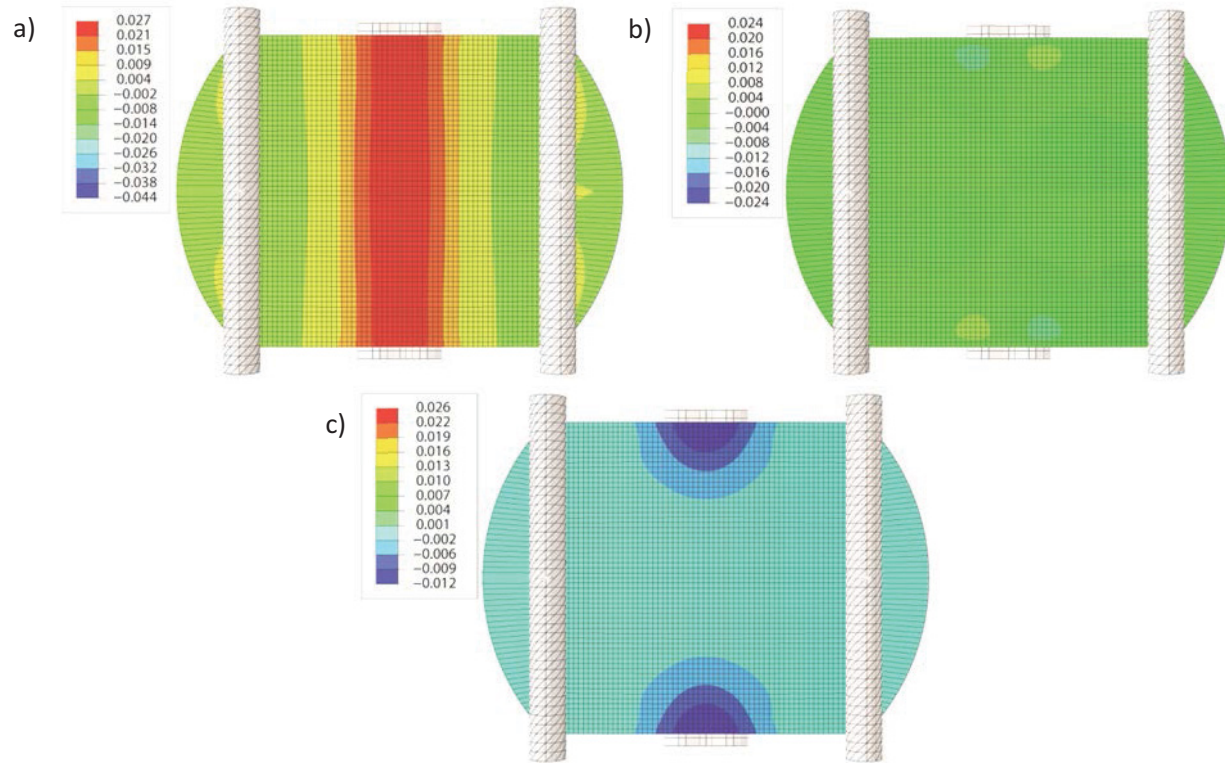


Figure 4-27. Color representations of total strain (elastic plus plastic) components a) ϵ_{xx} , b) ϵ_{xy} , c) ϵ_{yy} . 16.9% CW condition after a sample deflection of 58.3 μm .

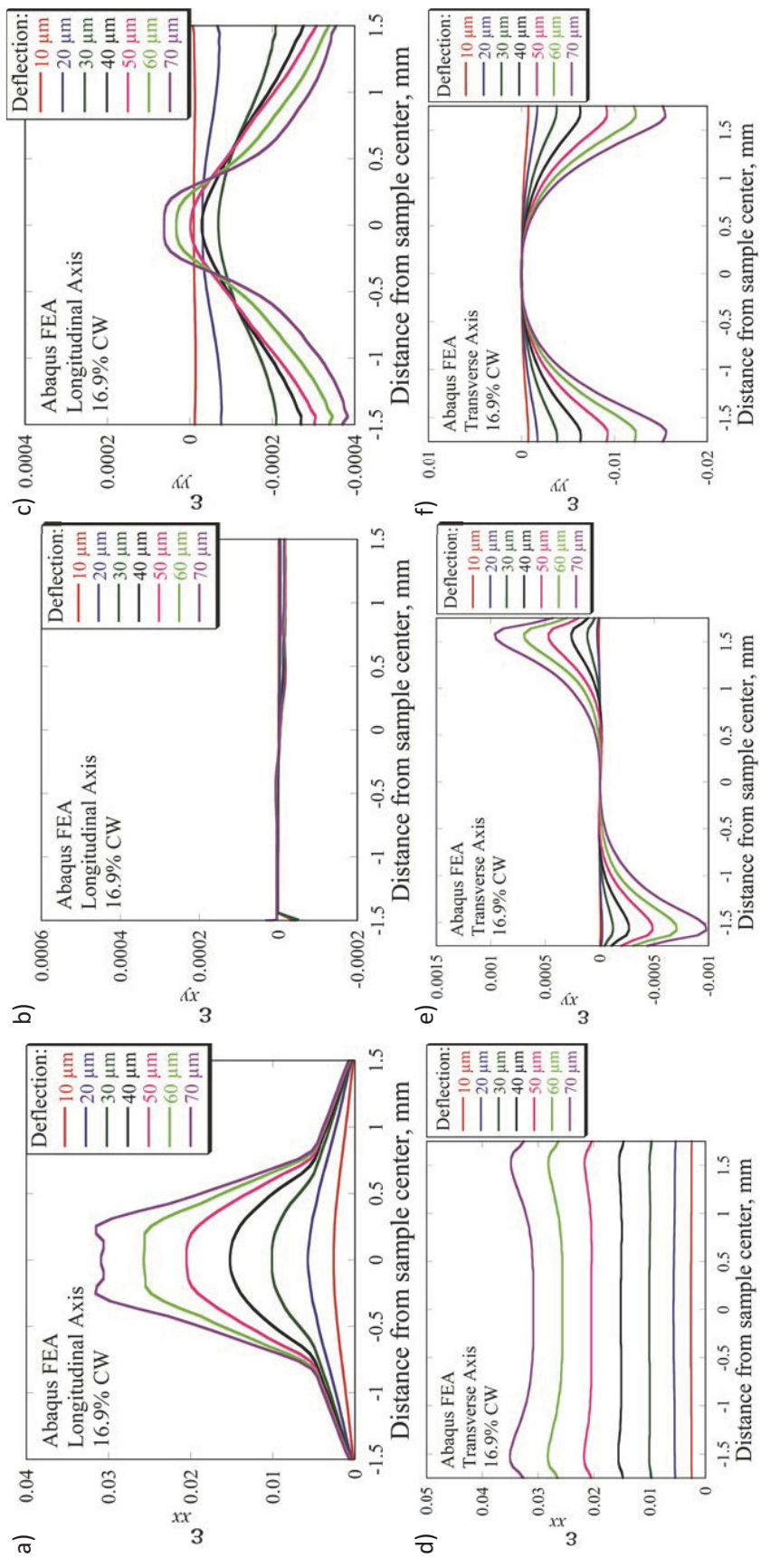


Figure 4-28. Quantitative strain profiles for the 16.9% CW condition at a variety of sample deflections. a) ϵ_{xx} , b) ϵ_{yy} , and c) ϵ_{yy} components of strain along the longitudinal axis, and d) ϵ_{xx} , e) ϵ_{xy} , and f) ϵ_{xy} components of strain along the transverse axis of the bend sample.

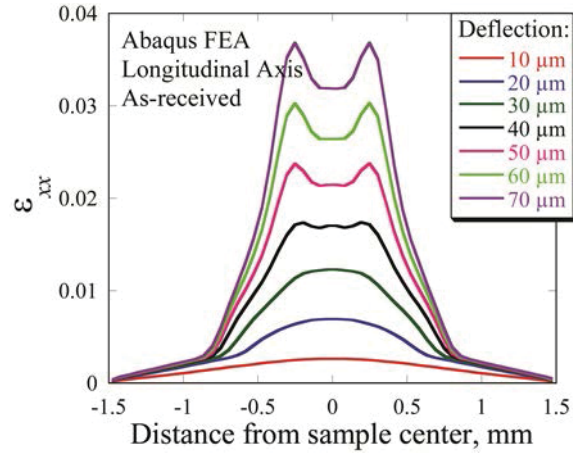


Figure 4-29. Quantitative strain profiles (xx component) at various sample deflections for the AR condition.

Experimental error determination

Several modifications were made to the model to determine the effect of possible errors that could occur during experimentation. Specifically, the effect of off-centering the sample laterally (Figure 4-10), off-centering the load points laterally (Figure 4-11), or varying sample thickness along the longitudinal direction (Figure 4-12). Comparing these effects at similar amounts of bend deflection in the simulation showed that none would significantly impact the strain profile during bend testing.

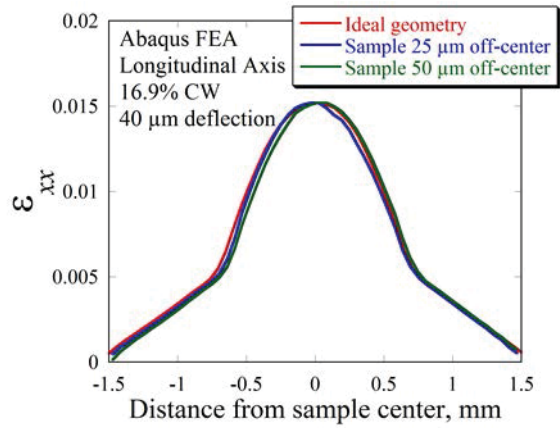


Figure 4-10. The effect of sample lateral off-centering on the xx component strain profile for the 16.9% CW condition at 40 μm deflection.

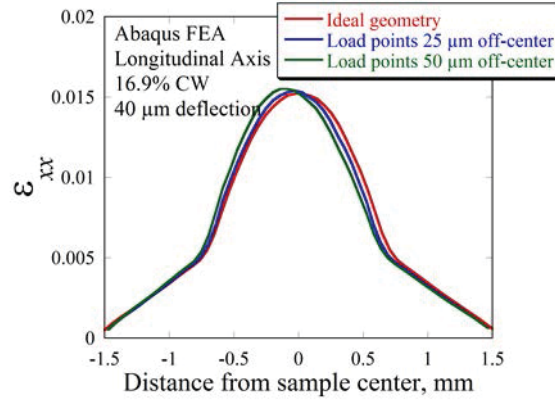


Figure 4-11. The effect of load point lateral off-centering on the xx component strain profile for the 16.9% CW condition at 40 μm deflection.

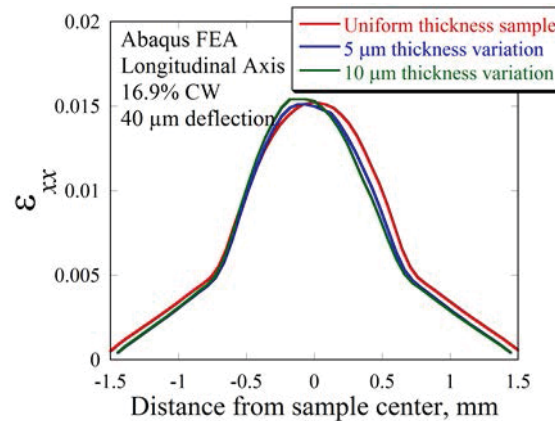


Figure 4-12. The effect of sample thickness variation on the xx component strain profile for the 16.9% CW condition at 40 μm deflection.

4.5.2 Four-point bend tests of unirradiated samples

To confirm accurate results in the FEA model, a series of bend tests were performed on unirradiated, cold-worked materials with a yield strength range similar to the neutron irradiated samples planned for testing. A summary of the unirradiated bend tests performed and their results is shown in Table 4-15..

Table 4-15. Unirradiated four point bend test matrix.

Environment	Sample Condition and ID	Increment No.	Sample Deflection, μm	Deflection Measurement Technique	Sample Center Strain	Strain Standard Deviation	Strain Measurement Technique
RT Air	AR #1	1	21.3	Direct	0.004	0.002	DIC
RT Air	AR #1	2	29.2	Direct	0.007	0.003	DIC
RT Air	AR #1	3	39.0	Direct	0.014	0.004	DIC
RT Air	AR #1	4	49.6	Direct	0.020	0.006	DIC
RT Air	16.9% CW #1	1	30.0	Direct	0.005	0.003	DIC
RT Air	16.9% CW #1	2	40.1	Direct	0.008	0.003	DIC
RT Air	16.9% CW #1	3	49.9	Direct	0.019	0.004	DIC
RT Air	16.9% CW #1	4	59.7	Direct	0.027	0.005	DIC
RT Air	16.9% CW #2	1	38.4	Direct	0.008	0.002	DIC
288 °C Ar	16.9% CW #3	1	39.8	Correction Factor	0.008	0.002	DIC, Indent Spacing
288 °C NWC	16.9% CW #4	1	42.2	Correction Factor	0.013	0.004	Indent Spacing

Bend tests were first performed in air on samples at both levels of CW, utilizing direct sample deflection measurement as well as DIC for surface strain measurement. One sample of each condition was tested, stopping the experiment after pre-determined increments to record the surface strain. Increments were stopped at sample deflections of 20, 30, 40 and 50 μm on an AR condition sample, and at deflections of 30, 40, 50, and 60 μm on a 16.9% CW condition sample. Load vs. deflection curves for each material condition after the incremented tests are shown in Figure 4-13.

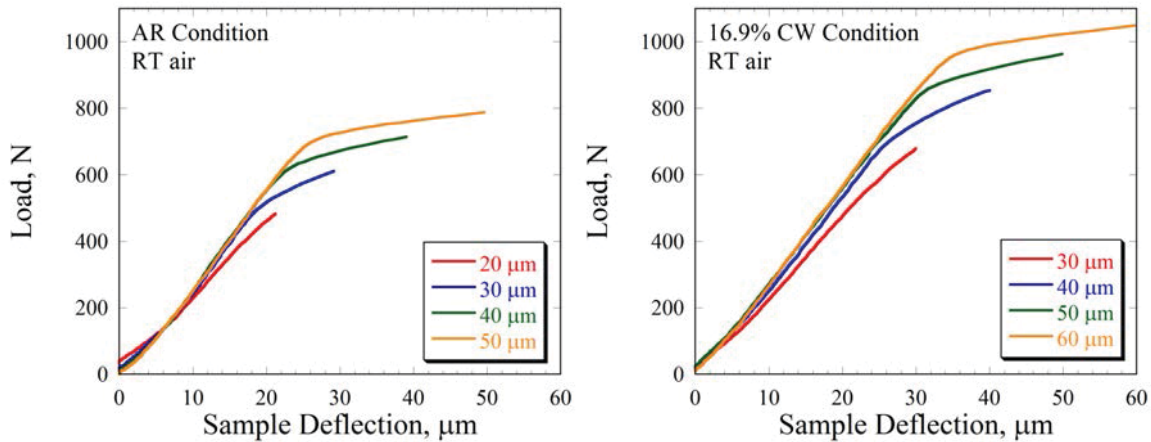


Figure 4-13. Load vs. sample deflection curves for incremented bend tests performed in RT air on a sample of the AR condition (left) and 16.9% CW condition (right).

Surface strain in the center of the bend sample after the incremented bend tests is compared with FEA model results in Figure 4-14. The small amount of disagreement is likely due to elastic spring-back between increments, which was not accounted for in the measurements.

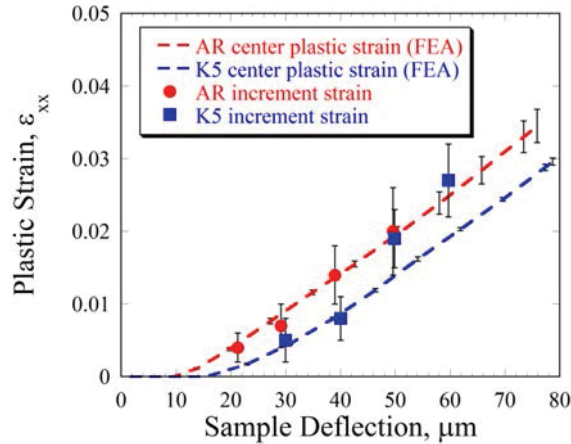


Figure 4-14. Average strain at the bend sample center after incremented experiments and FEA on each unirradiated material condition.

Three additional bend tests were performed to a nominal sample deflection of 40 μm on samples in the unirradiated 16.9% CW condition. These three bend tests were performed in RT air, 288°C Ar, and 288°C NWC environments to confirm similar bend test behavior in the three environments, and compare strain behavior with the results from the incremented bend tests. Load vs. deflection curves for these experiments are shown in Figure 4-15. Due to the high temperature in Ar and NWC, sample deflection was not directly measured and the aforementioned compliance correction technique was used. Use of this technique caused increased noise in the deflection measurement, because small changes in load (due to noise in the load cell reading) cause relatively large changes in the correction factor. All average strain measurements from the 16.9% CW condition are compared in Figure 4-16, showing that results in the high temperature environments agree well with those measured at room temperature, and incrementing strain in the bend test also has a negligible effect.

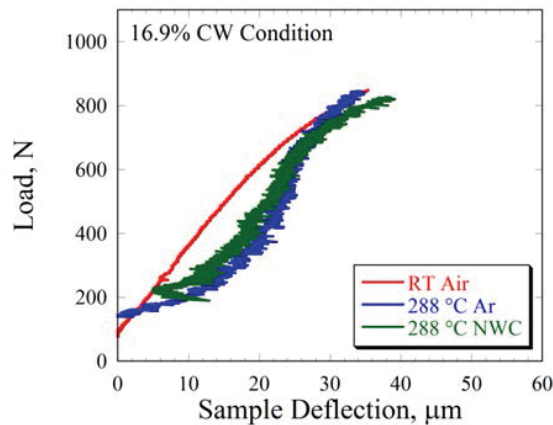


Figure 4-15. Load vs. sample deflection curves for bend tests on a 16.9% CW sample performed in RT air, 288 °C Ar, and 288 °C NWC.

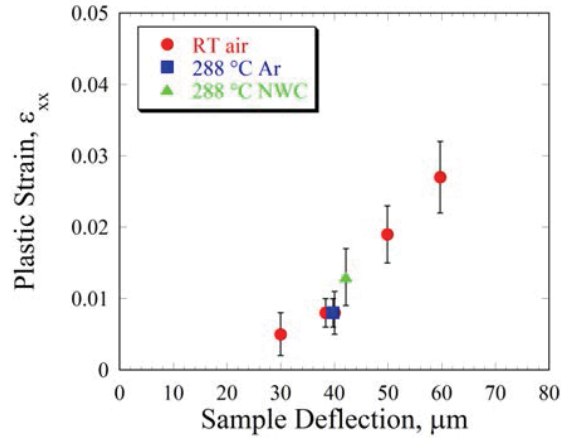


Figure 4-16. Average strain at the bend sample center for 16.9% CW samples in the three environments described in Fig. 13.

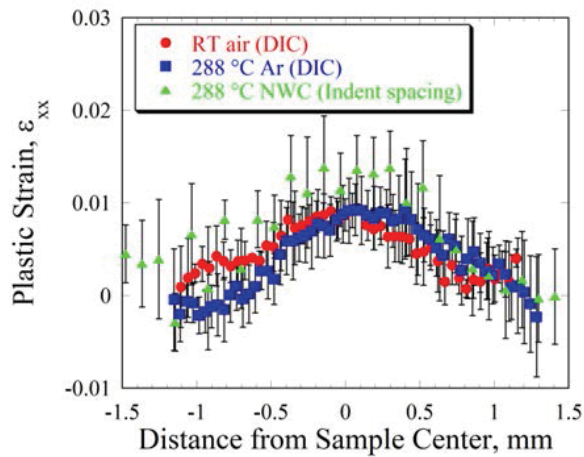


Figure 4-17. Plastic strain (ϵ_{xx}) in the longitudinal axis of 16.9% CW samples after 40 μm sample deflection in each test environment. Strain in NWC was measured via indent spacing.

4.5.3 Four-point bend tests of irradiated samples

The first neutron irradiated bend sample, alloy AS (CP 304L) irradiated to 10.2 dpa, was strained to failure in a single increment. This procedure determined general susceptibility to cracking, and established a guideline for subsequent bend tests, which were performed in small strain increments with the intent of stopping deformation at a point when a crack had become visible, but with a limited amount of propagation. An example of the load vs. LVDT deflection curves determined from an incremented bend experiment is shown in Figure 4-18, and a summary of the neutron irradiated bend test results are presented in Table 4-16. Fractions of the yield strength in bending were calculated based on the correlation observed between tensile yield strength and load required to cause yield in the bend test, Figure 4-19. When the yield strength was exceeded, plastic strain in the longitudinal direction (ϵ_{xx}) is used to track the deformation. Tensile yield strength values for

irradiated alloys were obtained from [24]. The final increment of each sample presented in Table 4-16 indicates when a crack initiation site was first observed, except in the case of alloy KS which was not expected to form an intergranular crack based on the purely ductile failure mode observed after CERT testing [11].

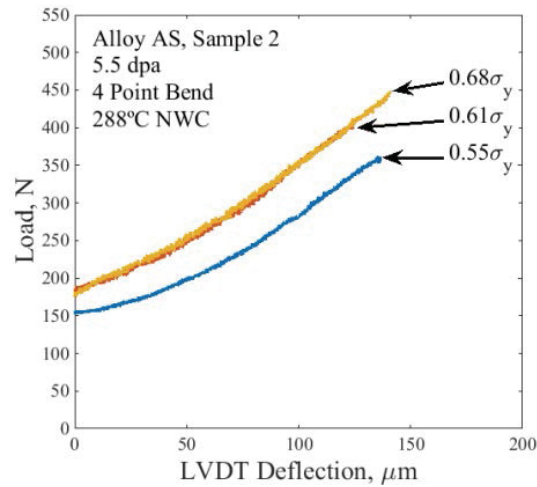


Figure 4-18. Load vs LVDT deflection curves for alloy AS at 5.5 dpa (#2), which was strained in three increments.

Table 4-16. Irradiated four point bend test matrix. N/A indicates that value was not available.

Alloy, dpa	ID No.		Increment											
			1	2	3	4	5	6	7	8	9	10		
AS, 10.2	1	σ_y	1.12											
		ϵ_{xx}	N/A											
AS, 5.5	1	σ_y	0.63	0.82										
		ϵ_{xx}	–	–										
AS, 10.2	2	σ_y	0.45	0.51	0.58	0.61								
		ϵ_{xx}	–	–	–	–								
AS, 5.5	2	σ_y	0.54	0.61	0.68									
		ϵ_{xx}	–	–	–									
ES, 10.2	1	σ_y	0.68	0.76	0.83	0.89	0.97	1.16	1.28	1.11				
		ϵ_{xx}	–	–	–	–	–	0.0033	0.0029	N/A				
KS, 9.6	2	σ_y	0.73	0.98	1.21	1.41	1.42	1.46	1.46	1.56	1.57	1.69		
		ϵ_{xx}	–	–	0.0013	0.0054	0.0050	0.0046	0.0000	0.0059	0.0050	0.0059		
AS, 47.5	1	σ_y	0.38	0.43										
		ϵ_{xx}	–	–										

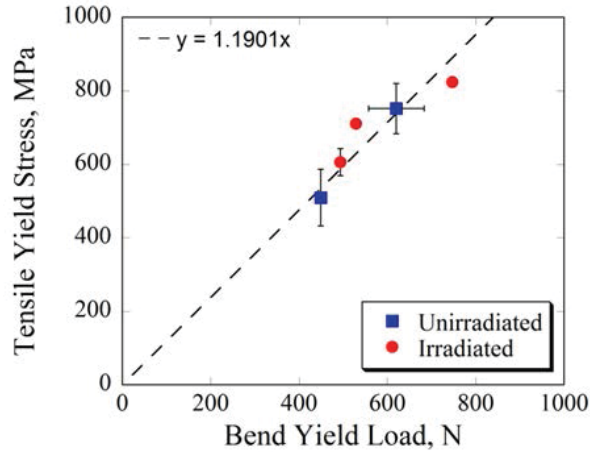


Figure 4-19. Comparison between tensile yield strength (determined from CERT experiments in 288°C NWC) and load required to cause yield in the four point bend test (in RT air for unirradiated samples and 288°C NWC for irradiated samples).

The CP 304L alloy was highly susceptible to IGSCC; crack initiation occurred prior to macroscopic yield at all three irradiation doses. By performing the bend tests in increments, several observations were made, which resulted in a better understanding of the crack initiation process. The first observation was of deformation (dislocation channels) at applied stress as low as 38% of the anticipated yield stress in bending. Examples of this deformation, Figure 4-20 and Figure 4-, show that despite the highly brittle state of alloy AS and the limited sites of localized deformation observed outside of IGSCC initiation sites, deformation occurs prior to IGSCC initiation in alloy AS. Deformation was not observed prior to crack initiation in the 10.2 dpa alloy, however it is assumed to occur due to the observations on the 5.5 and 47.5 dpa samples. Localized deformation was also typically observed at IGSCC initiation points, as observed in Figure 4- as well as Figure 4-, which show crack initiation sites after oxide removal. Despite the observation of localized deformation adjacent to IGSCC initiation sites, it is still unknown whether the deformation is a necessary precursor to crack formation.

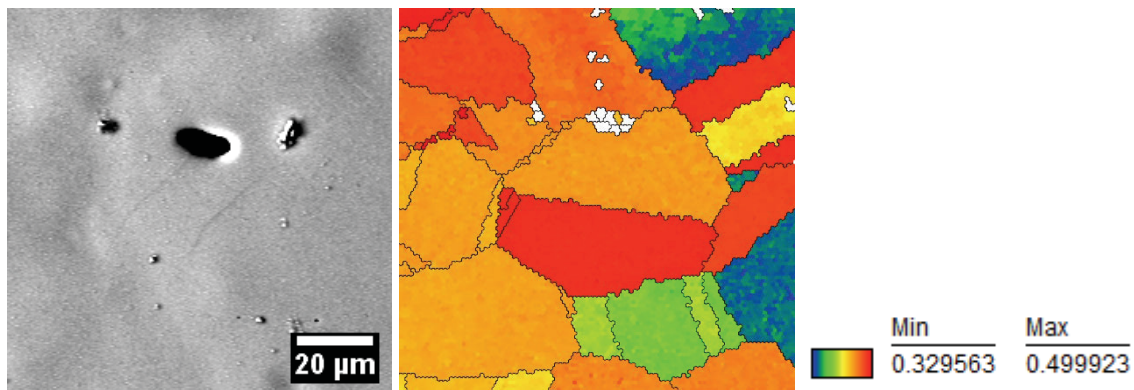


Figure 4-20. Deformation observed on the surface of the 47.5 dpa alloy AS sample after loading to $0.38\sigma_{ys}$ (left), and corresponding EBSD pattern showing schmid factor of grains in this region (right).

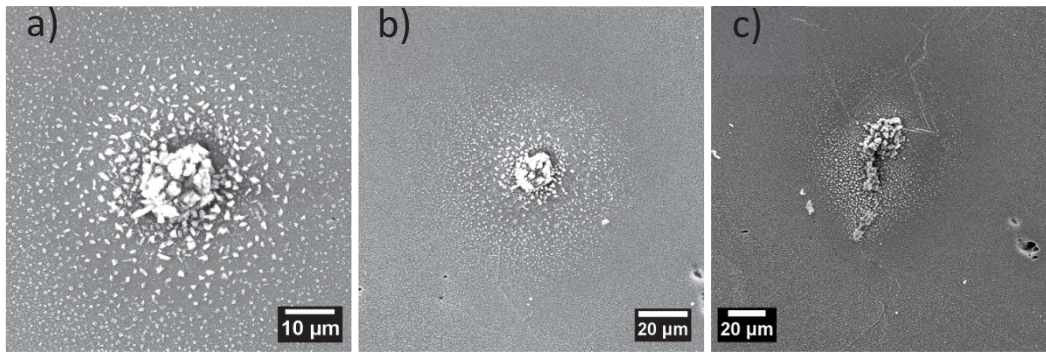


Figure 4-41. The location of the IGSCC crack on the 5.5 dpa alloy AS bend sample (#2) after a) $0.55\sigma_y$, b) $0.61\sigma_y$, c) and $0.68\sigma_y$.

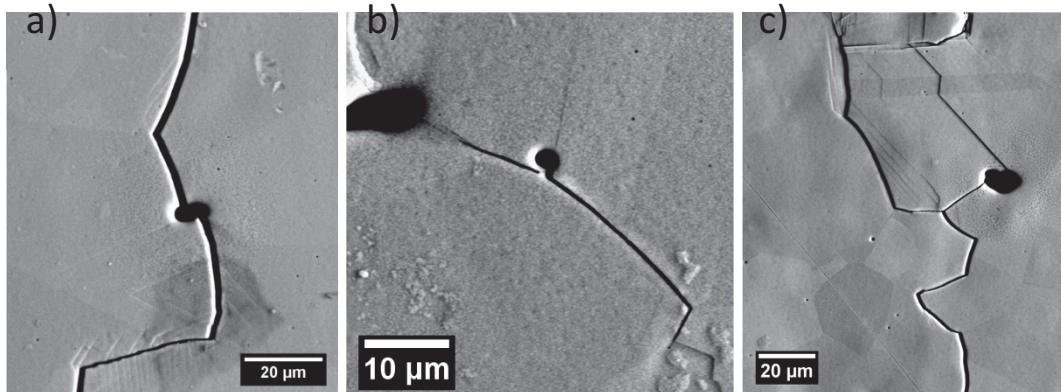


Figure 4-42. IGSCC initiation points in alloy AS at a) 5.5, b) 10.2, and c) 47.5 dpa after oxide removal.

Also seen in Figure 4-42 are surface inclusions at IGSCC initiation sites. Stress concentration at these large surface inclusions was a likely a factor causing the high susceptibility of alloy AS, and its difference in susceptibility compared to alloy ES and KS, which still had inclusions but were much smaller than in alloy AS, Figure 4-43. IGSCC initiation sites were typically observed at inclusion sites within the largest 10% of all inclusions observed on the sample surface.

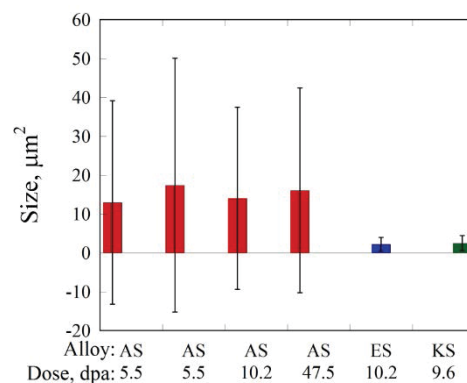


Figure 4-21. Average inclusion size on irradiated sample surfaces, recorded in the center 1 mm x 3.5 mm area of bend sample surfaces. Error bars represent standard deviations of the mean.

A final commonality observed at alloy AS IGSCC initiation points was large corrosion product formation. As observed in Figure 4-, corrosion product formation occurred at inclusions prior to the formation of a crack, but the corrosion product increased in size with the opening of an IG crack. Corrosion was not typically observed at inclusion sites, only occurring at <1% of surface inclusions on alloy AS bend sample surfaces. It is still unknown whether this corrosion product forms without the application of stress, or whether a crack in the inclusion is necessary for the formation of a crevice to induce corrosion product formation. Further experiments are necessary to separate the effects of corrosion product formation and localized deformation in the IGSCC initiation process of alloy AS samples.

HP alloys ES and KS displayed very different behavior than alloy AS, as expected from previous CERT test results. Significant amounts of localized deformation were observed prior to both the point of macroscopic sample yielding, as well as IGSCC initiation in the HP 304L alloy. As expected from the observation of ductile failure in the CERT test, the HP 304L+Ni alloy did not form an IGSCC crack after straining to $\sim 3.3\%$ plastic strain. Two cracks initiated in the HP 304L alloy at the sample edge, Figure 4-22 and Figure 4-23. Due to interaction with the sample edge, it is difficult to identify the exact cause of crack formation, and what caused the difference in cracking susceptibility between the two HP alloys. However, comparing localized deformation at similar levels of strain on the two alloy surfaces showed that the density of slip transmission sites in alloy KS were higher than in alloy ES, Figure 4-24. Slip transmission can refer to locations where a dislocation channel transmits across a grain boundary, cross slips onto a separate slip system in the same grain, or causes grain boundary sliding. All events result in a sharp direction change in the dislocation channel, and are assumed to impart less stress to a grain boundary than interactions where the dislocation channel forms a discontinuous interaction with the grain boundary [16]. Since less stress is able to accumulate at channel – grain boundary intersections in alloy KS (due to the higher probability of slip transmission), the stress required for IG crack initiation is likely unable to be achieved.

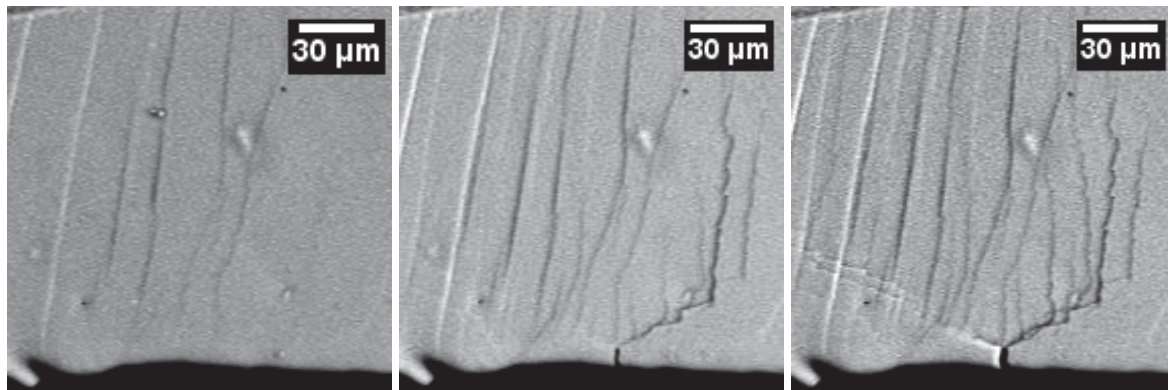


Figure 4-22. The region where the small IGSCC crack formed at the edge of the 10.2 dpa alloy ES sample a) prior to initiation after straining to $0.97\sigma_y$, b) after crack initiation at $\epsilon_{xx} = 0.3\%$, and c) after straining to $\epsilon_{xx} = 0.6\%$.

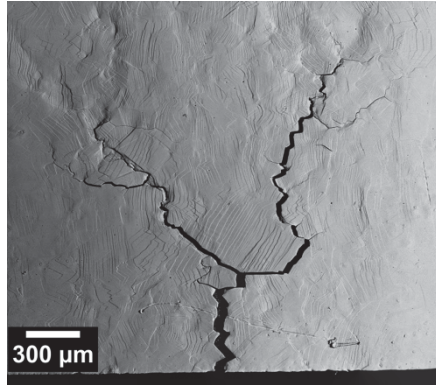


Figure 4-23. The large IGSCC crack on the 10.2 dpa alloy ES sample after the final straining increment.

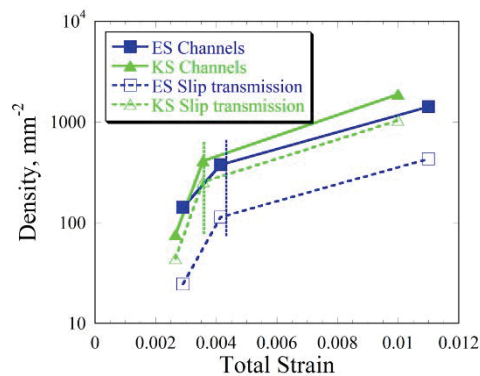


Figure 4-24. Comparison of localized deformation on HP alloys ES and KS after a range of applied strain. Vertical solid lines represent the point at which macroscopic yield was observed.

4.5.4 Comparison to Literature

The strain to crack initiation from the four-point bend tests is compared with strain to failure determined from previous CERT tests in Figure 4-25. A linear relationship is observed between the two measures of strain obtained from the experiments, indicating that the CERT test clearly gives a good indication of crack initiation susceptibility, and the strain to failure determined from this test is representative of the strain to crack initiation.

The stress at crack initiation determined from four-point bend tests can also be compared with the results of constant load experiments on neutron irradiated stainless steels. Constant load test databases have been accumulated by Chopra [25] and Bosch [26], both indicating that a stress threshold to initiate IASCC in a primary water test environment exists at ~40% of the yield strength in high dose materials, greater than approximately 20 dpa. Results of the four point bend tests have been superimposed over these databases in Figure 4-26, with a red dashed line plotted to indicate the lower threshold for cracking observed in the bend test. Excellent agreement was observed between the two test types, as crack initiation occurred in the bend test between 38% and 43% of the yield strength in the 47.5 dpa specimen. The consistency in this stress threshold between the two test types, despite differences in test environment and material condition (many constant load

results were from CW materials), highlights the importance of stress in the IASCC initiation mechanism.

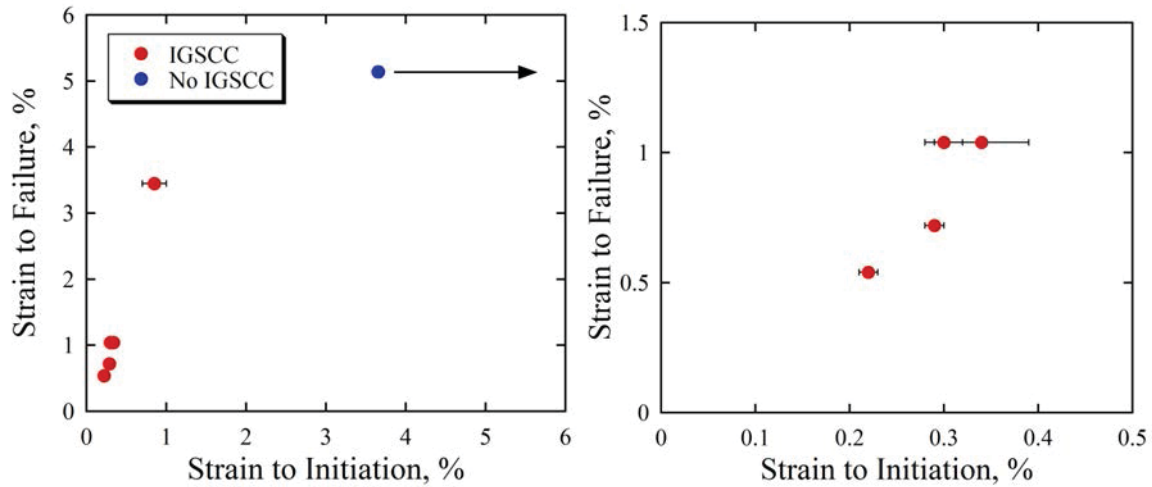


Figure 4-25. Comparison between strain to failure (by CERT testing, from [11]) and strain to initiation determined (from four point bend tests) on neutron irradiated samples tested in 288 °C NWC at a strain rate of $3.5 \times 10^{-7} s^{-1}$. The right figure is a magnified view of the small strains in the left figure.

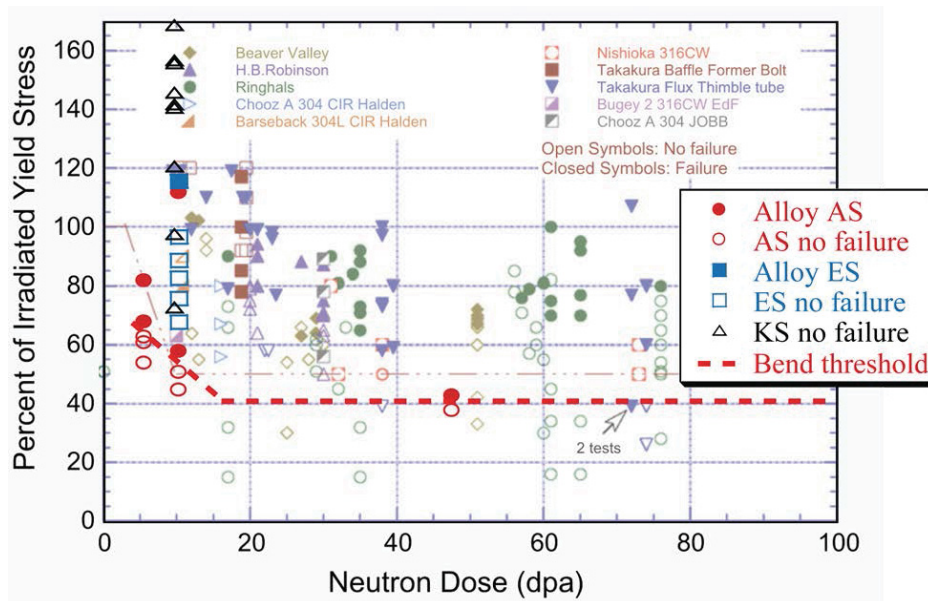


Figure 4-26. Stress required to initiate IASCC as a function of dose. Four point bend test data from the current study in 288°C NWC has been superimposed on the constant load database available from Chopra in primary water conditions [25].

4.6 CGR test results

4.6.1 304L Irradiated in Barseback 1 BWR

RCT-2: As-irradiated

Precracking

Figure 4-49 shows the a/W and corrosion potential of RCT-2 during precracking stage. The precracking steps were started when the corrosion potential of sample stabilized. The crack didn't initiate after about 400 h at a nominal K_{\max} of $18 \text{ MPa}\cdot\text{m}^{1/2}$. In order to accelerate the precracking, K_{\max} was increased to $20 \text{ MPa}\cdot\text{m}^{1/2}$ and R was gradually decreased to 0.2. Afterwards, crack initiated and CGR stabilized at $3.0 \times 10^{-6} \text{ mm/s}$. Then R was increased to 0.6 in steps and K_{\max} was gradually decreased to $18 \text{ MPa}\cdot\text{m}^{1/2}$, which caused CGR to decrease. Thereafter, the waveform frequency was gradually decreased to 0.001. CGR further decreased during these steps. The waveform was switched to trapezoid and then maintained at constant K of $18 \text{ MPa}\cdot\text{m}^{1/2}$. A anticipatory correction factor (CF) of 2.5 was applied. The CGRs on the graph have been corrected after the fracture surface was analyzed.

Changing environment

To study the effect of hydrogen environment, the environment was switched from NWC to HWC as the CGR stabilized at $1.8 \times 10^{-6} \text{ mm/s}$. Nominal K was kept at $18 \text{ MPa}\cdot\text{m}^{1/2}$. As shown in Figure 4-50, the CGR dropped significantly to $2.6 \times 10^{-7} \text{ mm/s}$ after the switch. Then the environment was switched back to NWC and the CGR increased to a value of $1.9 \times 10^{-6} \text{ mm/s}$, which is very consistent with the previous value in NWC. During this process, no action was taken to control the outlet conductivity which ranged between 0.055 and 0.085 $\mu\text{S/cm}$ when the environment stabilized.

Conductivity has significant effect on the CGR. In order to study the effect of conductivity on the CGR, dilute H_2SO_4 solution was injected into the main water column via a peristaltic pump. As shown in Figure 4-51, CGR increased from $1.9 \times 10^{-6} \text{ mm/s}$ to $3.7 \times 10^{-6} \text{ mm/s}$ when the outlet conductivity was increased from ~ 0.075 to $\sim 0.2 \mu\text{S/cm}$. As the conductivity was further decreased to $\sim 0.1 \mu\text{S/cm}$, CGR dropped to $3.2 \times 10^{-6} \text{ mm/s}$. The outlet conductivity was maintained at $\sim 0.1 \mu\text{S/cm}$ thereafter.

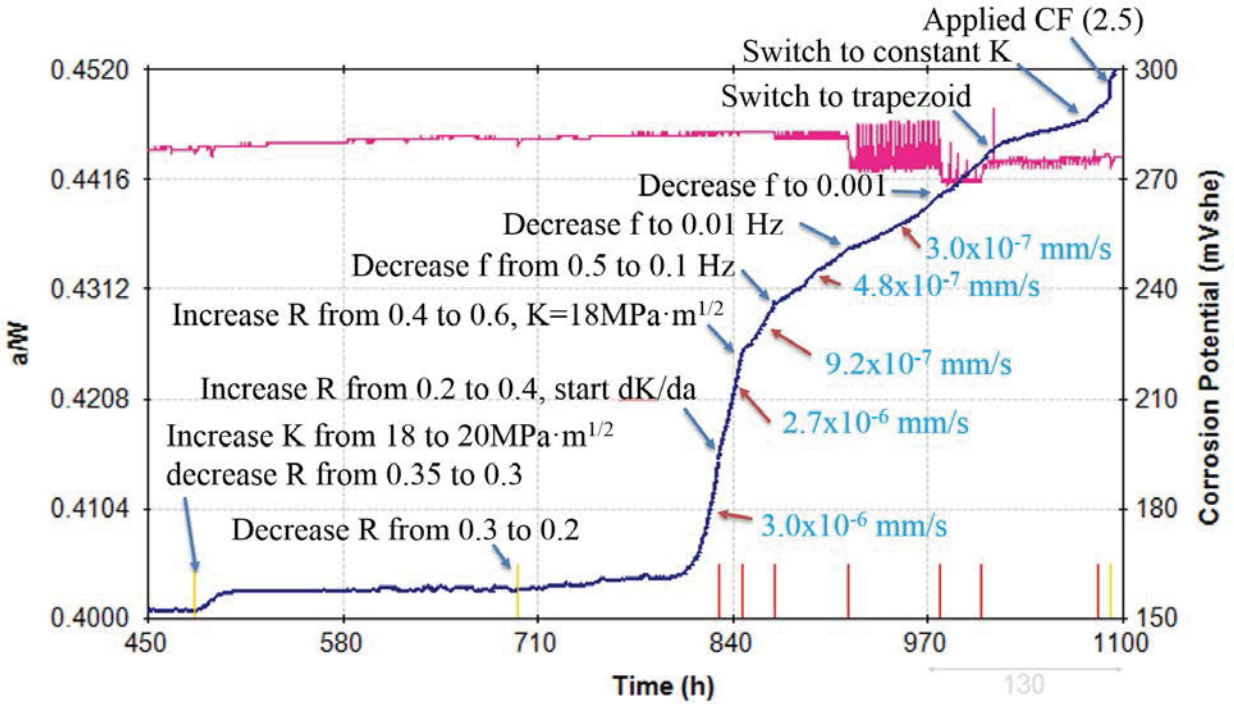


Figure 4-49: a/W and corrosion potential of RCT-2 in 288 °C water containing 2 ppm O_2 during precracking stage.

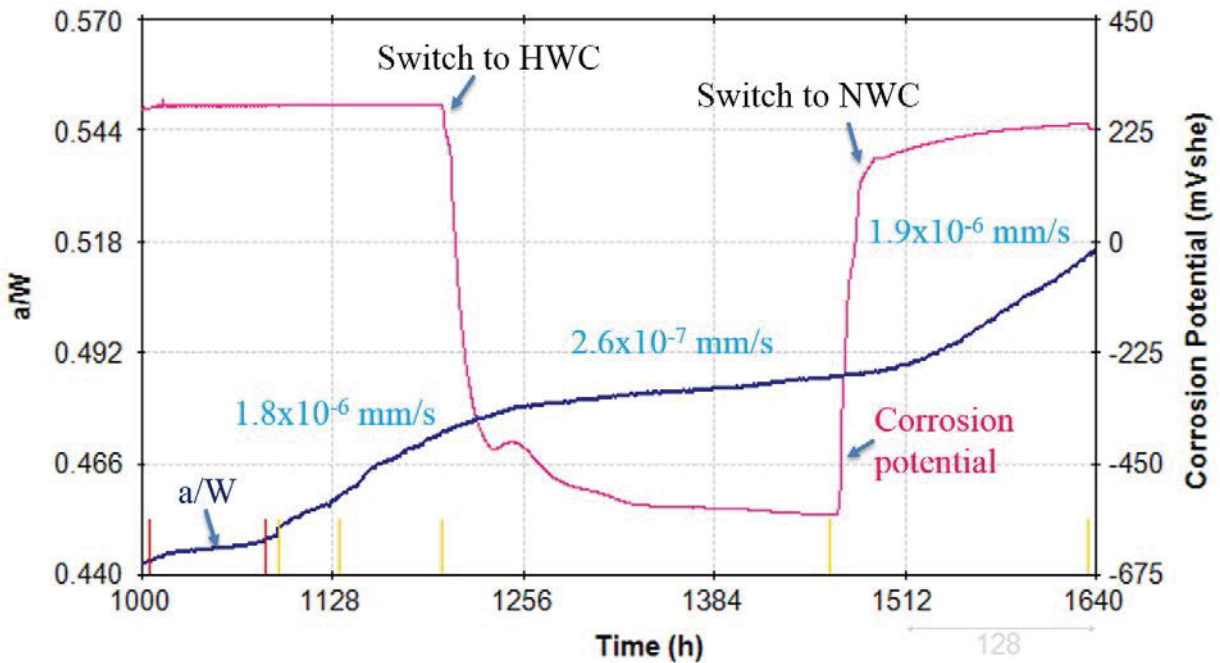


Figure 4-50: a/W and corrosion potential of RCT-2 in 288 °C water at a nominal K of 18 $MPa \cdot m^{1/2}$ during the change of corrosion potential.

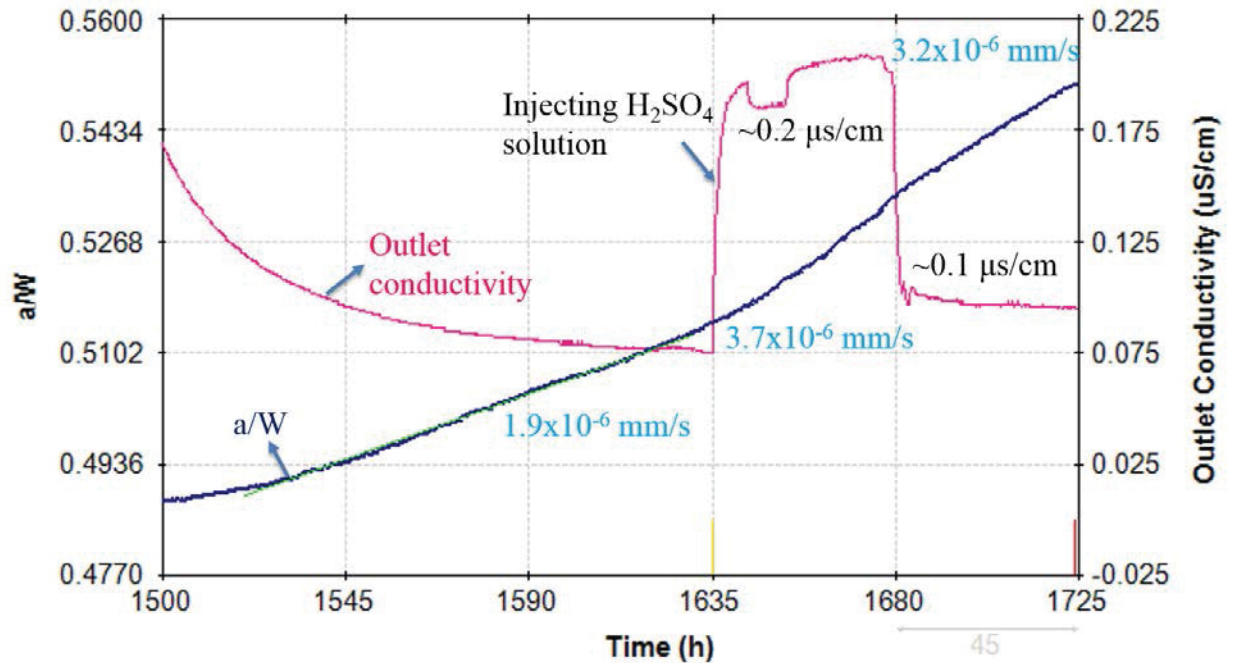


Figure 4-51: a/W and corrosion potential of RCT-2 in 288 °C water containing 2 ppm O_2 at a nominal K of 18 $MPa \cdot m^{1/2}$ during the change of conductivity.

Decreasing K

In order to study the CGR dependence on K , the nominal K value was first dropped from 18 to 14 $MPa \cdot m^{1/2}$ and then to 11 $MPa \cdot m^{1/2}$. As shown in Figure 4-52, the CGR dropped from 3.2×10^{-6} mm/s to 6.7×10^{-7} mm/s as K was decreased from 18 to 14 $MPa \cdot m^{1/2}$. Then K was further dropped to 11 $MPa \cdot m^{1/2}$ and CGR decreased to 9.5×10^{-8} mm/s.

The effect of corrosion potential was also studied at nominal K of 11 $MPa \cdot m^{1/2}$. As shown in Figure 4-53, CGR decreased from 9.5×10^{-8} mm/s to 2.3×10^{-8} mm/s after the switch from NWC to HWC.

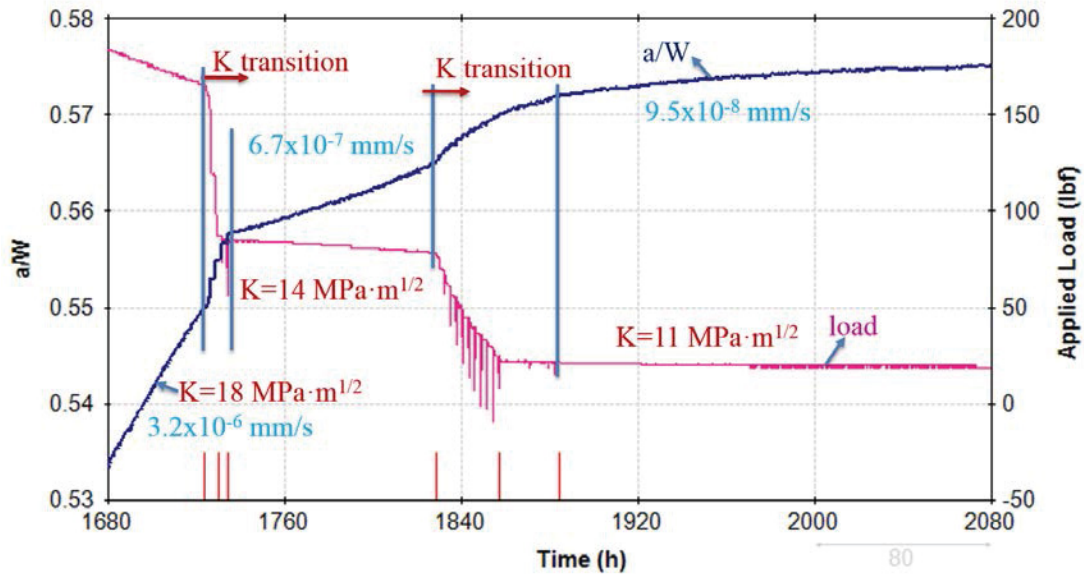


Figure 4-52: a/W and applied load of RCT-2 in 288 °C water containing 2 ppm O_2 during the change of corrosion potential.

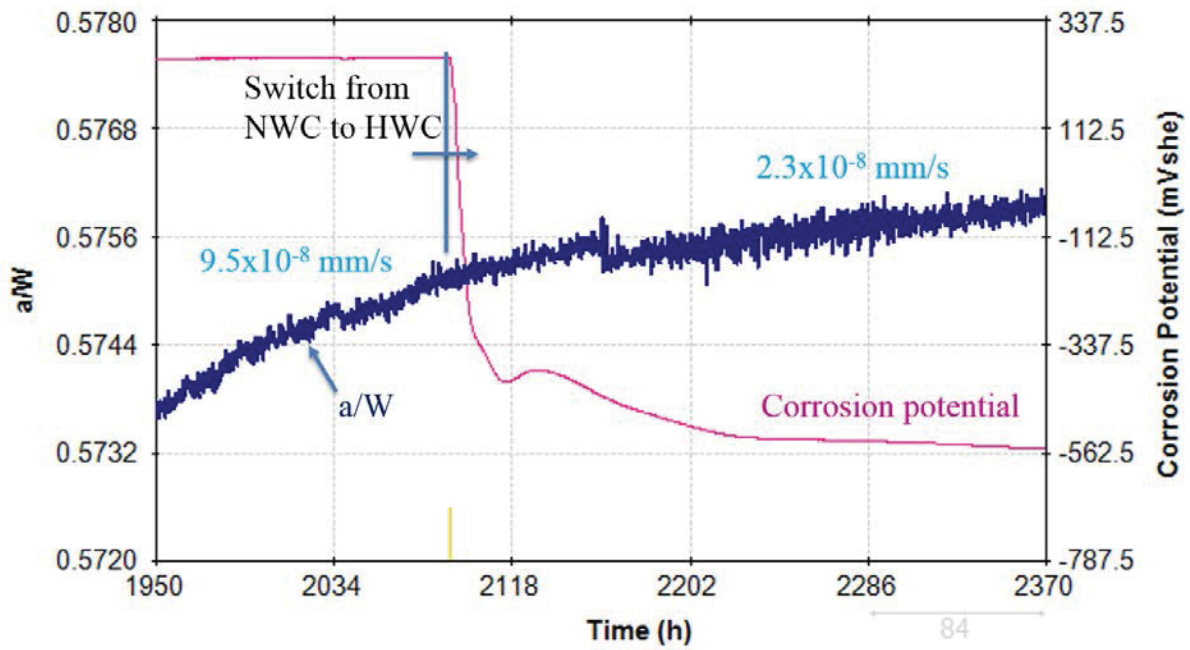


Figure 4-53: a/W and corrosion potential of RCT-2 in 288 °C water at a nominal K of 11 $MPa \cdot m^{1/2}$ during the change of corrosion potential.

Fracture surface analysis

The sample was opened and the fracture surface was analyzed with SEM. Figure 4-54 shows the fracture surface from the side views. The images show that the crack is in plane and follows the side grooves well.

Figure 4-55 shows the plane view image of fracture surface. The fracture surface is intergranular except the pre-cracking and the post-cracking region. The pre-cracking region is not even. The SCC region is mainly intergranular and no significant ligament was found. Nevertheless, DCPD still underestimated the real CGR even an anticipatory correction factor was applied.

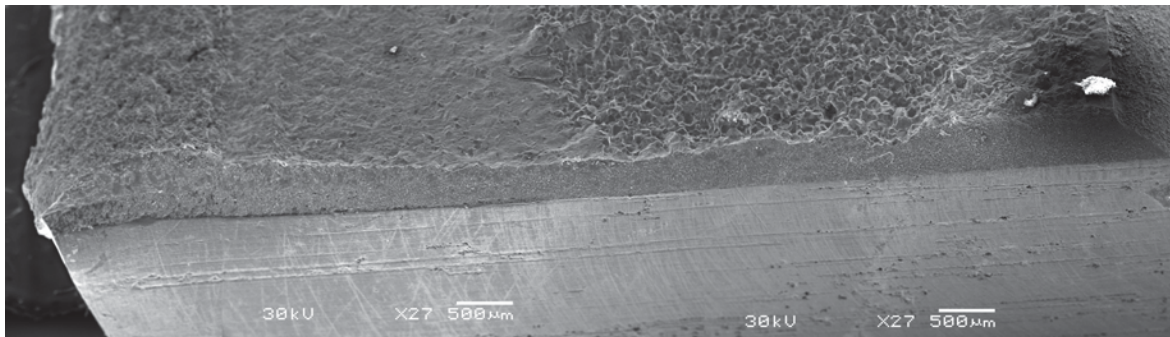


Figure 4-54: Side view image of fracture surface of RCT-2.

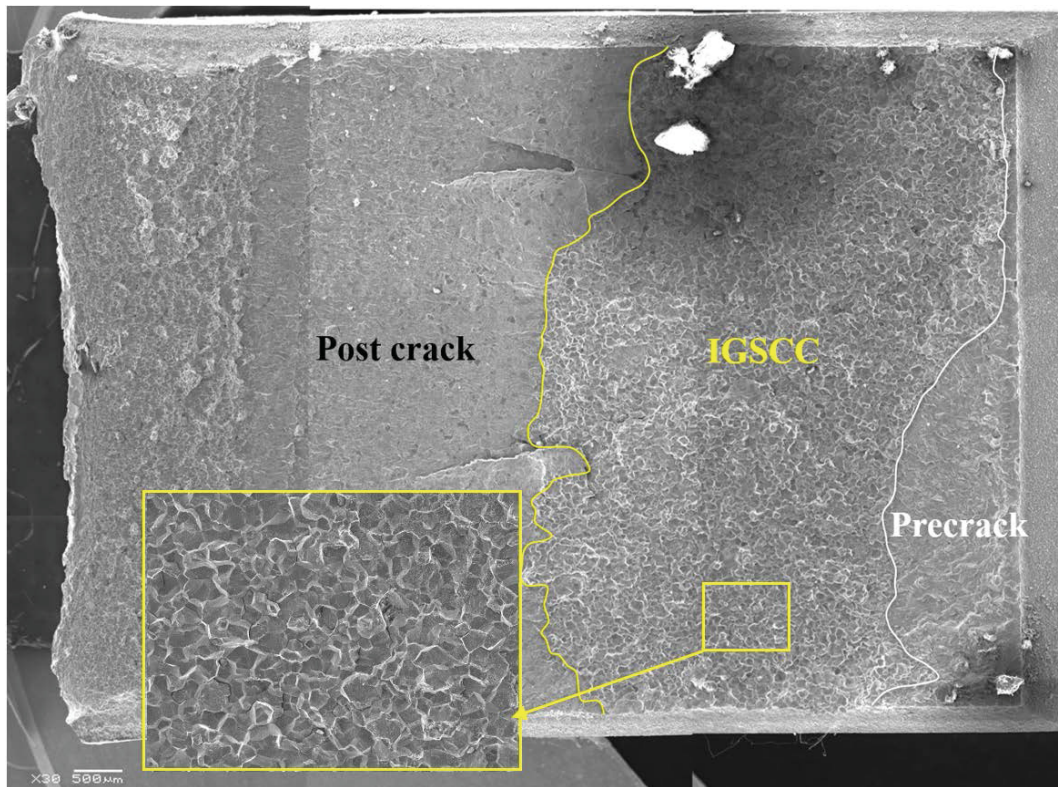


Figure 4-55: Fracture surface of RCT-2.

DCPD Estimation and Actual CGR

The crack lengths measured by DCPD and SEM are listed in Table 4-17. For the pre-crack region, the length measured by DCPD is close to the real length even this region is not even. DCPD underestimated the crack length for the IGSCC region. The CGRs measured by DCPD were adjusted with the post-cracking correction factors listed in Table 4-17. K values were also recalculated with the applied load and the corrected crack length. The K validity was checked using the corrected K values and the yield strength of this sample which is measured to be 685.5 MPa at 288 °C [28]. The results are listed in Table 4-18.

Table 4-17: Results of crack length measurements and correction factor.

	Pre-Crack	IGSCC
Length by DCPD (mm)	0.775	2.073
Length by SEM (mm)	0.737	3.83
Correction factor	0.95	1.85

Table 4-18: Corrected K values and CGRs of RCT-2 and RCT-3.

Sample	Environment	Corrected K (MPa·m ^{1/2})	Corrected CGR (mm/s)	K validity, %		
				E399	Δ YS/2	Δ YS/3
RCT-2 (as-irradiated)	NWC	19.9~19.1	1.8×10^{-6}	49	78	98
	HWC	19.5~19.8	2.6×10^{-7}	52	83	104
	NWC	23.3~25.0	3.2×10^{-6}	71	114	143
	NWC	20.1~20.9	6.7×10^{-7}	64	102	128
	NWC	17.0~17.3	9.5×10^{-8}	55	88	111
	HWC	17.3~17.4	2.3×10^{-8}	56	89	112
RCT-3 (PIA:550°C:20 hr)	NWC	18.9~19.5	5.9×10^{-7}	82	117	137
	HWC	19.5~19.6	1.5×10^{-8}	84	120	140
	NWC	19.7~31.3	6.6×10^{-7}	117	166	194

RCT-3: PIA 550°C:20 hr

Precracking

Figure 4-56 shows the a/W and corrosion potential of RCT-3 during precracking stage. The precracking steps were started when the corrosion potential of sample stabilized. Nominal K_{\max} was set to $18 \text{ MPa}\cdot\text{m}^{1/2}$. The CGR decreased as R was increased from 0.4 to 0.6. It decreased further as the frequency was decreased. The CGRs on the graph have been corrected after the fracture surface was analyzed.

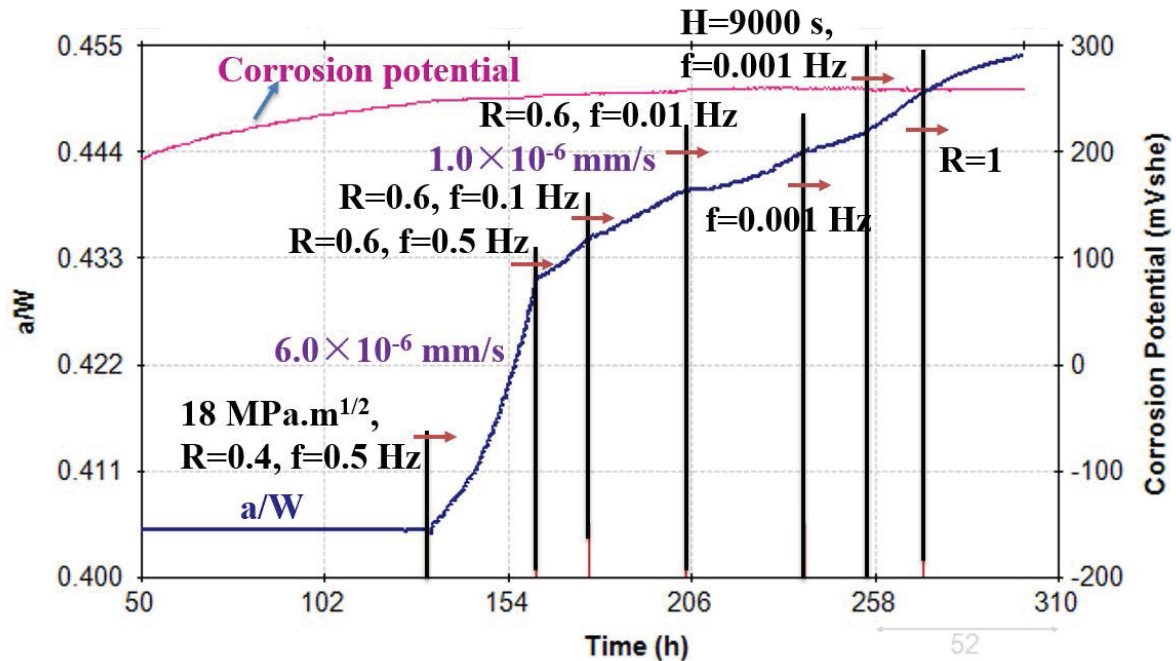


Figure 4-56: a/W and corrosion potential of RCT-3 in 288 °C water containing 2 ppm O_2 during precracking stage.

Changing environment

To study the effect of hydrogen environment, the environment was switched from NWC to HWC as the CGR stabilized at $5.9 \times 10^{-7} \text{ mm/s}$. Nominal K was kept at $18 \text{ MPa}\cdot\text{m}^{1/2}$. As shown in Figure 4-57, the CGR dropped significantly to $1.5 \times 10^{-8} \text{ mm/s}$ after the switch. Then the environment was switched back to NWC, requiring $\sim 650 \text{ hr}$ before the CGR stabilized at a value of $6.6 \times 10^{-7} \text{ mm/s}$, which was very consistent with the previous value in NWC. There are some jumps in a/W (up to 0.002) which may be due to the break of ligaments on the crack propagation path. Such jumps have also been reported in the results from the sample of the same heat [1].

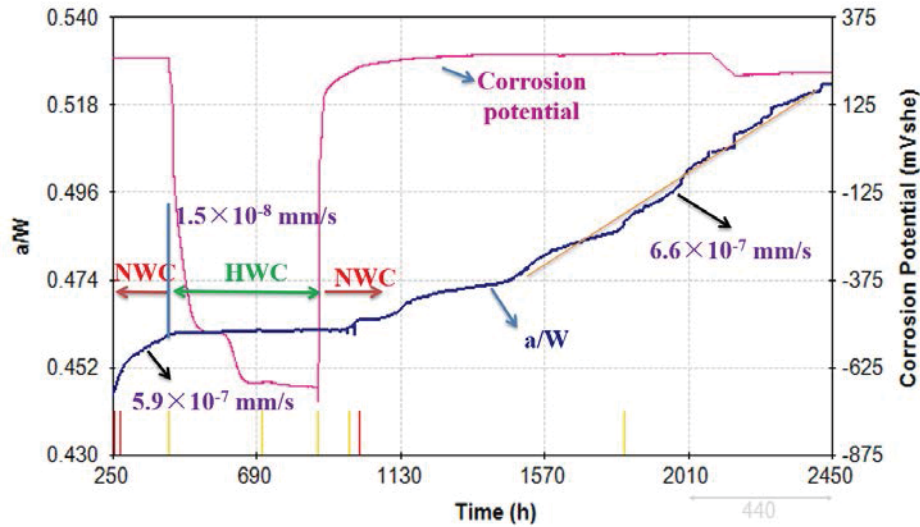


Figure 4-57: a/W and corrosion potential of RCT-3 in 288 °C water at a nominal K of 18 $\text{MPa}\cdot\text{m}^{1/2}$ during the change of corrosion potential.

Decreasing K

In order to study the CGR dependence on K , the nominal K value was dropped from 18 to 14 $\text{MPa}\cdot\text{m}^{1/2}$. As shown in Figure 4-58, there is a large jump in a/W (~ 0.033) after the K was held at 14 $\text{MPa}\cdot\text{m}^{1/2}$. It is very unusual to see such a large jump. This jump was confirmed to be due to the fracture of the ligament on the fracture surface which is shown in Figure 4-60. The CGR at 14 $\text{MPa}\cdot\text{m}^{1/2}$ finally stabilized at 2.9×10^{-7} mm/s. Then K was further dropped to 11 $\text{MPa}\cdot\text{m}^{1/2}$. Unexpectedly, two large jumps in a/W (over 0.14) occurred. The final a/W was around 0.92 when the test was stopped.

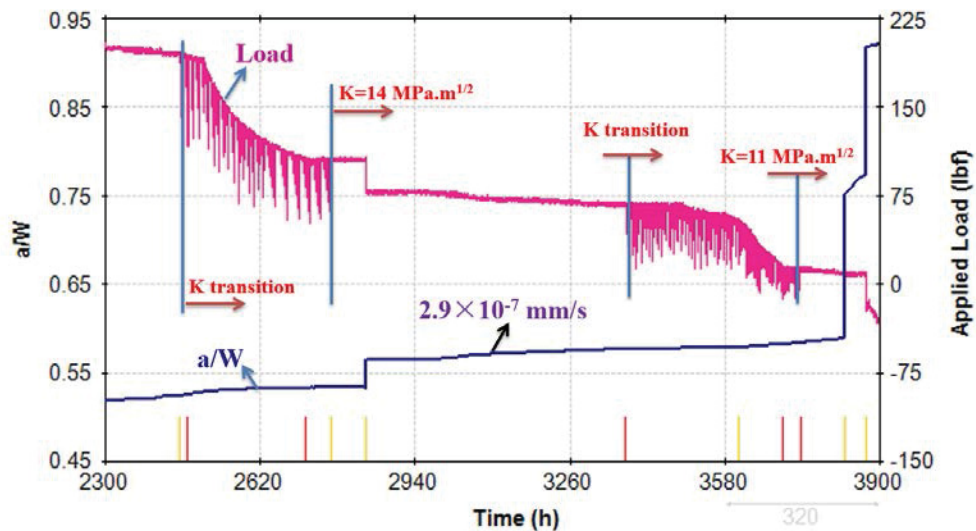


Figure 4-58: a/W and applied load of RCT-3 in 288 °C water containing 2 ppm O_2 during K transition from 18 to 11 $\text{MPa}\cdot\text{m}^{1/2}$.

Fracture surface analysis

The sample was opened and the fracture surface was analyzed with SEM. Figure 4-59 shows the fracture surface from the side views. The images show that the crack is in plane and follows the side grooves well.

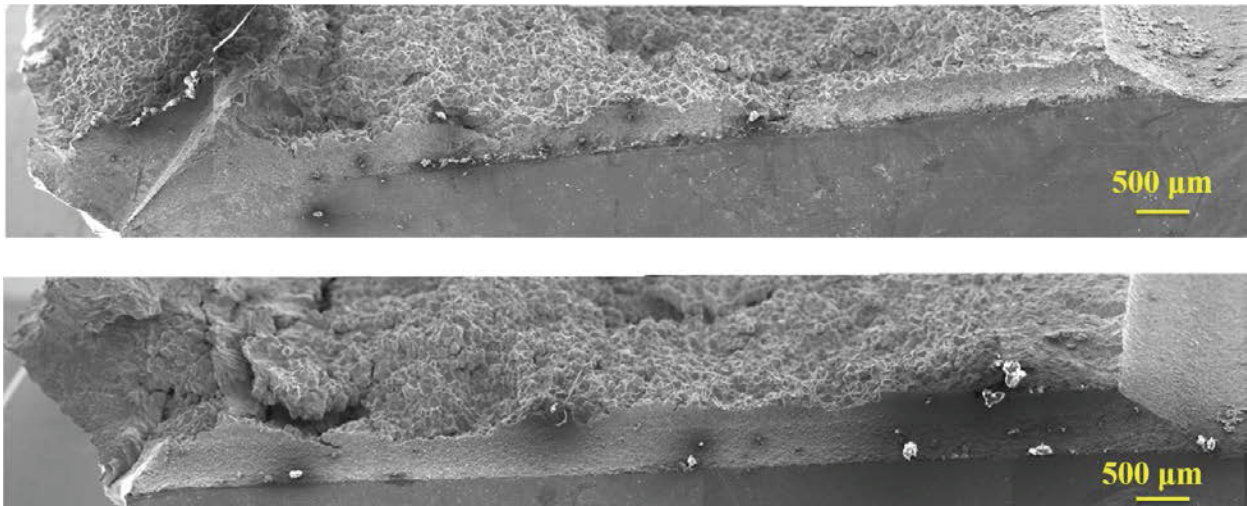


Figure 4-59: Side view images of fracture surface of RCT-3.

Figure 4-60 shows the plane view image of fracture surface. The fracture surface is intergranular excepting the pre-cracking and the post-test fracture region, which are due to cyclic loading and mechanical overloading at room temperature respectively. The large crack on the left side was due to the post-test loading procedure. It should be noted that there is a ligament between the upper part and the lower part in the IGSCC region. The enlarged images from the starting and ending parts of the boundary show that the two parts of IGSCC region are not on the same plane and the interface between them is transgranular and almost perpendicular to the fracture surface. Such interface would not crack open until the opening of the main crack is larger than the distance between the two crack propagating planes. So, the main crack front should be beyond the crack front at the interface. This could explain why DCPD underestimated the crack length. The jumps in a/W were probably caused by the sudden opening of the interface.

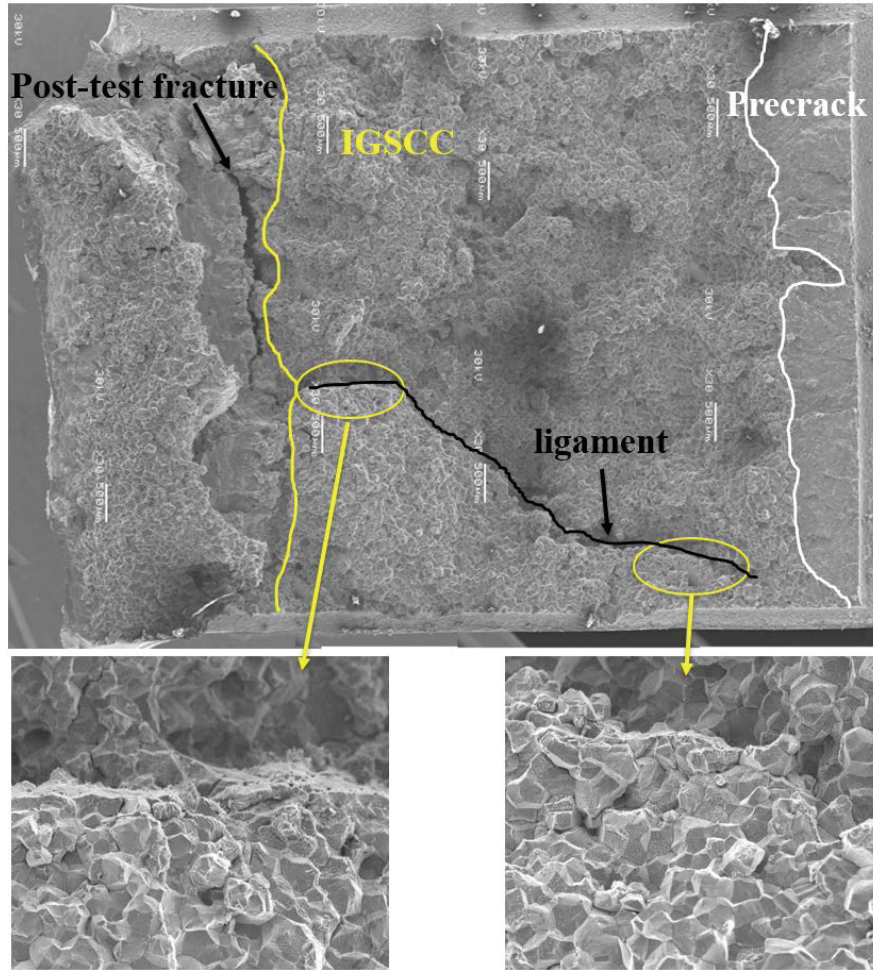


Figure 4-60: Fracture surface of RCT-3.

DCPD Estimation and Actual CGR

The crack lengths measured by DCPD and SEM are listed in Table 4-19. The post-cracking correction factor is the ratio between the real length (by SEM) and the length measured by DCPD. For the pre-crack region, the length measured by DCPD is close to the real length. However, DCPD underestimated the crack length significantly for the IGSCC region. Therefore, the CGRs measured by DCPD were adjusted with the post-cracking correction factors listed in Table 4-19. Meanwhile, the K values were also recalculated with the applied load and the corrected crack length. The K validity was checked using the corrected K values and the yield strength of this sample which is measured to be 421.3 MPa at 288 °C [27]. The results are listed in Table 4-18. It should be mentioned that this sample has been aggressively annealed and it shows strain hardening during tensile test [27]. So adjustment of the yield stress by $\Delta YS/3$ for K validity check would be over stringent.

Table 4-19: Results of crack length measurements and correction factor.

	Pre-Crack	IGSCC
Length by DCPD (mm)	0.725	2.157
Length by SEM (mm)	0.856	6.018
Correction factor	1.18	2.79

4.6.2 Neutron-irradiated CIR Alloys

Results of crack growth rate experiments on the CIR alloys was described in full in Ref. [29], so they will be summarized here for reference in the Discussion on effects of microstructure, environment and PIA. CGR data are generated from fracture mechanics based SCC experiments (i.e., linear elastic fracture mechanics is used for studying SCC and the crack tip K in the opening mode K_I) using compact tension (CT) specimens that meet dimensional requirements to maintain plain strain conditions in the vicinity of the crack tip. As shown in 4-61, the CGR data are typically plotted as a function of stress intensity factor K on a log-linear scale, as K is considered to be the mechanical driving force for crack growth. Note that only the CGR data under constant K conditions are used for their K -dependence. Historically, empirical CGR disposition curves based on experimental data are used as guidelines for industry. The typical disposition curves include EPRI curves for NWC BWR and HWC BWR, and the NRC disposition curve described in report NUREG-0313.

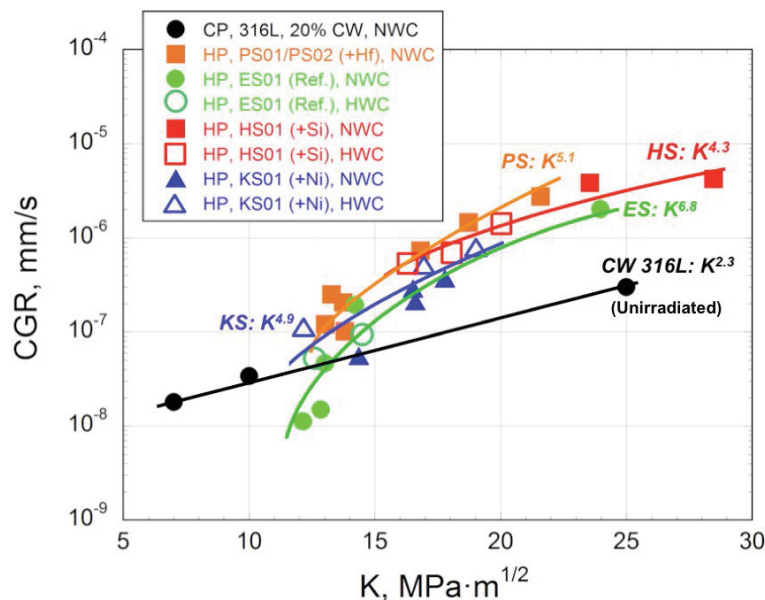


Figure 4-61. K -dependence of CGR for irradiated alloys PS, HS, KS, ES, and an unirradiated alloy 316L (20% CW).

The CGR data on the CIR alloys is summarized in Table 4-20. Each test was conducted in NWC at 288 and 320°C and in HWC at 288°C. The stress intensity, K , was also varied to obtain the K -dependence in both NWC and HWC as shown in Figure 4-61. Results are analyzed in the Discussion for the effect of environment on crack growth rate.

Table 4-20. DCPD estimation and actual crack growth length.

Sample ID	TG Pre-Cracking			IGSCC			Total Cracking		
	DCPD Measurement (mm)	Actual Length (mm)	Error (%)	DCPD Measurement (mm)	Actual Length (mm)	Error (%)	DCPD Measurement (mm)	Actual Length (mm)	Error (%)
PS02	0.252	0.220	14.5	3.778	4.620	-18.2	4.030	4.840	-16.7
IP01	0.290	0.330	-12.1	4.390	2.890	51.9	4.680	3.220	45.3
MS01	0.397	0.390	1.8	3.305	2.050	61.2	3.702	2.440	51.7
NS01	0.158	0.152	3.9	4.112	3.415	20.4	4.270	3.567	19.7
BS01	0.000	0.000	-	4.422	5.896	-25.0	4.422	5.896	-25.0
GS01	0.000	0.000	-	1.495	NM	-	1.495	NM	-

NM: Not Measured.

5 DISCUSSION

To better understand the crack initiation behavior in irradiated austenitic stainless steels, the discussion section of this report is divided into following three sections: role of environment, role of annealing treatment and role of dislocation channeling on cracking behavior. Role of environment and annealing treatment on crack propagation is also discussed.

5.1 Role of Environment on IASCC

Since multiple tensile bars for same heat were available at similar damage levels, the role of environment on cracking behavior in alloys irradiated in BOR-60 reactor was studied using CERT testing method.

The primary difference between the three environment conditions is the electrochemical corrosion potential (ECP) of the material which was measured to be 215 mV, -570 mV and -860 mV (vs SHE) for NWC, HWC and PWR environments respectively. The results have previously been published as part of detailed report on the crack initiation alloys for these materials [reference].

5.1.1 Crack Initiation

The effect of environment on crack initiation was studied mainly through CERT tests. While CERT tests pulled to failure include both crack initiation and crack growth, they spend most of their time in the initiation stage as the growth stage in this type of test for irradiated samples is generally very short. Figure 5-1a shows the effect of environment conditions of the cracking behavior of the neutron irradiated CIR alloys in CERT tests in NWC, HWC and PW.

Reduction in the ECP of the material resulted in a reduction in the cracking susceptibility of the material with the material being least susceptible to cracking in primary water (PW) environment. Alloy BR which is the cold worked type 316 stainless steel alloy showed low cracking susceptibility in all the environment. Other than this one exception, all the alloys showed increase in cracking susceptibility at higher ECP levels. This increase in the % IG indicates that the cracking susceptibility of the material (crack initiation + growth) is highly dependent on the environment it is exposed to. Increase in the aggressiveness of the environment (higher ECP) results in a higher cracking susceptibility. Another aspect to look at is the role of composition in cracking behavior. Although alloy AS and SW are both commercial purity alloys with similar damage levels (AS 5.5 dpa, SW 4.4 dpa) the cracking behavior is different for both the alloys. Alloy AS has higher susceptible to cracking compared to the alloy SW. This can be attributed to the presence of MnS inclusions in alloys AS which accelerates the crack initiation in this material as observed from the

four-point bend test studies on this material. Alloy SW on the other hand has a lower Mn content and negligible S content which suppresses the formation of MnS inclusion in this material.

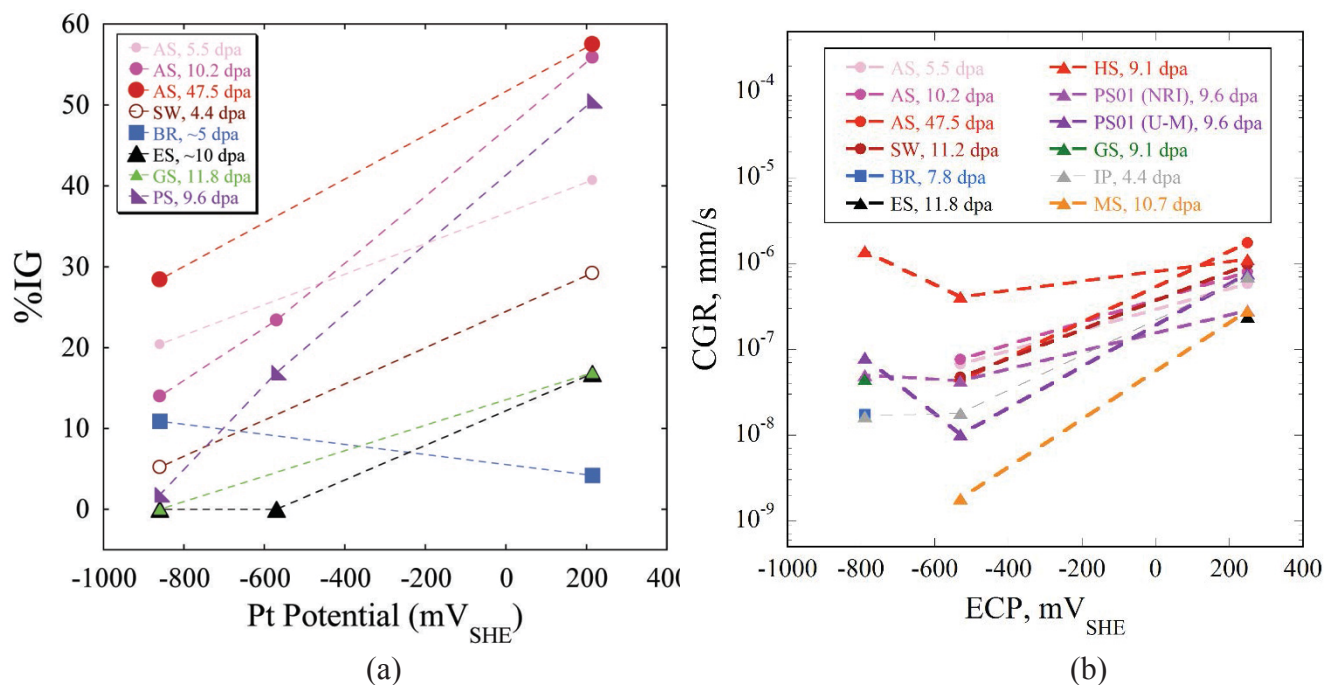


Figure 5-1. Comparison of a) %IG in CERT tests [24]]and b) CGR versus ECP for alloys tested in multiple environmental conditions.

From CERT tests, we can correlate the cracking behavior based on the %IG content observed on the fracture surface. However, this parameter includes the contribution from both crack initiation and crack growth. To isolate these two different phenomena, crack morphology is a useful tool to provide insight. Fig 5-2 shows an example of crack morphology for the 10.2 dpa AS alloy in these three environmental conditions. There is a vast difference in the crack morphology between the alloying conditions. Both NWC and HWC showed limited number of crack nucleation sites, with the crack nucleation being limited to one nucleation site while the primary water conditions showed a large number of crack nucleation sites irrespective of the material condition (dose, cold-work or solute addition). The lower % IG in the PWR environment can be attributed to a lower crack propagation rate in this environment. Even though a large number of cracks are being nucleated on the specimen surface, the environment allows for limited crack propagation and thus the overall susceptibility of the material to IASCC in this environment is low. On the other hand, the crack initiation is limited to one nucleation site in NWC and HWC environments. Subsequently, the crack propagates at a fast rate and thus resulting in IASCC failure of the material in these two environments. Compare to NWC environment, the lower ECP in HWC environment would result in a lower crack propagation rate. Thus, the material shows a lower fraction of intergranular failure in this environment compared to NWC environment.

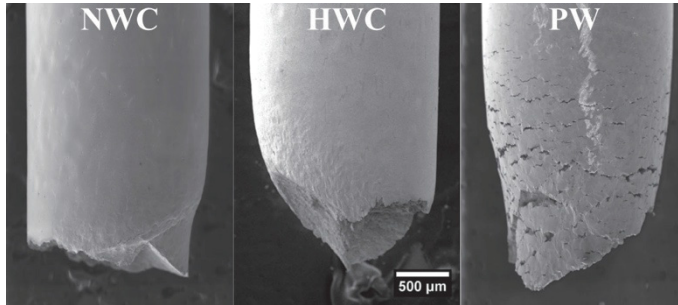


Figure 5-2. Secondary cracking on the gage surface of the 10.2 dpa alloy AS tensile bars tested in NWC, HWC, and PW conditions.

5.1.2 Crack Growth Rate

Solute addition CIR alloys

CGRs were higher in NWC than in HWC, with the exception of alloy HS, which had very high CGRs in all three environments. The variation in CGR between HWC and PW was less pronounced. CGR increased from 3×10^{-8} mm/s to 4×10^{-7} mm/s in alloy PS (U-M data) when changing water chemistry from HWC to PW, while no change in CGR was observed for sample IP01 over the same ECP range. For alloy PS, it has been previously reported that the addition of hydrogen to PW caused an increase in CGR at 288°C. It is unclear what role hydrogen plays in promoting crack growth. However, inclusions were observed on the dimples of post-cracking fracture surface, which were likely hafnium carbide based on their size and distribution [30]. It is possible that the carbides acted as traps for hydrogen and the CGR was accelerated because of the hydrogen-assisted change in mechanical properties around the crack tip. Compared to the CGRs in NWC, larger variations in CGR were observed among alloys in HWC and PW environments. This can be attributed to the difficulty of crack growth at low potentials, as cyclic loading was frequently used when the crack stopped growing. The retardation and the reactivation of crack growth affected the accuracy of the reported CGR.

304L from Barseback 1 BWR

It has been shown that [28] decreasing the corrosion potential by switching to HWC could suppress the CGR of non-irradiated stainless steel significantly (up to two orders of magnitude). The results on neutron-irradiated stainless steel from Jessen also shows that there is a significant mitigating effect HWC environment on CGR when the dose is below 7 dpa [1]. One of the samples in ref. [1] was made from the same bulk material as the samples used in this work. The results from that sample can be directly used for comparison. The results of that sample along with the results from this work are plotted in Figure 5-3. From Figure 5-1, HWC environment still has significant effect in mitigating CGR on all the samples, irrespective of the post annealing treatment. The CGRs of RCT-2 (as-irradiated) and RCT-3 (annealed at 550 °C for 20 h) were decreased by factors of 7 and 39 respectively in HWC. The mitigating effect on RCT-3 is larger than that on RCT-2, indicating that the HWC effect may be dependent on the inherent SCC susceptibility of material. Consistently,

the results from ref. [1] also show that there is little mitigating effect of HWC on samples with doses above 7 dpa. So it seems that HWC has smaller mitigating effect on material with higher SCC susceptibility. It is to be confirmed by testing samples with other annealing treatments and higher doses in the further.

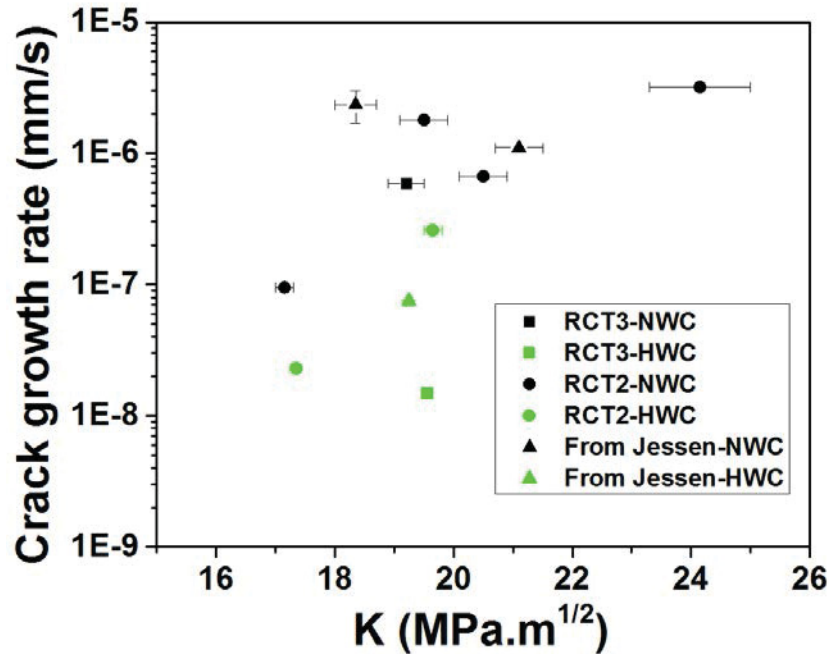


Figure 5-3. CGRs of 304 L SS irradiated to 5.9 dpa in 288 °C water.

5.2 Post irradiation Annealing

5.2.1 Crack Initiation

Post-irradiation annealing (PIA) treatments have been reported to result in improvement in the cracking behavior of austenitic stainless steels in both proton and neutron irradiated conditions [31-42]. However, due to a multitude of variables involved and difference between alloy compositions between different studies, a clear understanding of the effect of role of post irradiation annealing is still not there. All of these studies focus on the role of PIA on different properties (mechanical and microstructural) and its correlation with the cracking behavior observed in the environment using constant extension rate testing in simulated reactor environment. With regard to the cracking susceptibility of the material in different annealing conditions, most of the cracking data reported in literature has been based on the % intergranular fracture observed on the fracture surface. As such, for the sake of comparing to previous PIA results, this value will be utilized for comparing the relative removal of IASCC susceptibility. Following CERT straining to failure, the fracture and gage surfaces were fully examined by SEM; where the separate fracture regions were quantified to determine %IG, %TG, % mixed IG/TG, and % ductile failure.

The as-irradiated tensile specimen was observed to have the highest degree of IASCC susceptibility, with 48.4% of the final fracture area by a pure intergranular fracture, 28.6% of the

region showed a mixed intergranular-transgranular fracture, while only 3.6% showed a pure transgranular failure and the remaining 19.4% was the final ductile failure of the specimen. As seen in Figure 4-17, this susceptibility of 48.4% IG failure is moderate as compared to other PIA studies in the literature. Only two cases reported in the literature had a lower %IG fracture of the as-irradiated material: a 316L steel at 12 dpa [32] and a 304 at ~4 dpa [33]. In general, most other studied alloys had both a higher degree of irradiation damage and higher %IG fracture in the as-irradiated condition [32, 40,43]

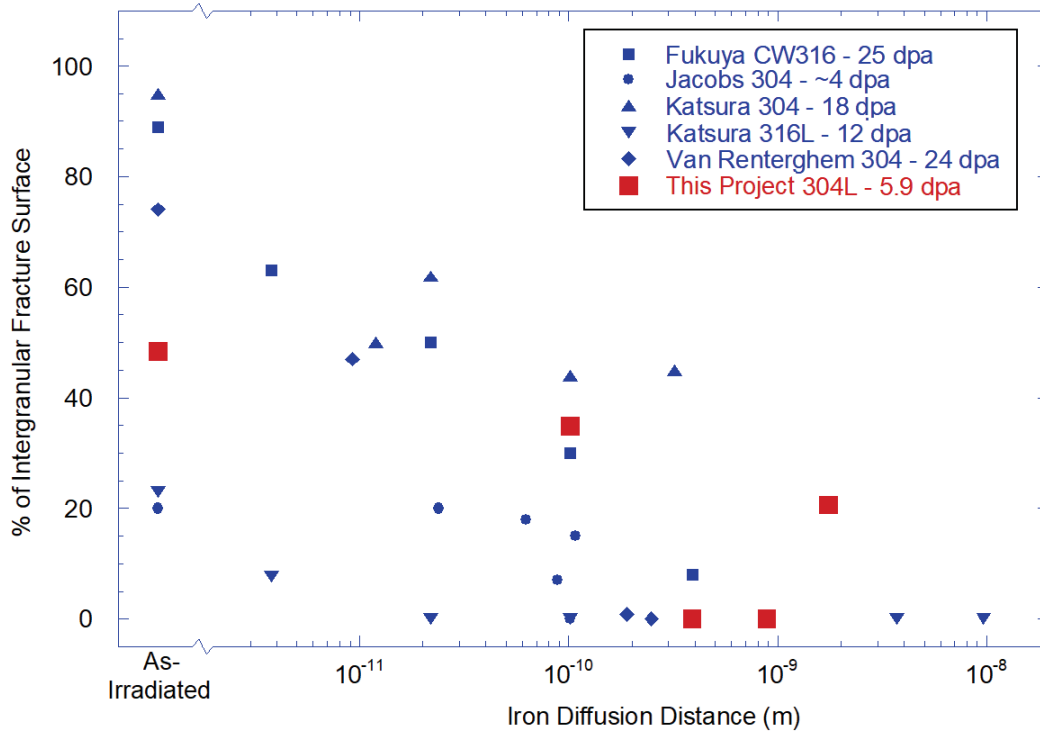


Figure 5-4: Comparison of the measured intergranular fracture area as a percentage of the final fracture area from this project and several neutron-irradiated PIA specimens in the literature.

Annealing at 500°C: 1hr and 550°C:20 hr resulted in a lower percentage of pure intergranular fracture of 34.9% and 20.6% respectively. The degree of mixed mode fracture seemed to vary between the studied conditions while the degree of transgranular fracture was significantly enhanced after annealing to ~30% of the fracture area for both examined conditions. It is worth noting that characterizing IASCC susceptibility by %IG produces a higher susceptibility for the 550°C: 20 hr condition than expected. This tensile specimen initiated multiple cracks, presumably around the same strain level, and two cracks began to grow from opposite sides of the tensile specimen slightly off plane from one another. After both cracks progressed through roughly 50% of the cross section, the specimen failed as a shear between the two separate crack planes. As such the final fracture, cross section displays two independent crack fronts, thus a higher percentage of pure intergranular fracture. Annealing at both 550°C: 1 and 5hr caused a full mitigation of IASCC susceptibility and displayed a 100% ductile fracture.

Figure 5-4, shows the change in % of intergranular failure with varying PIA treatments for both this material and several similar alloys in literature. By comparison, many of the other alloys studied in literature display a faster removal of IASCC susceptibility than the 304L alloy studied

in this project when examining all four PIA conditions, though the exact reason for this behavior is not known. A possible exception to this is a 304 alloy at 18 dpa studied by Katsura *et al.* [31]; this material displayed a much higher as-irradiated susceptibility (95% IG), but was seen to stabilize at ~45% intergranular fracture after annealing 500°C: 10 hr. However, due to the lack of additional annealing treatments, the behavior of the 304 alloy after annealing at higher temperatures is unknown. However, if one excludes the 550°C: 20 hr annealing condition, it is clear that the mitigation of IASCC susceptibility follows a very similar trend to that in literature.

5.2.2 Crack Growth Rate

Figure 5-3 shows that the CGRs of the as-irradiated sample (RCT-2) are comparable with those from ref. [1] and higher than the CGRs of the sample annealed at 550 °C for 20 h (RCT-3). The mitigation factor of this annealing treatment (ratio of CGRs between annealed and as-irradiated samples) is 3 in NWC and 17 in HWC. The constant extension rate tensile test results [27] also showed that the post-irradiation annealing at 550 °C for 1 and 5 h completely mitigated the IASCC susceptibility while annealing at 550 °C for 20 h reduced the percentage of intergranular fracture area.

The IASCC susceptibility of irradiated material is dominated by the irradiated microstructures such as dislocation loops, solute clusters and irradiation induced segregation. The reduce in IASCC susceptibility should be related to the recovery in those microstructures. From Table 4-2 and Figure 4-4, post-irradiation annealing at 550 °C for 20 h significantly reduces the number density of dislocation loop (only 0.4% remains) while the loop size increases. Table 4-3 and 4-4 show that the densities of Ni-Si and Al-Cu clusters decrease while the diameters increase after annealing treatments. The increase of cluster size is due to solute diffusion during annealing as the cluster become more diffuse as it gets larger [27]. The volume fraction of Ni-Si cluster changes slightly while that of Al-Cu cluster decreases from 0.035% to 0.016% after annealing at 550 °C for 20 h. The element compositions of the clusters change as well due to the difference in diffusivity of element. Ni-Si ratio increases as Si diffuses faster than Ni and Al-Cu ratio decreases due to the faster diffusion of Al. As to the element segregation, Table 4-5 and Figure 4-10 show that the depletion of Cr and enrichment of Ni and Si drop significantly, indicating that the grain boundary segregation is mostly recovered. All these microstructure changes would contribute to the decrease in IASCC susceptibility. The effect of each changes could not be separated at this stage.

5.3 Irradiated Microstructure

In general, the irradiated microstructure is expected to influence the irradiation hardening, and hence possibly IASCC susceptibility, as a function of the defect density and average size. Based on the theoretical hardening in a matrix, these factors are compared as a function of \sqrt{Nd} , where N is the number density of the irradiation defect and d is the average defect size [33]. Figure 5-5 compares the IASCC susceptibility, as measured by the fraction of intergranular fracture, and the \sqrt{Nd} of the three observed microstructural defects: dislocation loops, Ni-Si and Al-Cu solute clusters, displayed in orange. Except for the Al-Cu clusters at 550°C: 5hr, the theoretical impact of the microstructure generally decreases with IASCC susceptibility. However, while the 550°C: 1 and 5hr conditions have a higher impact from the residual microstructure as compared to the 550°C: 20hr condition, they displayed a full mitigation of IASCC susceptibility.

While previous PIA literature has indicated a clear relation between IASCC susceptibility and the irradiation hardening or yield stress [43], likely in relation to the reduction of the irradiated microstructure. In general, this study has also indicated a general trend between the irradiation hardness and yield stress and the measured IASCC susceptibility, as shown in Figure 5-5, where the mechanical properties are displayed in blue. Once again, this trend is broken by the PIA treatment of 550°C: 20hr, whereas 550°C: 1 and 5hr conditions have a higher irradiation hardening, but a reduced susceptibility. It is important to note that the fraction of intergranular fracture area, is not the only representation of the IASCC susceptibility, but the overall ductility is also relevant. Figure 5-5 displays a comparison between the intergranular fracture and the ductility, as measured by $1/\epsilon$, where a good agreement is observed between an increased ductility and decreased IASCC susceptibility. Though once again the 550°C: 20 hr condition shows a rather high ductility while also having an increased IASCC susceptibility.

The decrease in both the depletion of chromium and enrichment of nickel and silicon grain boundary segregation with annealing is also displayed in Figure 5-5, where the RIS data is plotted in green. With an increase of annealing time and temperature the segregation is increasingly reduced, but the lack of a complete data set makes a full comparison to the IASCC susceptibility difficult.

Overall, it was observed that PIA treatments are successful in reducing the irradiated microstructure, irradiation hardening, and IASCC susceptibility of 304L stainless steel BWR-irradiated to a dose of 5.9 dpa. While the irradiated microstructure, radiation-induced segregation, and hardening were all incrementally decreased with increasing annealing time and temperature, IASCC susceptibility was not evenly reduced. A full mitigation of IASCC occurred after 550°C: 1 and 5hr annealing treatments, but IASCC susceptibility returned after 550°C: 20hr annealing. Based on this data, it is suspected that the 550°C: 20 hr tensile specimen examined is an outlying result as compared to the remaining data. However, due to the small size of the data set, and the high value of neutron-irradiated material, it is difficult to fully remove the specimen, and a later examination is planned to study the specific irradiated microstructure of this specimen.

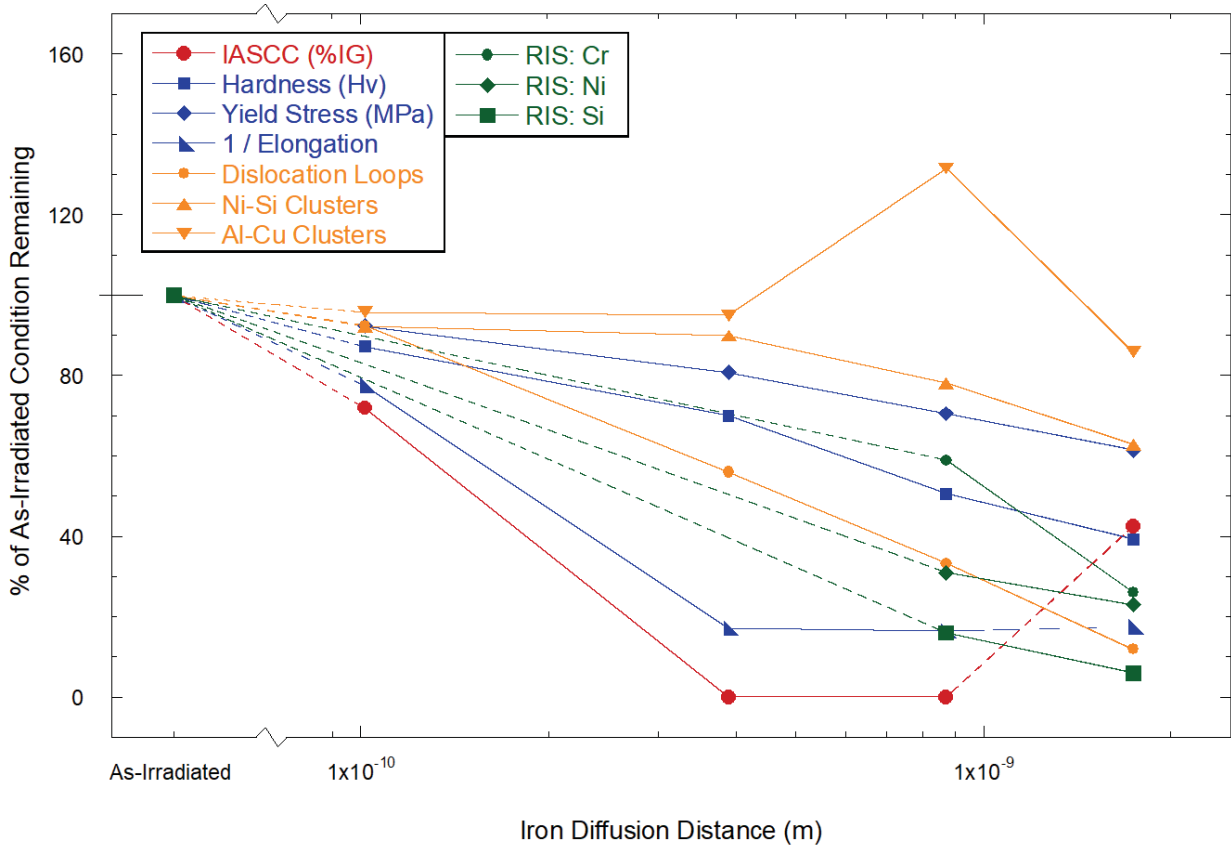


Figure 5-5. Comparison of the IASCC susceptibility to the mechanical properties and irradiated microstructure as a percentage of the as-irradiated condition.

5.4 Dislocation Channeling in IASCC

Localized deformation (dislocation channeling) plays a significant role in determining the cracking susceptibility of the material. A direct correlation has been observed between localized deformation and the cracking susceptibility of the material in environment. In this study, an attempt has been made to study the effect of PIA on localized deformation and to correlate it with the observed cracking behavior.

To study the role of channeling in crack initiation behavior, four-point bend tests were carried out on neutron irradiated specimens of alloy AS, ES and KS. The commercial purity alloy AS showed very high susceptibility to IASCC crack initiation in all three damage conditions (5.5 dpa, 10.2 dpa and 47.5 dpa). In all three conditions the crack was found to initiate at very low stresses (fraction of yield stress) as shown in Figure 4-48. Crack initiation was found to always be preceded by the activation of dislocation channels in the material. Upon further straining, the cracks were found to initiate at a dislocation channel-grain boundary intersection. An important observation was that with increase in the damage level in the material, the stress required to initiate a channel and initiate a crack was reduced as shown in Figure 5-6.

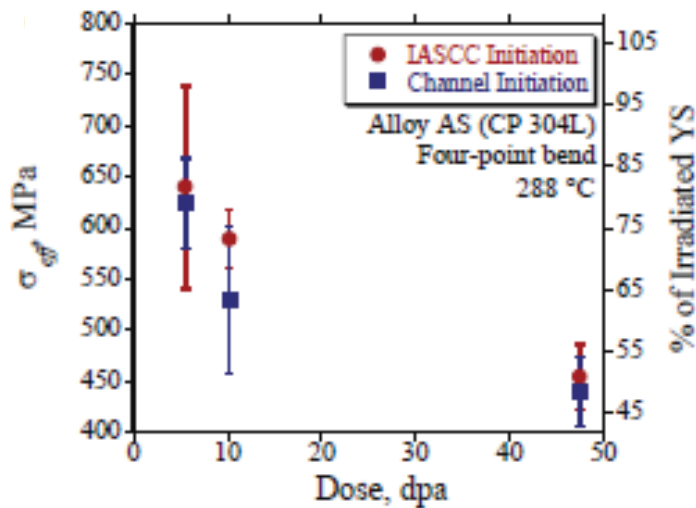


Figure 5-6: Average stress required to cause formation of dislocation channeling and crack initiation in commercial purity austenitic stainless steels as a function of damage level [44].

Furthermore, the majority of the cracking in alloy AS was found to occur at the intersection of dislocation channels with MnS inclusions. MnS inclusions were found to undergo dissolution in the environment resulting in the formation of a cap consisting of the dissolution product. This results in the formation of an aggressive environment below the cap. This environment combined with the intersection of a dislocation channel with the inclusion (thereby causing stress concentration) is believed to be the reason for cracks to initiate at these sites. Similar cracking behavior at MnS inclusions was previously observed by Jiao et al. [45] after proton irradiation of the same heat of material (Alloy AS). They attributed the crack initiation at the MnS inclusions to the cracking of the inclusion to the stress accumulation caused by dislocation channeling.

Comparison also needs to be made for the difference between the cracking behavior of three different alloy systems: AS (CP304L), ES (HP 304 L), and KS (HP + Ni). The difference between the three alloys lies in the composition. Alloy ES and KS shows higher resistance to crack initiation compared to alloy AS, as shown by the higher stresses required to cause cracking in these alloys. While alloy AS failed predominantly at the dislocation channel-inclusion intersection, such behavior was not observed for alloys ES and KS. Both of these alloys failed at the intersection of a dislocation channel with a grain boundary. Furthermore, the higher stress required to cause crack initiation shows that the alloys needed to be deformed to higher strain levels. This could be due to the lack of a microstructural-based stress riser like an inclusion, or perhaps, to the higher stacking fault energy (SFE) for these materials. Higher stacking fault energy (SFE) materials have been found to be more resistant to localized deformation and thus a higher strain is required to cause crack initiation in these alloys [46]. Among the alloys ES and KS, alloy KS was found to be most resistant to crack initiation owing to the higher stacking fault energy due to the additional nickel present in it.

For the 304L alloy irradiated in Barseback 1, the change in dislocation channel density for different annealing conditions and at different strain levels is shown in Figure 4-18. The dislocation channel density was unaffected by the annealing at 500°C:1hr and 550°C:1hr, but was reduced following the 550°C:5 hr annealing, and significantly reduced after PIA at 550°C:20 hr. This reduction in the

channel density can be attributed to the removal of irradiation induced defects during the annealing treatment, suggesting that the density of dislocation channels is primarily affected by the irradiation-induced defect densities, which agrees with previous work by Jiao *et al.* [6], in which the dislocation loop density was the primary contributor to the development of localized deformation. However, the dislocation channeling behavior for the tensile bar subjected to 550 °C:1hr annealing treatment shows high channel density even though a significant reduction in dislocation loop density was observed. Furthermore, a high recovery of ductility was observed in this condition indicating the removal of defects during the annealing treatment. From the data the amount of localized deformation behavior does not follow the annealing behavior, indicating that it is not just the channel density that is important, but perhaps other factors such as the stress at the channel-grain boundary intersection.

Localized deformation has been found to greatly influence the crack initiation behavior in austenitic stainless steels, particularly at sites where a discontinuous dislocation channel interacts with the grain boundary. [8,9]. With regard to the deformation behavior in these different annealing conditions, it is believed that cracking at the channel-grain boundary intersection requires a threshold stress be exceeded. Due to the reduction in the defect density during annealing treatments, the extent of localized deformation taking place in the material is reduced significantly for higher annealing treatments. For example, in the case of the as-irradiated state and lower annealing treatments, i.e. 500°C:1hr and 550°C:1hr, a significant portion of the plastic deformation could only be through the formation of dislocation channels, hence the higher density. While for the 550°C:20hr condition most of the plastic strain could be homogeneously accommodated, hence the much lower observed channel density. However, it can be speculated that even for the 500°C:1hr and 550°C:1hr conditions a certain portion of the bulk plastic strain may have been homogeneously distributed. The relative amount of strain included in dislocation channels, may reflect on the resultant IASCC susceptibility.

6 CONCLUSION

Crack initiation was studied by both CERT and four-point bend tests. CERT studies on neutron irradiated stainless steel alloys showed that the increase in the corrosion potential of the environment results in an increase in the cracking susceptibility of the alloy. At high potentials, the cracking susceptibility was determined by crack propagation since very few cracks nucleate and the nucleated crack rapidly grows to cause failure. At low potentials, cracking was predominantly controlled by crack initiation resulting in high crack density on the surface of the specimen. Thus, the degree of susceptibility depends on the figure of merit. When measured by %IG on the fracture surface, samples in NWC show greater susceptibility. But when measured by surface crack density, samples in PW are more susceptible.

Four-point bend tests were conducted to correlate the crack initiation behavior with dislocation channeling. The advantage of these tests is that the growth of crack nuclei was suppressed due to the increasingly compressive stress field normal to the surface. These tests were highly successful in catching crack initiation and linking it to the microstructure and dislocation channels. Depending on the dpa, dislocation channels form at stresses as low as 40% of the bulk yield stress. Cracks always nucleated at dislocation channel-grain boundary intersections either with the formation of the channel or after it. MnS inclusion on the grain boundary were often the site of crack nucleation and their dissolution in NWC is likely a key factor in producing an environment that is conducive to cracking. It was also found that increasing dpa resulted in a lower stress to both initiate dislocation channels and cracks.

Post irradiation annealing was found to mitigate the cracking susceptibility in 304L irradiated in the Barseback 1 BWR. With one exception, higher degrees of annealing (time+temperature) resulted in a decrease in SCC susceptibility. All measures of IGSCC susceptibility (max stress, uniform strain, total strain, %IG) changed monotonically with heat treatment severity. The exception was an increase in IGSCC in the most severely annealed sample, which is not explainable with the resulting microstructure. Close inspection of the microstructure showed that all features (dislocation loops, clusters, RIS) changed smoothly and monotonically and comparison of the microstructures at annealing treatments that bracketed the onset of cracking were not able to identify the feature or features responsible for cracking.

The CGRs of as-irradiated (5.9 dpa) and irradiated + annealed (550 °C:20 h) 304L stainless steel samples have been measured in NWC and HWC environments. The results show that HWC can significantly mitigate the SCC propagation of both samples. The mitigating effect may be dependent on the inherent SCC susceptibility of material. The post-irradiation treatment has a significant mitigating effect on the SCC propagation of this material with a larger effect in HWC. The reduced SCC susceptibility of the annealed sample is consistent with the recovery in the irradiated microstructure (dislocation loop, solute cluster and grain boundary segregation). However, the determining microstructure change could not be specifically identified.

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