

INITIAL TENSILE TEST RESULTS FROM J316 STAINLESS STEEL IRRADIATED IN THE HFIR SPECTRALLY TAILORED EXPERIMENT – J. E. Pawel, M. L. Grossbeck, A. F. Rowcliffe (Oak Ridge National Laboratory) and K. Shiba (Japan Atomic Energy Research Institute)

## OBJECTIVE

The objective of this work is to determine the effects of neutron irradiation on the mechanical properties of austenitic stainless steel alloys. In this experiment, the spectrum has been tailored to reduce the thermal neutron flux and achieve a He/dpa level near that expected in a fusion reactor.

## SUMMARY

The HFIR-MFE-RB\* experiments are designed for irradiation in the removable beryllium (RB\*) positions of the High Flux Isotope Reactor. A hafnium shield surrounds the capsules in order to reduce the thermal neutron flux and achieve a He/dpa level near that expected in a fusion reactor. The J316 austenitic alloy specimens irradiated in this experiment were in the solution annealed (SA) and 20% cold-worked (CW) condition. The specimens were irradiated at 60 and 330°C to a total of 19 dpa (11 appm He/dpa). For both irradiation temperatures, there was no significant difference between the strength properties of the CW J316 following irradiation to 7 dpa or 19 dpa. The strength properties saturate at a fluence less than 7 dpa. The same is true for the SA J316 irradiated at 60°C. However, at 330°C, there is a small but significant further increase in yield stress between 7 and 19 dpa. There is a marked difference in deformation behavior seen after irradiation at 60°C and 330°C. After irradiation to 19 dpa at 60°C, J316 maintains a uniform elongation greater than 20% while the uniform elongation of the 330°C material is less than 0.5%. The yield strength of the cold-worked material remains higher than that of the solution annealed material at both 7 and 19 dpa. The severe reduction in uniform elongation seen at 330°C is a synergistic effect of both the irradiation temperature and the test temperature.

## PROGRESS AND STATUS

### Introduction

An austenitic stainless steel with the 316 LN composition has been chosen for the first wall/shield (FW/S) structure for the International Thermonuclear Experimental Reactor (ITER). Austenitic stainless steel was selected because, in addition to favorable strength, toughness, and fabrication properties, there is an enormous reservoir of experience in fabricating and operating code qualified austenitic stainless steel components in nuclear systems. Also, the austenitics do not suffer from the ductile-to-brittle transition that is characteristic of body-centered-cubic alloy systems. However, a drastic loss of uniform elongation and work hardening capacity is often observed in tensile tests after irradiation at temperatures between 200 and 350°C [1-3]. Definition of the irradiation regimes in which this phenomenon occurs are essential to the establishment of design rules to protect against various modes of failure.

The HFIR-MFE-RB\* experiments are designed for irradiation in the removable beryllium (RB\*) positions of the High Flux Isotope Reactor (HFIR). A hafnium shield surrounds the capsules in order to reduce the thermal neutron flux and achieve a He/dpa level near that expected in a fusion reactor. The J316 austenitic alloy specimens irradiated in this experiment were in the solution annealed and 20% cold-worked conditions. They had been previously irradiated to about 7 dpa in the Oak Ridge Research Reactor (ORR) in dual-temperature capsules designated ORR-MFE-6J (which operated at 60 and 200°C) and ORR-MFE-7J (which operated at 330 and 400°C). The operating temperature of the HFIR-MFE-RB\*-60J-1 capsule was 60°C, with the specimens in direct contact with the reactor cooling water. The HFIR-MFE-RB\*-200J-1, 330J-1, and 400J-1 capsules operate at 200, 330, and 400°C, respectively, with the temperature actively controlled by changing the gas mixture around the specimen holder in response to 21 thermocouples located inside the holder. The 60J-1 and 330J-1 capsules were irradiated from July 1990 to November 1992 and accumulated approximately 12 dpa in the HFIR (in addition to 7 dpa in the ORR). The irradiation of the 200J-1 and 400J-1 capsules is in progress. Initial results obtained during this reporting period concerning

tensile properties of J316 solution annealed and cold worked alloys irradiated to about 19 dpa at 60 and 330°C are discussed in this report.

### Experimental Procedure

The composition of J316 is given in Table 1. Solution annealed (SA) and 20% cold-worked (CW) specimens were irradiated. The specimens were in the form of SS-1 flat tensile specimens with an overall length of 44.45 mm. The gage section of this type of specimen is 20.32 mm long by 1.52 mm wide by 0.76 mm thick.

The irradiation in the ORR produced approximately 75-100 appm He in the steel, giving a fusion-relevant He/dpa ratio of about 11 appm/dpa. The details of this irradiation are described elsewhere [4-6]. Some of these specimens were then re-encapsulated into irradiation vehicles designed to operate in the HFIR RB\* positions with a hafnium shield to reduce the thermal neutron flux and continue the irradiation at a He/dpa level near that expected in a fusion reactor. Two of the four capsules in this set have achieved the goal fluence in the HFIR of 11.6 dpa ( $1.94 \times 10^{22}$  n/cm<sup>2</sup>, E > 0.1 MeV) [7]. Preliminary dosimetry results also indicate that the 316 stainless steel specimens accumulated approximately 188 appm He in 60J-1 and 225 appm He in 330J-1 [7]. The helium content calculation was confirmed by isotope-dilution gas mass spectrometry [7]. This irradiation, combined with the ORR irradiation, resulted in a helium to displacement ratio of 10.2 appm He/dpa in 60J-1 and 11.8 appm He/dpa in 330J-1. Details of the irradiation can be found in Table 2 and elsewhere [7-9].

An Instron universal testing machine was used for the tensile testing. The specimens irradiated at 60°C (from the 60J-1 capsule) were tested at room temperature (25°C) in air. The specimens irradiated at 330°C (from 330J-1) were tested at 330°C under vacuum. In each case, the strain rate was 0.0004/s. The 0.2% offset yield strength (YS), ultimate tensile strength (UTS), uniform elongation (E<sub>U</sub>), and total elongation (E<sub>T</sub>) were calculated from the engineering load-elongation curves.

### Results and Discussion

The results of these tensile tests were compared with data from control specimens as well as with data taken from sibling specimens irradiated to 7 dpa in the ORR. These data, as well as the previously obtained results, are given in Tables 3 and 4. This report will focus primarily on the new, 19 dpa, data as the 7 dpa (60, 200, 330, and 400°C) data have been discussed previously [1, 3].

Table 1. Composition of the J316 (Japanese Atomic Energy Research Institute) Alloy

Alloy	Composition, weight percent											
	C	Mn	P	S	Si	Ni	Cr	Mo	Nb	Ti	Co	Fe
J316	0.058	1.8	0.028	0.003	0.61	13.52	16.75	2.46	<0.1	0.05	<0.1	bal.

Table 2. Maximum dpa and Helium Production (Reactor Centerline) in ORR and HFIR Irradiations

Experiment	dpa	He, appm	appm He/dpa
ORR-MFE-6J	6.9	75.3	
HFIR-MFE-RB*-60J-1	12	113	
Total	19	188	10
ORR-MFE-7J	7.4	102	
HFIR-MFE-RB*-330J-1	12	123	
Total	19	225	12

Table 3. Tensile Data From Solution Annealed J316.

Specimen	Dose, dpa (peak)	He, appm (peak)	He/dpa	Irradiation Temp., °C	Test Temp., °C	YS, MPa	UTS, MPa	E <sub>u</sub> , %	E <sub>t</sub> , %
EL-61	0	0	...	...	25	327	602	50	57
EL-26	0	0	...	...	25	283	552	50	54
EL-30	0	0	...	...	200	254	492	32	36
EL-62	0	0	...	...	200	263	503	31	35
EL-31	0	0	...	...	330	230	484	29	31
EL-63	0	0	...	...	330	262	508	28	35
EL-32	0	0	...	...	400	252	476	26	28
EL-64	0	0	...	...	400	222	476	33	35
EL-33	6.9	75	11	60	25	703	752	25	30
EL-34	6.9	75	11	60	25	690	745	28	33
EL-43	6.9	75	11	200	200	745	745	0.2	4.0
EL-44	6.9	75	11	200	200	758	765	0.2	16
EL-46	6.9	75	11	200	200	733	737	12	15
EL-1	7.4	100	14	330	330	848	855	0.3	3.1
EL-2	7.4	100	14	330	330	869	869	0.3	2.9
EL-14	7.4	100	14	400	400	595	677	4.6	7.0
EL-15	7.4	100	14	400	400	650	717	4.3	6.8
EL-16	7.4	100	14	400	25	725	850	17	21
EL-36	19	190	10	60	25	716	743	20	25
EL-37	19	190	10	60	25	747	765	20	26
EL-38	19	190	10	60	330	596	614	10	13
EL-4	19	220	12	330	330	903	913	0.4	3.1
EL-5	19	220	12	330	330	909	921	0.4	3.1

Table 4. Tensile data from 20% cold-worked J316.

Specimen	Dose, dpa (peak)	He, appm (peak)	He/dpa	Irradiation Temp., °C	Test Temp., °C	YS, MPa	UTS, MPa	E <sub>u</sub> , %	E <sub>t</sub> , %
FL-63	0	0	...	...	25	743	808	6.8	12
FL-26	0	0	...	...	25	672	723	14	21
FL-28	0	0	...	...	200	516	592	3.7	7.1
FL-64	0	0	...	...	200	685	724	1.0	4.3
FL-56	0	0	...	...	330	630	668	1.0	3.4
FL-65	0	0	...	...	330	632	674	1.0	3.7
FL-57	0	0	...	...	400	545	601	1.2	3.1
FL-58	0	0	...	...	400	403	487	1.3	3.4
FL-31	6.9	75	11	60	25	834	869	0.6	8.5
FL-32	6.9	75	11	60	25	855	883	0.6	7.5
FL-39	6.9	75	11	200	200	793	821	0.6	4.4
FL-42	6.9	75	11	200	200	843	870	0.5	3.3
FL-47	6.9	75	11	200	200	821	862	0.6	3.4
FL-13	7.4	100	14	330	330	862	876	0.4	2.3
FL-14	7.4	100	14	330	330	993	1007	0.3	2.0
FL-15	7.4	100	14	330	330	972	993	0.4	2.4
FL-3	7.4	100	14	400	400	800	848	1.6	3.4
FL-2	7.4	100	14	400	400	811	876	1.3	3.4
FL-37	19	190	10	60	25	840	852	0.6	10
FL-36	19	190	10	60	25	869	884	0.6	9.1
FL-16	19	220	12	330	330	988	1008	0.4	2.4
FL-17	19	220	12	330	330	1000	1011	0.4	2.9

Typical engineering stress-strain curves for the solution annealed material are shown in Figures 1 and 2. Zero strain is set where the modulus line extrapolates to the x-axis. However, this is not a true modulus line since it includes machine load train deflection. Irradiation at 60°C results in an approximately threefold increase in yield stress. Following an initial yield drop, the material work hardens at a rate similar to that of the unirradiated material and elongates ~20% before necking and failure occur. At both 6.9 and 19 dpa, the material work hardens after the yield point but the UTS is less than 10% higher than the YS. After irradiation and testing at 330°C (Figure 2), deformation behavior is significantly different. The increase in yield stress is greater than that at 60°C by ~200 MPa. After yielding, the material does not exhibit any work hardening capability. The applied load falls rapidly and failure occurs after only ~3% total elongation. The uniform elongation is less than 0.5%.

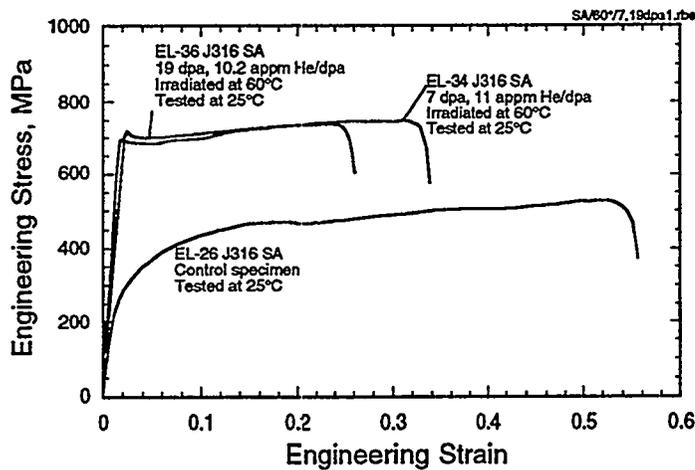


Figure 1. Engineering stress-strain curves for solution annealed material irradiated at 60°C and tested at 25°C. A control specimen curve is shown for comparison.

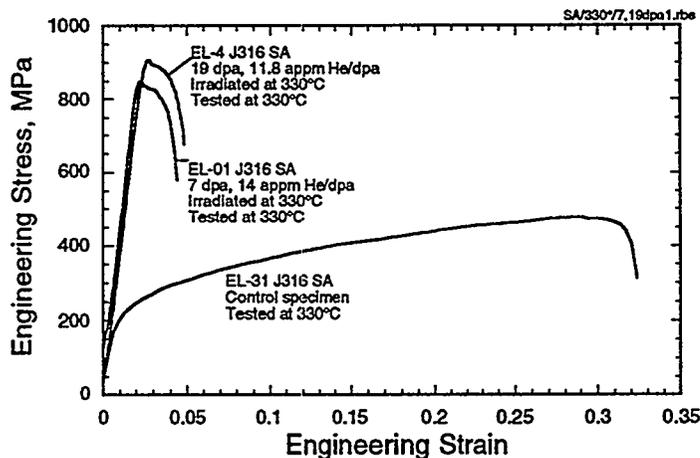


Figure 2. Engineering stress-strain curves for solution annealed material irradiated and tested at 330°C. A control specimen curve is shown for comparison.

The solution annealed material irradiated to 7 dpa showed a peak in the radiation-induced hardening curve as a function of temperature at 330°C (Figure 3). The 19 dpa data superimposed on this figure for comparison follow the same trend. This peak in hardening corresponds to a minimum in ductility.

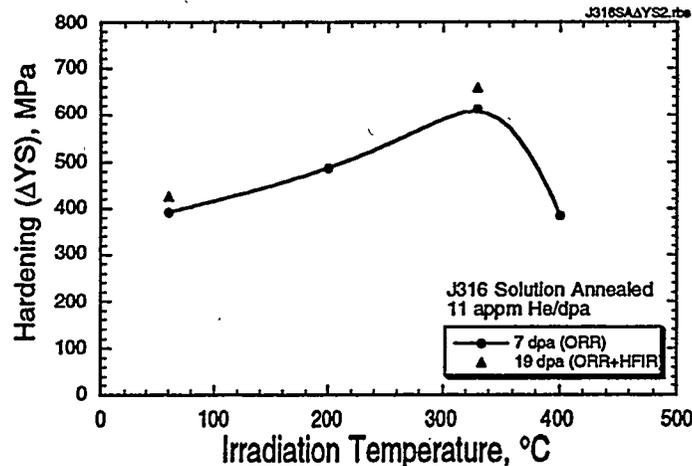


Figure 3. Radiation-induced hardening as a function of irradiation temperature for solution annealed J316. The curve is fit to the 7 dpa data.

The yield strength and the ultimate tensile strength of both the solution annealed and cold-worked specimens irradiated to a total of 19 dpa were only slightly higher than the values obtained after irradiation to 7 dpa (Figures 4 and 5). At both fluences, the yield strength is about equal to the UTS after irradiation and testing at these temperatures. There is some evidence that the strength as a function of fluence curve, as shown in Figures 4 and 5 should be steeper at the lower fluences. Tavassoli [10] reports significant increases in yield and ultimate tensile strength at fluences less than 1 dpa and saturation of these strengths at approximately 3 dpa for 316LN irradiated at temperatures less than 400°C. Kallstrom et al. [11] irradiated 316LN to only 0.3 dpa at 35°C and report a yield strength of 552 MPa after testing at 75°C. Heinisch [12] also reports significant increases in the yield strength of 316 as the result of very low dose irradiations (< 0.01 dpa). The present data are not inconsistent with these observations since displacement levels below 7 dpa were not investigated.

Irradiation to 7 and 19 dpa at either temperature resulted in significant hardening, rather than softening, of the cold-worked material. The yield strength of the cold-worked material remains higher than the that of the solution annealed material at both 7 and 19 dpa, but the difference is less at 330°C than at 60°C (Figures 6 and 7). This observation is consistent with Grossbeck et al. [2, 13], who reported convergence of the SA and CW yield strengths after irradiations to dpa levels between 20 and 30. Elen and Fenici [14] report CW strengths 200 MPa higher than solution annealed strengths for irradiations of 316L up to 11 dpa at 250°C. Garner et al. [15] also reported hardening of CW 316 after irradiation at 427°C in a fast reactor; similar irradiations at 538 and 650°C resulted in softening.

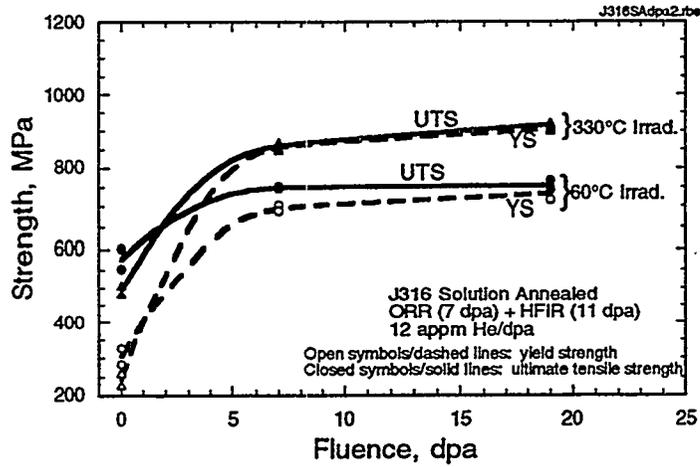


Figure 4. Yield strength and ultimate tensile strength of solution annealed J316 as a function of fluence for 60 and 330°C irradiations.

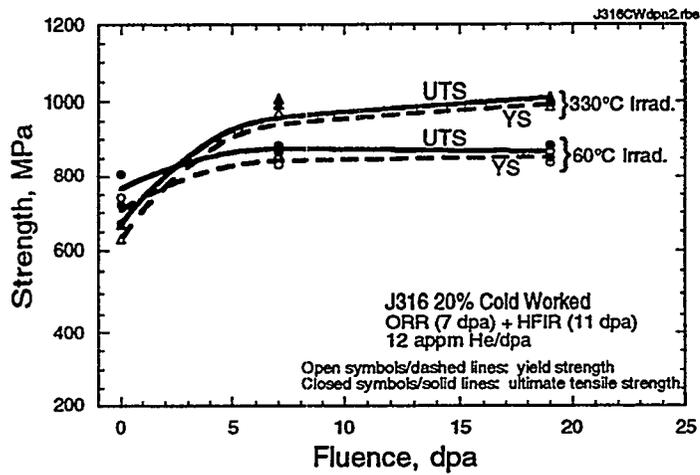


Figure 5. Yield strength and ultimate tensile strength of cold-worked J316 as a function of fluence for 60 and 330°C irradiations.

To further investigate the behavior seen after irradiation at 330°C, one solution annealed specimen irradiated to 19 dpa at 60°C was tested at 330°C. Compared to the specimens irradiated and tested at 330°C, this specimen showed less hardening and larger  $E_{11}$ . Compared to the 60°C specimens, this specimen showed less hardening but also less  $E_{11}$ . This latter comparison is consistent with the unirradiated data, which show a decrease in both YS and  $E_{11}$  with increasing test temperature. The stress-strain curves are shown in Figure 8. This implies that the severe reduction in uniform elongation seen at 330°C is a synergistic effect of both the irradiation temperature and the test temperature.

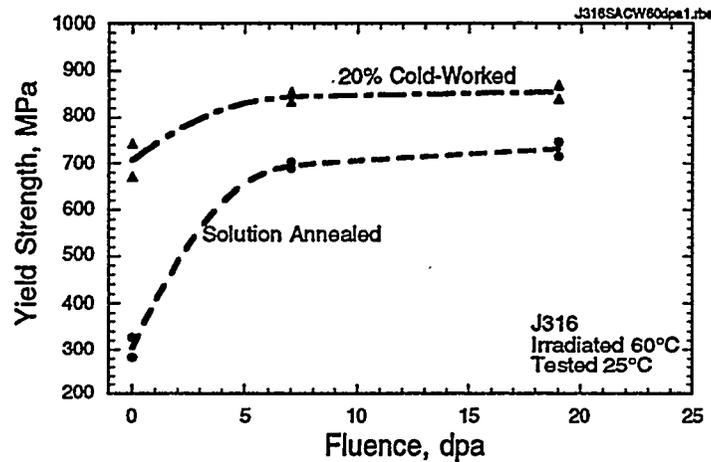


Figure 6. Yield strength as a function of fluence for solution annealed and cold-worked J316 material irradiated at 60°C.

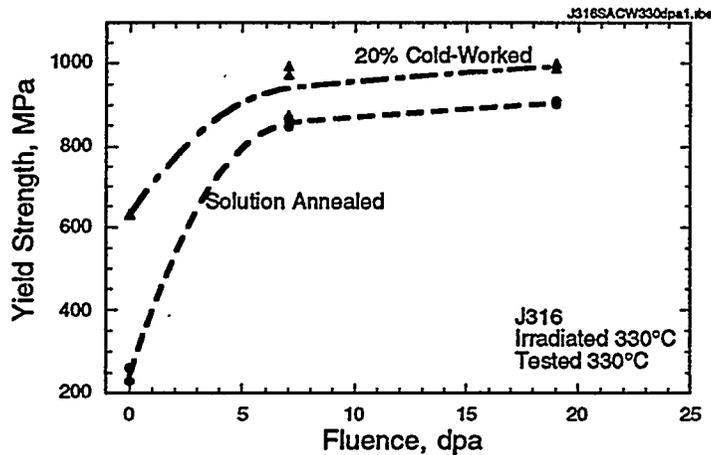


Figure 7. Yield strength as a function of fluence for solution annealed and cold-worked J316 material irradiated at 330°C.

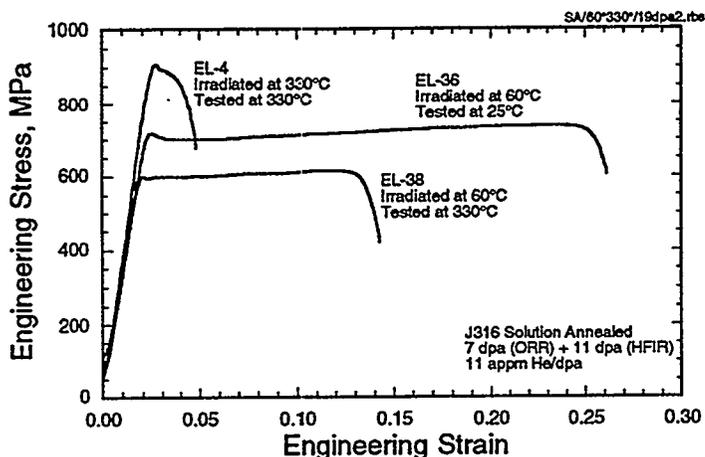


Figure 8. Engineering stress-strain curves of solution annealed J316 irradiated to 19 dpa at 60 and 330°C, and tested at 25 and 330°C.

### Conclusions

For both irradiation temperatures, there was no significant difference between the strength properties of the CW J316 following irradiation to 7 dpa or 19 dpa. The strength properties saturate at a fluence less than 7 dpa. The same is true for the SA J316 irradiated at 60°C. However, at 330°C there is a small but significant further increase in yield stress between 7 and 19 dpa. There is a marked difference in ductility behavior seen after irradiation at 60°C and 330°C. Both have high yield strengths compared to the unirradiated values, but the 60°C material maintains a uniform elongation greater than 20% while the uniform elongation of the 330°C material is less than 0.5%. The yield strength of the cold-worked material remains higher than that of the solution annealed material at both 7 and 19 dpa. The severe reduction in uniform elongation seen at 330°C is a synergistic effect of both the irradiation temperature and the test temperature.

### FUTURE WORK

Future work includes analyses to better define the low work hardening regime as a function of fluence and temperature and the comparison of these data with other results on the basis of helium content, He/dpa ratio, and dose rate. In particular, similar materials irradiated at 200 and 400°C remain to be tested.

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