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TENSILE BEHAVIOR OF THREE COMMERCIAL FERRITIC STEELS AFTER LOW-TEMPERATURE IRRADIATION*

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The ferritic (martensitic) steels and the austenitic stainless steels are being considered for use as first wall and blanket structural components for fusion reactors. Tensile specimens of normalized-and-tempered 9 Cr–1 MoVNb and 12 Cr–1 MoVW steels, normalized-and-tempered and isothermally annealed 2¼ Cr–1 Mo steel, and 20%-cold-worked type 316 stainless steel were irradiated at approximately 50°C to damage levels of up to about 9 displacements per atom (dpa) in the High Flux Isotope Reactor (HFIR). The preirradiated microstructures of the 9 Cr–1 MoVNb and 12 Cr–1 MoVW steels were a tempered martensite; the microstructure of the normalized-and-tempered 2¼ Cr–1 Mo steel was tempered bainite, and that of the isothermally annealed 2¼ Cr–1 Mo steel was primarily polygonal ferrite. The postirradiation tensile behavior at room temperature and 300°C of all alloys was similar: irradiation hardening was observed as increased yield strength, increased ultimate tensile strength, and decreased ductility (uniform and total elongation). After irradiation, normalized-and-tempered 2¼ Cr–1 Mo and 12 Cr–1 MoVW had similar strengths and were stronger than the 9 Cr–1 MoVNb and type 316 stainless steel, which had similar strengths. The irradiated isothermally annealed 2¼ Cr–1 Mo steel hardened to a value considerably below the other four steels, although this material was also much weaker than the other steels before irradiation. With one exception, there was relatively little difference in ductility. At 300°C the ductility of the isothermally annealed 2¼ Cr–1 Mo steel was substantially better than for the other steels.

INTRODUCTION

The irradiation-resistant properties of the high-chromium ferritic (martensitic) steels based on 9% Cr-1% Mo and 12% Cr–1% Mo have been demonstrated in the fast breeder reactor program (1,2). As a result, these alloys are now being considered for use as fusion reactor first-wall and blanket structural components. However, because the criteria for materials for breeder reactors and fusion reactors differ, it may also be possible to use a lower chromium ferritic steel for fusion reactor applications (3).

We previously reported on the tensile properties of irradiated 9 Cr–1 MoVNb (ref. 4) and 12 Cr–1 MoVW (ref. 5) steels. In the present work, the irradiated tensile properties of 2¼ Cr–1 Mo steel were determined and were compared with those for 9 Cr–1 MoVNb and 12 Cr–1 MoVW steels irradiated and tested under similar conditions. Because 20%-cold-worked type 316 stainless steel (CW 316) is often used as a reference, the tensile properties of this material were also determined after irradiation under similar conditions.

EXPERIMENTAL PROCEDURE

Chemical compositions of the alloys tested are given in Table I. The 9 Cr–1 MoVNb and the 12 Cr–1 MoVW steels were electroslag remelted by Combustion Engineering, Inc., Chattanooga, Tennessee. The 2¼ Cr–1 Mo steel was from a commercial heat obtained from Babcock and Wilcox Corporation (heat 72768). The type 316 stainless steel was taken from the fusion reference heat (X15893). Prior to heat treatment of the ferritic steels, they were rolled to 0.76-mm sheet. The type 316 stainless steel was annealed for 1 h at 1050°C prior to the final 20% cold work.

Table I. Chemical Compositions of Steels

Element[a]	Alloy Content, wt % 2¼ Cr–1 Mo (72768)	9 Cr–1 MoVNb (XA-3590)	12 Cr–1 MoVW (XAA-3587)	316 SS (X15893)
C	0.12	0.09	0.21	0.061
Mn	0.48	0.37	0.50	1.70
P		0.011	0.011	0.037
S		0.004	0.004	0.18
Si	0.31	0.19	0.18	0.67
Ni	0.07	0.09	0.43	12.44
Cr	2.2	8.47	11.99	17.28
Mo	0.8	0.88	0.93	2.10
V		0.21	0.27	
Nb		0.07	0.018	<0.05
Ti	0.01	0.001	0.003	<0.05
Co	0.003	0.017	0.017	0.3
Cu	0.1	0.03	0.05	0.3
Al		0.009	0.030	
B		0.0006	<0.001	0.0004
W		0.01	0.54	
Sn		0.003	0.002	
N	0.016	0.050	0.020	
O	0.007	0.007	0.005	

[a]Balance iron.

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Ferritic steels are often used in a normalized-and-tempered condition. In steelmaking terminology, normalizing consists of heating the steel above the A_{c3} temperature to transform the steel to austenite (austenization) and then air cooling. For the present study, we austenitized the three ferritic steel tensile specimens in a tube furnace containing a dynamic helium atmosphere, after which the specimens were pulled into the cold zone and cooled in flowing helium. The tempering treatments were carried out in the same helium-atmosphere furnace with the same cooling procedure.

The 9 Cr–1 MoVNb steel was austenitized 0.5 h at 1040°C and tempered 1 h at 760°C, while the 12 Cr–1 MoVW steel was austenitized 0.5 h at 1050°C and tempered 2.5 h at 780°C (ref. 4). For the 2¼ Cr–1 Mo steel, austenization was for 0.5 h at 900°C; tempering was 1 h at 700°C.

In addition to the normalized-and-tempered heat treatment the 2¼ Cr–1 Mo steel was also irradiated and tested in an isothermally annealed condition. The isothermal anneal was carried out in a helium atmosphere. This heat treatment consisted of heating for 0.5 h at 900°C, furnace cooling to 700°C, holding for 2 h, then cooling in flowing helium.

The sheet tensile specimens had reduced gage sections 20.3-mm long by 1.5-mm wide by 0.76-mm thick and were irradiated in HFIR at 50–55°C (the reactor coolant temperature). The maximum total fluence was 5×10^{26} neutrons/m^2 and the fast fluence 1.3×10^{26} neutrons/m^2 (>0.1 MeV); this resulted in a maximum of 9 dpa. Some of the specimens tested in this experiment were taken from capsule positions that received less than the maximum radiation dose. After immersion density measurements, tensile tests were conducted at room temperature and 300°C in a vacuum chamber on a 44-kN-capacity Instron universal testing machine at a strain rate of 4×10^{-5}/s. Unirradiated control specimens were tested for each steel.

RESULTS AND DISCUSSION

After irradiation, immersion density measurements were made on all specimens. No detectable change in density was observed on any of the specimens.

Measured tensile properties are given in Table II. Table II also lists the calculated displacement levels and the helium concentration resulting from irradiation. Helium is generated by the thermal portion of the neutron spectrum by a two-step transmutation reaction with ^{58}Ni and by transmutation of ^{10}B. In the previous work on 9 Cr–1 MoVNb and 12 Cr–1 MoVW steels, the effect of helium on the low-temperature tensile properties (room temperature and 300°C) was investigated by irradiating nickel-doped steels (4,5). No helium effect was detected; helium effects will not be further discussed in this paper. It should be noted, however, that the CW 316 contains orders of magnitude more helium than the ferritic steels. Such high concentrations could result in an increased hardening above that due to displacement damage. Essentially all of the hardening in the ferritic steels is due to the displacement damage.

Table II. Tensile Properties of Unirradiated and Irradiated[a] Steels

Fluence (>0.1 MeV) (neutrons/m^2)	Displacement Level (dpa)	Helium Concentration[b] (at. ppm)	Test Temperature (°C)	Strength (MPa) Yield	Strength (MPa) Ultimate	Elongation (%) Uniform	Elongation (%) Total
		9 Cr–1 MoVNb Steel (NT)[c]					
$\times 10^{26}$							
0			25	541	656	5.1	9.6
1.3	9.3	11	25	878	878	0.2	3.2
0			300	483	581	3.6	7.1
1.1	7.6	10	300	716	716	0.2	3.7
		12 Cr–1 MoVW Steel (NT)					
0			25	553	759	8.1	11.2
1.3	9.3	26	25	980	992	0.4	2.9
0			300	483	652	5.1	8.0
1.2	9.1	18	300	783	815	1.8	5.1
		2¼ Cr–1 Mo Steel (NT)					
0			25	581	663	8.4	12.8
0.9	6.4	8	25	1027	1027	0.1	1.7
0			300	541	632	6.3	9.4
1.2	8.6	9	300	807	809	0.4	3.8
		2¼ Cr–1 Mo Steel (IA)[d]					
0			25	372	504	14.6	19.0
1.2	8.6	9	25	729	729	0.1	3.0
0			300	343	528	8.8	13.1
1.2	8.6	9	300	552	574	6.7	10.1
		Type 316 Stainless Steel (CW)[e]					
0			25	665	743	11.1	14.9
1.3	9.5	416	25	892	905	0.8	7.7
0			300	595	645	1.8	4.4
1.1	7.8	305	300	723	731	0.6	3.3

[a]Irradiation was in HFIR at ~ 50°C. [b]Calculated level of helium from ^{58}Ni and ^{10}B.
[c]NT = normalized and tempered. [d]IA = isothermal anneal. [e]CW = 20% cold worked.

After normalizing and tempering, the microstructures of the 9 Cr–1 MoVNb [Fig. 1(a)], 12 Cr–1 MoVW [Fig. 1(b)] steels were entirely tempered martensite. For the 2¼ Cr–1 Mo steel in the normalized-and-tempered condition, the microstructure was essentially 100% tempered bainite [Fig. 1(c)]. In the isothermally annealed condition, the microstructure developed was 75–80% polygonal ferrite, the balance primarily bainite [Fig. 1(d)].

The tensile results for the 2¼ Cr–1 Mo steel depend on the microstructure, as can be seen from the engineering stress-strain curves in Figs. 2 and 3. Curves for the 9 Cr–1 MoVNb and 12 Cr–1 MoVW steels were presented previously (4,5). The behavior of these steels is similar to the normalized-and-tempered 2¼ Cr–1 Mo steel shown in Fig. 2. The normalized-and-tempered 2¼ Cr–1 Mo steel is considerably stronger than the isothermally annealed steel, both before and after irradiation. The strength difference in the unirradiated condition is a reflection of the different microstructures: tempered bainite is considerably stronger than the highly ductile polygonal ferrite.

Irradiation hardened the steel in both heat-treated conditions and resulted in a substantial decrease in ductility. After irradiation there was little difference in the ductility for the two microstructures tested at 25°C. The low uniform elongation in the room-temperature tests is similar to the observations on the 12 Cr–1 MoVW and 9 Cr–1 MoVNb steels (4). At the 300°C test temperature, however, the isothermally annealed steel retains much more ductility than does the normalized-and-tempered steel.

When the properties of all four steels are compared, the yield strength of the unirradiated CW 316 is greater than that of the other four types of materials (Fig. 4); the ultimate tensile strength of the unirradiated CW 316 exceeds all but that for the 12 Cr–1 MoVW steel (Fig. 5). Irradiation produces a much larger increase in strength in the ferritic steels than in the CW 316. The highest strengths were observed for the normalized-and-tempered 2¼ Cr–1 Mo steel and the 12 Cr–1 MoVW. The yield strength and ultimate tensile strength of the irradiated 9 Cr–1 MoVNb are similar to those of the CW 316, while the values for the irradiated, isothermally annealed 2¼ Cr–1 Mo steel are considerably below the respective values for the stainless steel.

To compare the relative hardening effects, the ratio of irradiated to unirradiated yield strength (R_Y) and ultimate tensile strength (R_U) was calculated — namely,

$$R_Y = Y_i/Y_u \quad \text{and} \quad R_U = U_i/U_u ,$$

where Y_i and Y_u are the irradiated and unirradiated yield strengths and U_i and U_u the irradiated and unirradiated ultimate tensile strengths (Table III). Although it is risky to draw definitive conclusions from single values for each alloy, the data at both

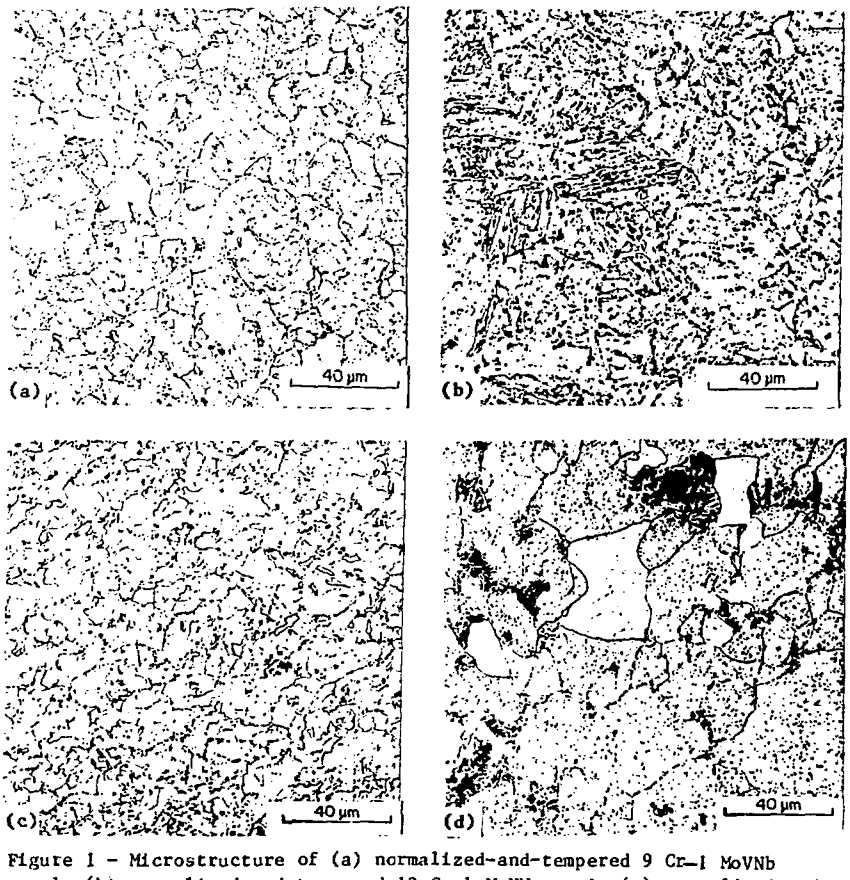

Figure 1 - Microstructure of (a) normalized-and-tempered 9 Cr–1 MoVNb steel, (b) normalized-and-tempered 12 Cr–1 MoVW steel, (c) normalized-and-tempered 2¼ Cr–1 Mo steel, and (d) isothermally annealed 2¼ Cr–1 Mo steel.

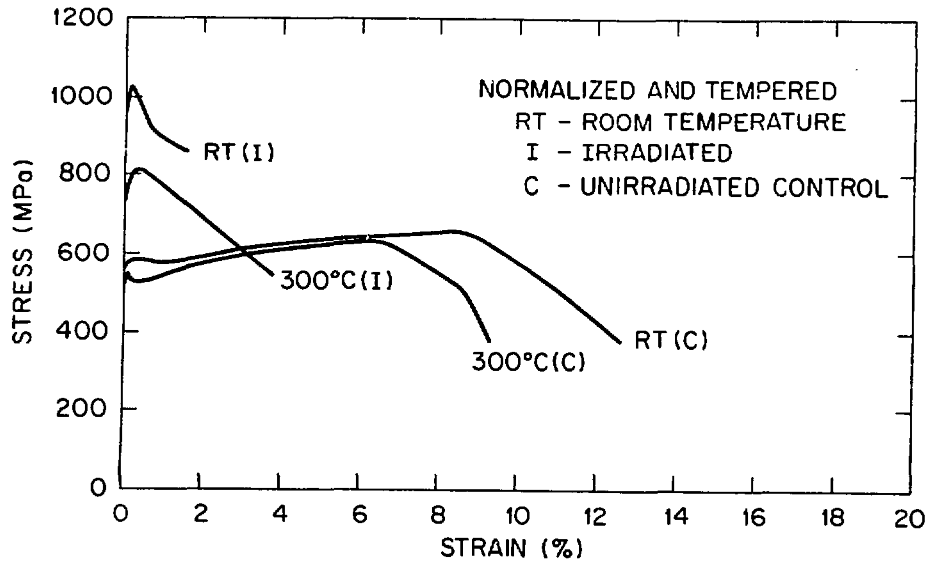

Figure 2 - Engineering stress-strain curves at room temperature and 300°C for normalized-and-tempered 2¼ Cr–1 Mo steel unirradiated and after HFIR irradiation at about 50°C.

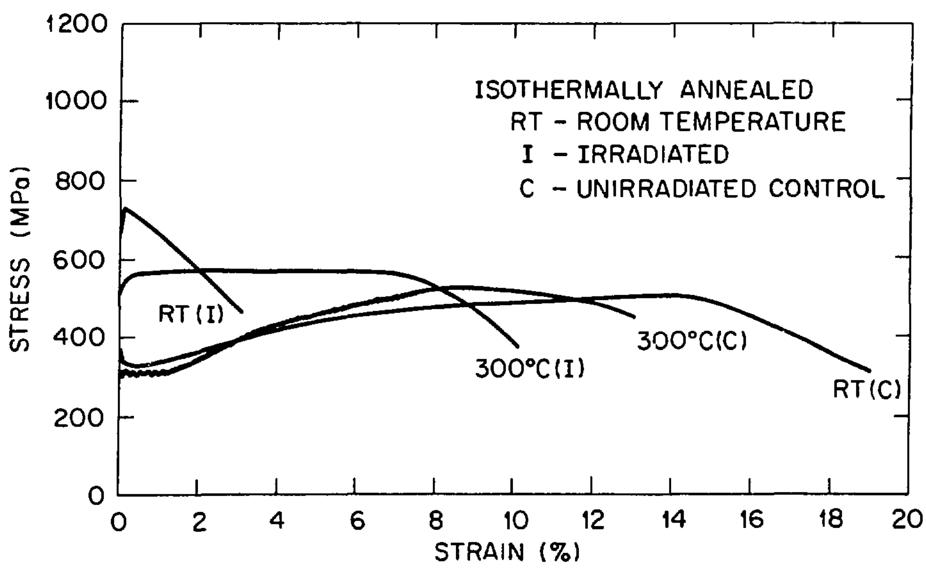

Figure 3 - Engineering stress-strain curves at room temperature and 300°C for isothermally annealed 2¼ Cr–1 Mo steel unirradiated and after HFIR irradiation at about 50°C.

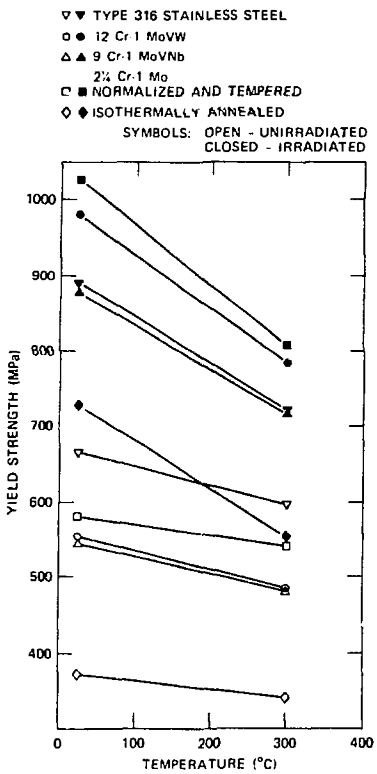

Figure 4 - The 0.2% yield strength plotted against test temperature for 20%-cold-worked type 316 stainless steel, 12 Cr-1 MoVW, 9 Cr-1 MoVNb and 2¼ Cr-1 Mo steel unirradiated and after HFIR irradiation at about 50°C.

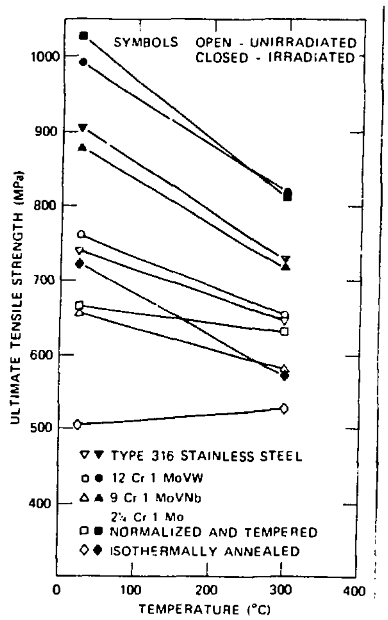

Figure 5 - The ultimate tensile strength plotted against test temperature for 20%-cold-worked type 316 stainless steel, 12 Cr-1 MoVW, 9 Cr-1 MoVNb and 2¼ Cr-1 Mo steel unirradiated and after HFIR irradiation at about 50°C.

Table III. Relative Hardening of Steels

Alloy	Relative Hardening at Each Test Temperature[a]			
	Room		300°C	
	R_Y	R_U	R_Y	R_U
CW 316	1.3	1.2	1.2	1.1
9 Cr–1 MoVNb	1.6	1.3	1.5	1.2
12 Cr–1 MoVW	1.8	1.3	1.6	1.3
2 1/4 Cr–1 Mo (normalized and tempered)	1.8	1.6	1.6	1.3
2 1/4 Cr–1 Mo (isothermally annealed)	2.1	1.5	1.6	1.1

[a]R_Y = ratio of irradiated to unirradiated yield strength.
[b]R_U = ratio of irradiated to unirradiated ultimate yield strength.

room temperature and 300°C show the relative increase in yield strength (R_Y) due to irradiation is greater for the ferritic steels than for the cold-worked stainless steel. Although the isothermally annealed 2¼ Cr–1 Mo steel has the lowest yield strength, it shows the greatest relative increase at room temperature. There are only minor differences in most of the R_U values; the only exceptions are the room-temperature values for the 2¼ Cr–1 Mo, which are larger than those for the high-chromium ferritics and the CW 316. Note the slightly smaller R_U for the isothermally annealed 2¼ Cr–1 Mo steel than for the other ferritic steels at 300°C.

In general. there is little difference in ductility between the irradiated CW 316 and the ferritic steels (Fig. 6) (the only exception is the isothermally annealed 2¼ Cr–1 Mo steel, which will be discussed below). We previously pointed out the low uniform elongation for the high-chromium ferritic steels after irradiation and concluded that this was the result of the irradiation and test conditions and similar behavior will be observed for most alloys irradiated and tested under these conditions (4,5). In agreement with that conclusion, similar, quite low uniform elongation values are noted for the CW 316 (Fig. 6). However, there is a difference in stress-strain curve shape for the CW 316 and the ferritic steels, as seen by comparing the engineering stress-strain curves for the CW 316 (Fig. 7) and the curves for 2¼ Cr–1 Mo steel (Figs. 2 and 3). Once the early uniform elongation is reached, the decrease in strength is much more gradual in the case of the CW 316 than the normalized-and-tempered 2¼ Cr–1 Mo steel at room temperature and 300°C and the isothermally annealed steel at room temperature. The stress-strain curves for the 9 Cr–1 MoVNb and 12 Cr–1 MoVW steels after irradiation were similar to those for the normalized-and-tempered 2¼ Cr–1 Mo steel (Fig. 2). At 300°C, the total elongations are similar for all the steels but the isothermally annealed 2¼ Cr–1 Mo steel.

Stress-strain curves of the type observed for the irradiated ferritic steels [i.e., low uniform elongation followed by a rapid decrease in stress beyond the ultimate tensile strength, which is indicative of a reduced strain-hardening coefficient (6)] have been found in the bcc refractory metals molybdenum, tantalum, niobium, and their alloys (7). The decreased

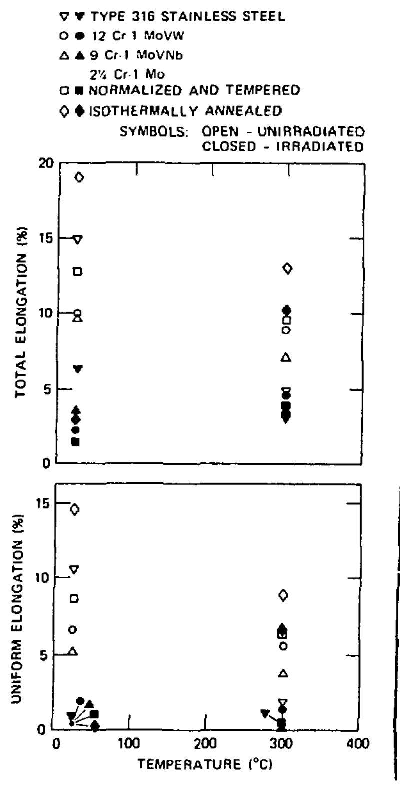

Figure 6 - The uniform and total elongation plotted against test temperature for 20%-cold-worked type 316 stainless steel, 12 Cr-1 MoVW, 9 Cr-1 MoVNb and 2¼ Cr-1 Mo steel unirradiated and after HFIR irradiation at about 50°C.

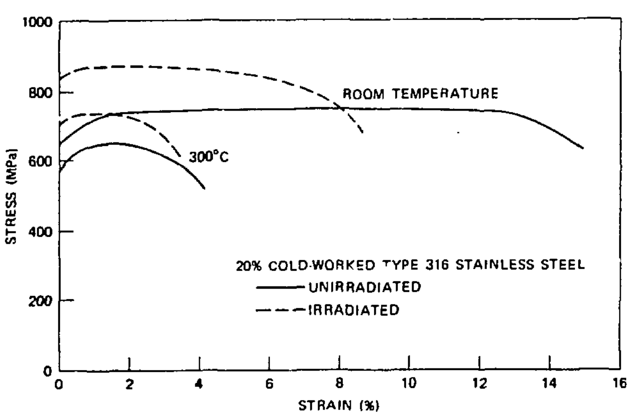

Figure 7 - Engineering stress-strain curves at room temperature and 300°C for unirradiated and irradiated 20%-cold-worked type 316 stainless steel. Irradiation was in HFIR at about 50°C.

uniform elongation has been attributed to a highly localized form of deformation termed channel deformation, because of its microscopic appearance (dislocation free channels are observed by transmission electron microscropy). Fish et al. (8) observed channel deformation in the austenitic stainless steel type 304 after it was irradiated at 400°C to very high neutron fluences in EBR-II (up to 10.7×10^{26} neutrons/m^2, $E > 0.1$ MeV). For 370°C tensile tests, scanning electron microscopy examination of the fracture surfaces revealed that ductile-type failure occurred after exposure to a low fluence, but as the fluence increased, slip was progressively confined to very narrow bands of planes. At the highest fluences, it was found that "shear on the active slip planes has become so extensive that the crystals have literally slipped apart" (8).

Smidt concluded that the ductility loss could be rationalized because channeling causes deformation to be highly localized on a very few slip planes (6). The ductility on the few active slip planes was expected to be high, but uniform elongation and reduction of area were expected to be quite small. Wiffen found that although the refractory metal alloys had stress-strain curves indicative of "work softening," and therefore channeling, there were nevertheless large reductions of area (7). The observations on the ferritic steels are in agreement with Wiffen's observations.

Figure 8 shows SEM photomicrographs of the fracture surfaces of the unirradiated and irradiated 12 Cr-1 MoVW steel tested at room temperature. A highly ductile fracture occurred in the irradiated steel that displayed the curve indicative of channel deformation. In fact, a much more highly dimpled surface is present on the irradiated specimen than the unirradiated one, which was more ductile. When the fracture surfaces of the irradiated normalized-and-tempered and isothermally annealed 2¼ Cr-1 Mo steel tested at 300°C are compared (Fig. 9), both are found to have dimpled surfaces. This, despite the fact that the total elongation of the isothermally annealed steel is almost three times that of the normalized-and-tempered steel and the uniform elongation is over 16 times as great.

On a microscopic scale, channel deformation is observed to occur by dislocations sweeping out defect-free channels (6), after which dislocation motion is confined to these channels. Once these microscopic channels form, further deformation is confined to the region of these channels. The present observations and those of Wiffen (7) indicate that although deformation is restricted to a few slip planes, quite large amounts of deformation must be possible in that localized region. It would appear that the planer, less-ductile features that Fish et al. observed and associated with channeling (8) will only be observed after higher irradiation doses. This might also mean that the low uniform elongation and the gradual decrease in strength beyond the ultimate tensile strength for the CW 316 (Fig. 7) is an indication of the early stages of channel deformation.

Wiffen and Maziasz (9) irradiated CW 316 in HFIR at 50°C to somewhat similar dpa values to those achieved in the present experiment. Tensile tests over the range 35 to 600°C produced results that were in general agreement with those reported here. Very low uniform elongations were found between 35 and 300°C; it decreased from 0.6% at 35°C to 0.2% at 300°C. From 300 to 600°C, the uniform elongation increased from 0.2 to 5.3%.

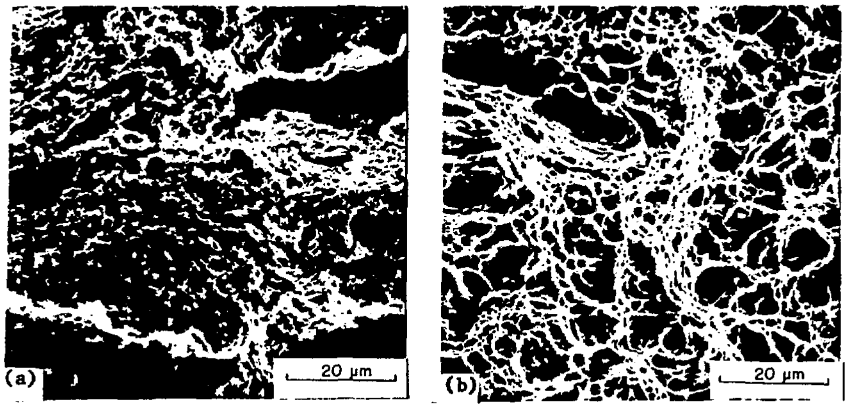

Figure 8 - Fracture surface of normalized-and-tempered 12 Cr—1 MoVW steel tensile tested at room temperature. (a) Unirradiated; (b) irradiated.

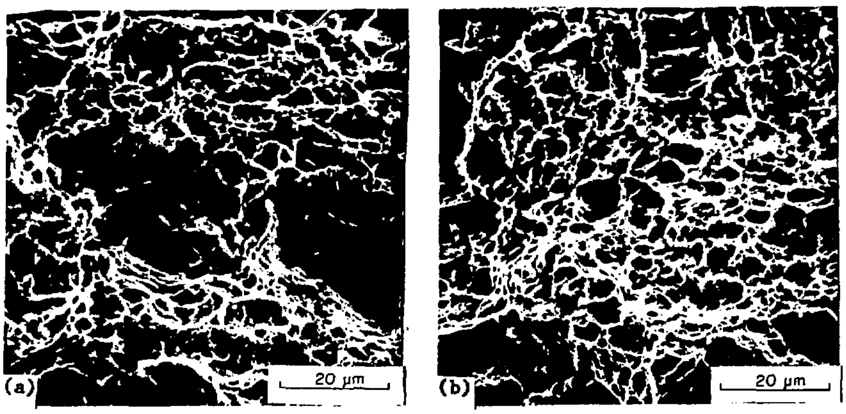

Figure 9 - Fracture surface of irradiated 2 1/4 Cr—1 Mo steel tensile tested at 300°C. (a) Normalized and tempered; (b) isothermally annealed.

There is a significant improvement in ductility at 300°C for the isothermally annealed 2 1/4 Cr—1 Mo steel over that of the other steels tested (Fig. 6). The irradiated isothermally annealed 2 1/4 Cr—1 Mo steel had uniform and total elongations of 6.7 and 10.1%, respectively, compared with 1.8 and 5.1% for 12 Cr—1 MoVW steel and less than 1 and less than 4% for 9 Cr—1 MoVNb steel, normalized-and-tempered 2 1/4 Cr—1 Mo steel, and CW 316 (Table II).

The isothermally annealed 2 1/4 Cr—1 Mo steel showed the smallest relative decrease in total elongation. However, the change in uniform elongation was most significant. At room temperature the uniform elongation of all the materials, including the isothermally annealed steel, was less than 1%. All but the 12 Cr—1 MoVW have uniform elongations of less than 1% at 300°C; the uniform elongation of the 12 Cr—1 MoVW was 1.8%. These low uniform elongations compare unfavorably with the high value of the isothermally annealed 2 1/4 Cr—1 Mo steel, which was 6.7%. This difference may reflect the fact that the unirradiated steel had a higher ductility or it may be an important clue to the effect of microstructure on the irradiation behavior of these materials. The isothermally annealed 2 1/4 Cr—1 Mo steel was primarily polygonal ferrite, while the other ferritic steels were either tempered bainite or tempered martensite. Another observation that may involve the microstructure and ductility of the 2 1/4 Cr—1 Mo steel is the channel deformation discussed above. The stress-strain curve for the isothermally annealed steel is indicative of channel deformation at room temperature, but not at 300°C (Fig. 3). More information will be required before these observations can be fully understood.

SUMMARY AND CONCLUSIONS

Tensile properties were determined at room temperature and 300°C on 9 Cr 1 MoVNb, 12 Cr—1 MoVW, 2 1/4 Cr—1 Mo steel and 20%-cold-worked type 316 stainless steel irradiated in HFIR at ~50°C to ~1.3 × 10^{26} neutrons/m^2, resulting in displacement damage levels up to about 9 dpa. All three ferritic steels were irradiated and tested in the normalized-and-tempered condition. The microstructures after this heat treatment were essentially 100% martensite for the 9 Cr—1 MoVNb and 12 Cr—1 MoVW steels and 100% tempered bainite for the 2 1/4 Cr—1 Mo steel. Specimens of the 2 1/4 Cr—1 Mo steel were also irradiated and tested in an isothermally annealed condition, where the microstructure was 75—80% polygonal ferrite with the balance bainite.

For all the alloys, irradiation caused a significant increase in yield strength and ultimate tensile strength at room temperature and 300°C. The increase in strength was accompanied by a decrease in ductility. The decrease for the three ferritic steels was greatest at room temperature: uniform elongations decreased from a range of 5.1–14.6% to 0.1–4%; total elongation decreased from 9.6–19.0% to 3.2–7.7%. These changes are comparable with the changes on type 316 stainless steel where the uniform elongation decreased from 11.1 to 0.8% and the total elongation from 14.9 to 7.7%. For tests at 300°C, the ductility of the three ferritic steels after irradiation was greater than the irradiated ductility at room temperature. Of special significance was the isothermally annealed 2¼ Cr–1 Mo steel tested at 300°C, which had uniform and total elongations of 6.7 and 10.1%, respectively. This compared with uniform and total elongation values of less than 1 and 4%, respectively, for the 9 Cr–1 MoVNb steel, normalized-and-tempered 2¼ Cr–1 Mo steel, and 20%-cold-worked type 316 stainless steel. The 12 Cr–1 MoVW steel had a uniform elongation of 1.8% and a total elongation of 5.1%.

Under the conditions of the present study, the results indicate that the normalized-and-tempered 2¼ Cr–1 Mo steel has irradiated tensile properties similar to the 9 Cr–1 MoVNb and 12 Cr–1 MoVW steels and 20%-cold-worked type 316 stainless steel. The results also indicate that the irradiation hardening of the 2¼ Cr–1 Mo steel depends on the microstructure, or starting strength, indicating that microstructural variation may offer the possibility for improving the irradiated properties of the ferritic steels.

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