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TENSILE PROPERTIES OF TYPE 316 STAINLESS STEEL IRRADIATED IN A SIMULATED FUSION REACTOR ENVIRONMENT*

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Tensile and fracture properties of type 316 stainless steel in the annealed and 20% cold-worked conditions were investigated to neutron fluences producing 16 dpa and 1000 at. ppm He resulting from irradiation in the High Flux Isotope Reactor. Tensile elongations remained above 6% at 350 and 450°C and 4% at 575°C. Fracture behavior has been examined in the 20% cold-worked material. At 350°C the fracture mode is unchanged by irradiation and is characterized by ductile tearing. At 450°C irradiation causes the fracture mode to change from ductile tearing to crystallographic fracture which may occur along the slip bands introduced by the cold working. At 575°C the fracture is changed from ductile transgranular to intergranular. The tensile properties observed are believed to be adequate for fusion reactor service within the values of damage parameters investigated.

1. INTRODUCTION

In a fusion reactor the first wall will be subjected to intense high-energy neutron irradiation, resulting in displacement damage and the formation of helium and hydrogen from transmutation reactions. Since type 316 stainless steel (referred to as 316) and compositional variations of this alloy are being considered for first-wall applications, the effects of the fusion reactor neutron irradiation on the ductility, strength, and fracture mechanisms are of interest. Because of the high production rate of helium in a fusion reactor, property measurements from irradiation in fast reactors, where very little helium is produced, are not directly applicable. In this investigation, irradiations were conducted in the High Flux Isotope Reactor (HFIR) which has a mixed energy neutron spectrum with a high fast flux providing a high displacement damage rate and a high thermal flux providing helium through a two-step transmutation reaction with the nickel in the stainless steel [1].

Previous investigators [2] have irradiated the same heat of 316 in HFIR to damage levels of 2–4 dpa and 35–60 dpa and reported very low ductility levels. However, no mechanical property measurements previously existed in 316 irradiated to intermediate damage levels. The purpose of this study is to evaluate strength and ductility and to study fracture of 316 in the range of 5–16 dpa and 200–1000 at. ppm He. Additional irradiation experiments to cover the range of 15–35 dpa have been initiated.

2. EXPERIMENTAL PROCEDURE

Miniature tensile specimens with a gage length of 18.3 mm and gage diameter of 2.03 mm were fabricated from 316 of the composition shown in Table 1. The specimens were irradiated in HFIR in a peripheral target position, where the peak thermal flux is 2.5×10^{19} n/m^2 s and the peak fast flux is 1.3×10^{19} n/m^2 s (>0.1 MeV). The specimen holder is of the same design as that used by Wiffen [3]. A gas gap was provided around each specimen to restrict radial heat transfer to obtain the desired temperatures.

Table 1. Composition of Type 316 Stainless Steel

Content, wt %		Content, wt %	
Cr	18.0	C	0.05
Ni	13.0	P	0.013
Mo	2.58	S	0.016
Mn	1.9	N	0.05
Ti	0.05	B	0.0005
Si	0.8	Fe	bal

Specimen material was initially annealed 1 h at 1150°C then swaged and annealed to obtain the proper diameter with a final anneal of 1 h at 1050°C. The cold-worked material (referred to as 20% C.W. 316) was obtained by then swaging to 20% reduction in area.

No instrumentation was installed in the irradiation capsule; however, extensive dosimetry has been done in the past and temperature monitors from more recent irradiations are now being analyzed. Preliminary analysis of temperature monitors indicates that irradiation temperatures may have been higher than calculated by a maximum of 50–75°C. However, the calculated temperatures are consistent with previously reported data, and the comparison between annealed and cold-worked material remains valid. Helium levels were calculated from an empirical relation determined by Wiffen et al. [4] and based on mass spectrographic analysis of HFIR irradiated specimens. The calculations are believed to have an accuracy of ±25%.

Tensile tests were conducted on an Instron tensile testing machine using a crosshead speed of 0.85 μm/s, resulting in a nominal strain rate

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of 4.6 × 10^{-5}/s. Tests were performed in air in a resistance furnace with a 150-mm hot zone to provide temperature uniformity over the center region of 50 mm. Test temperatures were selected as close as possible to calculated irradiation temperatures, and specimens tested at 350, 450, and 575°C were chosen for analysis. Fluences ranged from 0.63 to 2.1 × 10^{26} n/m^2 (>0.1 MeV), displacement damage levels from 5 to 16 dpa, and helium contents ranged from 200 to 1000 at. ppm.

Following tensile testing, reduction of area measurements were made. This was accomplished by making a low magnification fractograph in a scanning electron microscope. The fracture surface area was measured and reduction of area computed using a standard for magnification calibration for each specimen. The true fracture stress was obtained from the load and area at fracture.

3. RESULTS AND DISCUSSION

Results of the tensile property measurements are tabulated in Table 2. The yield stresses (0.2% offset) as functions of fast neutron fluence, dpa, and helium content for all samples at the test temperatures of 350, 450, and 575°C are plotted in Fig. 1. In cases where data are sparse, especially for the annealed condition, the data points are connected by straight-line segments.

For 450 and 575°C, annealed 316 initially strengthens with increasing fluence then appears to approach a constant value. Strengthening is also evident at 350°C although the data are limited. The increase in yield strength is accompanied by an increase in ultimate tensile strength (UTS) at 350 and 450°C but by a decrease in the UTS at 575°C, caused by fracture occurring

Table 2. Mechanical Properties of HFIR-Irradiated Types 316 and TiM 316 Stainless Steel

Temperature, °C		Neutron Fluence[b]	Helium Content[c]	Strength, MPa		Elongation, %		Reduction of Area	Fracture Stress
Test	Irradiation[a] ±50°C	>0.1 MeV (n/m^2 ×10^{26})	(at. ppm)	Yield	Ultimate	Uniform	Total	(%)	(MPa)
				Annealed Type 316					
350[d]		0		165	620	34	41		
350		0		176	558	32	37	70	1750
350	375	1.7	740	786	869	4.6	6.9	36	1100
450		0		156	565	32	39	60	1000
450	465	1.4	600	399	591	8.4	9.7	15	310
450	475	2.1	980	345	586	12	14	43	900
575[d]		0		138	524	32	38		
575	565	1.2	440	248	468	6.4	7.7	12	410
575	565	1.9	880	236	467	6.1	6.3	15	510
				20%-Cold-Worked Type 316					
300	325	1.0	390	998	998	0.22	5.1	83	4050
350		0		527	633	12	17	51	850
350		0		585	676	7.8	13	64	1250
350	370	0.63	180	780	848	4.5	9.4	59	1400
350	370	1.0	390	855	917	4.2	8.7	58	1450
350	375	1.1	380	611	752	3.3	6.8	54	1200
350	375	1.7	740	594	731	3.3	6.4	53	1150
350	375	1.7	740	688	800	4.6	8.3	53	1250
450		0		496	641	12	18	62	1100
450		0		572	663	8.3	14	56	750
450		0		542	645	10	16	63	1200
450	465	0.9	290	481	641	11	14	38	860
450	475	1.3	500	405	584	8.3	11	46	870
450	465	1.4	600	459	619	10	12	28	700
450	475	2.1	1020	395	613	11	13	30	760
575		0		480	576	10	17	63	990
575		0		638	758	8.2	15	68	1400
575		0		512	589	8.3	15	60	900
575	600	0.9	290	296	503	11	13	33	700
575	565	1.2	440	332	510	4.4	4.6	11	530
575	560	1.4	800	341	538	9.5	12	16	580
550	560	1.9	880	310	490	4.4	4.6	14	550
575	620	2.1	1020	290	448	4.8	4.9	14	500

[a] Irradiation temperatures calculated.

[b] Calculated from dosimetry of previous experiments.

[c] Calculated from empirical equation.

[d] From D. Fahr, ORNL/TM-4292 (November 1973).

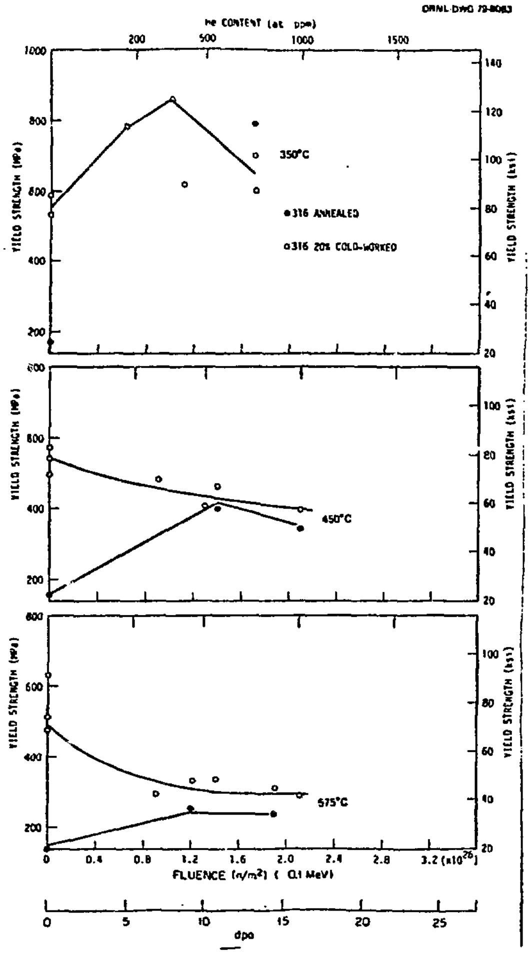

Fig. 1. Yield strength versus fluence, dpa, and helium content of type 316 stainless steel for test temperatures of 350, 450, and 575°C.

near the UTS at 575°C. Cold-worked 316 tested at 350°C also strengthens rapidly but appears to decrease in strength at fluences between 1.0 and 1.7×10^{26} n/m^2. At the two higher temperatures the yield strength of cold-worked material decreases with a progressively decreasing rate as fluence increases, primarily as a result of recovery of the cold work dislocation structure. The yield strengths of the annealed and cold-worked materials approach common values with increasing fluence at 450 and 575°C, and the strength of both materials decreases with increasing temperature at a given fluence. The initial strengthening in 20% cold-worked 316 at 350°C is more rapid than observed in other 20% C.W. 316 irradiated in EBR-II [5]. It is felt that this rapid strengthening results at least in part from microstructural differences resulting from the presence of helium. In contrast to HFIR, EBR-II produces only trace amounts of helium at the fluence levels attained in this investigation.

Total tensile elongation has been plotted in Fig. 2. Very abrupt decreases in ductility are observed for the annealed 316 at all test temperatures, probably due to irradiation strengthening. However, there are insufficient data to draw conclusions about the behavior beyond 1.5×10^{26} n/m^2. The ductility of 20% C.W. 316 also decreases less dramatically than the annealed 316 with increasing fluence at all three temperatures and at 450 and 575°C is accompanied by decreasing strength, a characteristic of severe high-temperature irradiation embrittlement. This decrease in ductility is much slower than for annealed 316 except for the rather rapid rate of decrease at 575°C at fluences greater than 1.5×10^{26} n/m^2. Nonetheless, even at 575°C, where ductility is lowest, it remains above 4% for fluences below 2.1×10^{26} n/m^2, the highest examined. This high ductility is inconsistent with the results of earlier investigations by Bloom and Wiffen [2] of HFIR-irradiated 20% C.W. 316 which reported total tensile elongations below 1% at fluences as low as 0.4×10^{26} n/m^2 at 575°C. Although this inconsistency is not understood at the present time, it is believed to arise at least in part from differences in fabrication of the specimen material since the same heat of material was used for the previous tests and since this material was less ductile prior to irradiation. The yield strength of the material investigated by Bloom and Wiffen [2] was approximately 50% higher than that used in the present study. This higher strength with correspondingly lower ductility in the unirradiated condition may be responsible for the lower ductilities after irradiation reported by Bloom and Wiffen. However, just as strength and ductility approach common values with increasing fluence, materials with different levels of cold work are also expected to approach a common value. Therefore at high fluences (8×10^{26} n/m^2), where Bloom and Wiffen [2] observed tensile elongations below 1%, it is felt that the results may be used as a guide to extrapolation of the present data.

Although uniform and total tensile elongations are useful measures of ductility for materials application, local plastic deformation is of greater interest for the study of fracture mechanisms, and this is better described by reduction of area. Values of reduction of area and true fracture stress appear in Table 2. Fracture stress closely parallels reduction of area, and there is a rather limited amount of data for material in the annealed condition. Therefore, only reduction of area for the cold-worked condition will be discussed. In Fig. 3 reduction of area has been plotted accompanied by representative fractographs for 20% C.W. 316. Reduction of area is seen to decline more rapidly with increasing temperature but approaching a constant value above 10% at 1.2×10^{26} n/m^2 for 575°C. At 350°C the fracture surfaces reveal ductile

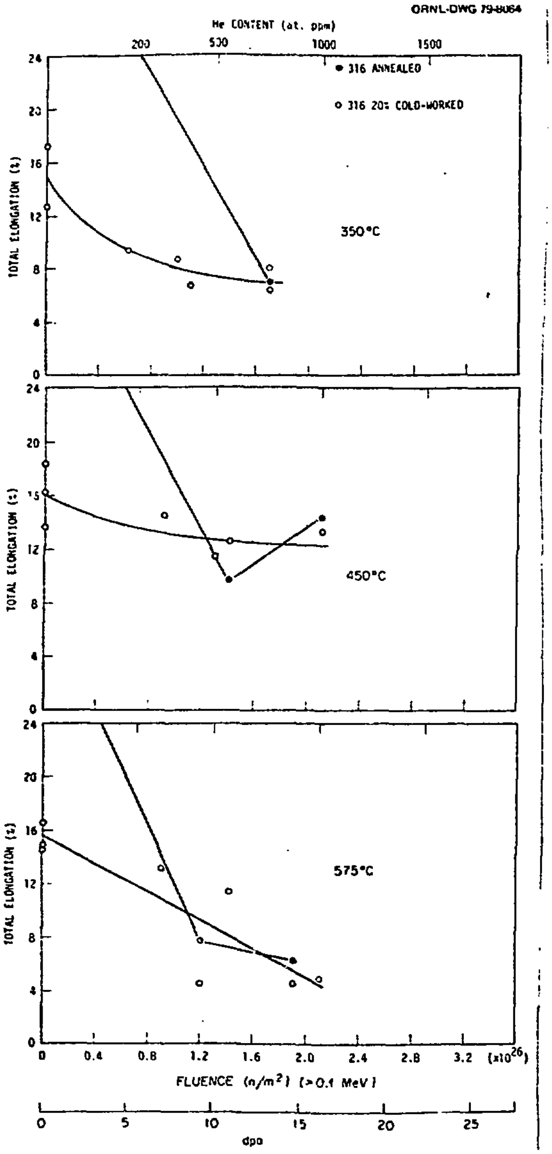

Fig. 2. Total tensile elongation versus fluence, dpa, and helium content of type 316 stainless steel for test temperatures of 350, 450, and 575°C. The elongations for annealed, unirradiated material are given in Table 2.

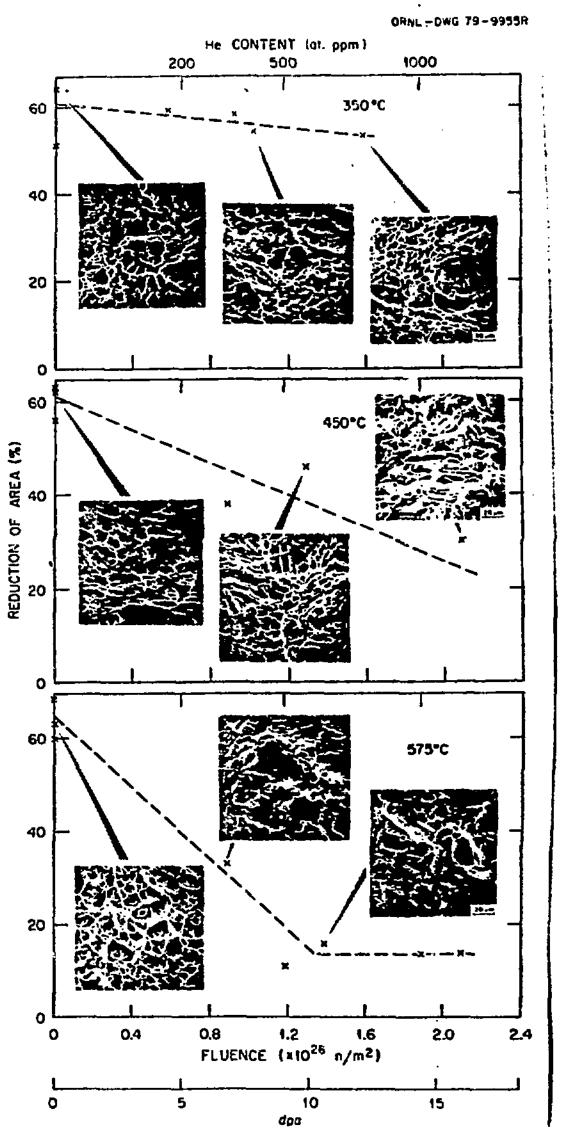

Fig. 3. Reduction of area versus fluence, dpa, and helium content for 20%-cold-worked 316 irradiated in HFIR with accompanying fractographs.

rupture, becoming finer in texture with increasing fluence, probably as a consequence of irradiation hardening. At 450°C crystallographic features absent in annealed 316 appear on the fracture surface of specimens subjected to a fast fluence above about 1×10^{26} n/m^2 which suggest cleavage or channel fracture [6]. Channel fracture is characterized by facets on the fracture surface but oriented 45° to the tensile axis suggesting a shear process. It has been positively identified only in highly irradiated annealed type 304 stainless steel. Cleavage can be eliminated as a candidate since river lines, characteristic of cleavage, are absent (Fig. 4a),

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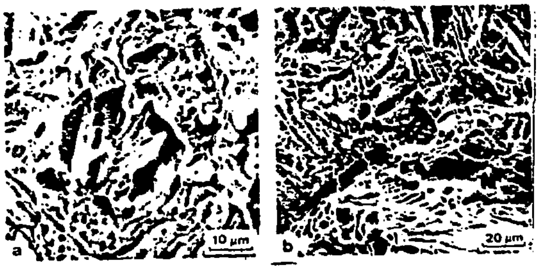

Fig. 4. Fractographs of HFIR-irradiated 20% C.W. 316 tested at 450°C. (a) Specimen showing crystallographic features [fluence = 1.4×10^{26} n/m^2 (>0.1 MeV), 600 at. ppm He], (b) specimen showing bubbles [fluence = 1.3×10^{26} n/m^2 (>0.1 MeV), 500 at. ppm He].

and cleavage fracture has never been observed in austenitic stainless steels. As Fig. 4b illustrates rather well, the plate-like fracture is accompanied by cavities 1–2 μm in diameter which are not accompanied by cup-like tearing characteristic of microvoid coalescence. There is even evidence that such cavities, which might contain helium, align in strings and link up along edges of the flat areas. From this evidence and from details of the original cold-worked structures (to be published), it is suggested that the cavities form either along slip bands resulting from preirradiation cold work or on precipitates which nucleate along slip bands during irradiation [7] in concentrations large enough to nucleate cracks along the weakened slip bands, thus producing the crystallographic features on the fracture surface.

At 575°C a rapid drop in reduction of area is accompanied by the onset of intergranular fracture (Fig. 3), a well-established type of failure by helium embrittlement. The rapid decline in reduction of area is followed by an apparent leveling-off at a value of approximately 10% beginning at a fluence of 1.3×10^{26} n/m^2. Although the plateau in reduction of area is encouraging, the fluence value attained is not high enough to warrant excessive optimism. Previous HFIR experiments have indicated total elongations below 1% at fast fluences of 8×10^{26} n/m^2 and helium contents of 4000 at. ppm He.

4. CONCLUSIONS

1. For 20% C.W. 316 irradiated to damage levels up to 16 dpa and 1000 at. ppm He, tensile elongation remains above 4% and reduction of area above 10% at temperatures as high as 575°C.
2. In 20% C.W. 316, an unexpected crystallographic fracture mechanism is observed at 450°C.
3. The appearance of a new fracture mechanism associated with residual slip bands from cold work emphasizes the sensitivity of mechanical properties to microstructure and suggests the importance of microstructural control in developing improved alloys.
4. The tensile strength and ductility of all materials examined appear adequate for early life as structural materials for a fusion reactor.

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