THE HIGH TEMPERATURE TENSILE PROPERTIES OF FERRITIC-MARTENSITIC AND AUSTENITIC STEELS AFTER IRRADIATION IN AN 800 MEV PROTON BEAM

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Abstract

This study examines the effect of tensile test temperatures ranging from 50°C to 600°C on the tensile properties of a modified 9Cr-1Mo ferritic steel (T91) and a 316L stainless steel (SS) after high energy proton irradiation at about 35°C-67°C. After 1-3 dpa, the proton-irradiated T91 steel was tensile tested at temperatures from 50°C to 600°C. It was observed that the yield strength and ultimate strength decreased monotonically as a function of tensile test temperature, whereas the uniform elongation remained at approximately 1% for tensile test temperatures up to 250°C and then increased for tensile test temperatures up to and including 500°C. At 600°C, the uniform elongation was observed to be less than the values at 400°C and 500°C. Uniform elongation of the irradiated material tensile tested at 400°C to 600°C was observed to be greater than the values for the unirradiated material at the same temperatures. No changes in microstructure were observed in T91 transmission electron microscopy specimens heated up to 500°C prior to observation. Tensile tests on the 9 dpa specimens of T91 followed similar trends. The 316L SS was irradiated to doses up to 10 dpa and tested at 300°C. Tensile tests done in conjunction with shear punch tests suggest that the tensile properties change substantially within the first 2 dpa, but further changes occur when irradiated to 10 dpa. Most notably, the uniform elongation after 10 dpa is estimated from shear punch data to drop to near zero at a mechanical test temperature of 300°C.

Introduction

Data in the literature on the effects of proton irradiation on the tensile properties of ferritic-martensitic steels and austenitic steels have been steadily growing over the last five years. [1-6] The majority of these proton irradiations have been conducted at temperatures below 300°C. It is now expected that some accelerator components to be used the Accelerator Transmutation of Waste (ATW) programme will operate at temperatures ranging from 300°C to 600°C while being exposed to a high energy (~1 GeV) proton beam. Because little data is available on materials irradiated in such an environment, the tensile properties were determined at temperatures ranging from 50°C to 600°C on a ferritic-martensitic steel (T91) and an austenitic stainless steel (316L SS) using specimens were proton-irradiated at temperatures between 35°C and 67°C at the Los Alamos Neutron Science Center (LANSCE) as part of the Accelerator Production of Tritium (APT) materials irradiation programme. The possible effects of irradiating at a temperature lower than the tensile test temperature were evaluated by analysing studies in the literature where irradiation and tensile tests were performed at the same temperature. [7,8]

Experiment

Materials and specimens

The composition of the T91 steel is shown in Table 1. Transmission electron microscopy (TEM) disks and S-1 tensile specimens (5 mm gage length and 1.2 mm gage width) were electro-spark machined (EDM) from the 0.25 mm thick sheet stock. Then, the T91 specimens were normalised at 1 038°C for 1 hour, air-cooled, and then tempered at 760°C for 1 hour resulting in a tempered martensite (i.e. ferrite) crystal structure containing dislocations and carbides. Two heats of 316L SS were used. One was used to make 0.75 mm thick sheet stock for the tensile specimens while the other was used to make 0.25 mm thick sheet stock for the TEM disks. The compositions are shown in Table 1. Prior to specimen fabrication, the 316L SS sheet stock was annealed at 1 050°C in a vacuum and then air-cooled. TEM disks and S-1 tensile specimens were then EDM fabricated from the sheet stock.

Table 1. **Composition of the modified 9Cr-1Mo steel and the two heats of 316L SS**

Modified 9Cr-1Mo (Lot Num. 10148) Composition in Weight Percent									
Fe	Cr	Ni	Mo	Mn	C	Si	P	S	Cu
Bal	9.24	0.16	0.96	0.47	0.089	0.28	0.030	0.006	0.08
Al	V	Nb	Co	N	O	Ti			
0.002	0.21	0.054	0.019	0.035	0.008	0.002			
316L SS (Lot Num. L406) Composition in Weight Percent (for tensile)									
Fe	Cr	Ni	Mo	Mn	C	Si	P	S	Cu
Bal.	17.33	10.62	2.09	1.61	0.022	0.43	0.024	0.019	0.18
316L SS (Lot Num. E385) Composition in Weight Percent (for TEM)									
Fe	Cr	Ni	Mo	Mn	C	Si	P	S	Cu
Bal.	17.26	12.16	2.57	1.75	0.019	0.65	0.022	0.006	0.26

Irradiation conditions

Irradiations were conducted in the LANSCE facility as part of the APT materials irradiation programme. The LANSCE accelerator generates an 800 MeV, 1 mA Gaussian proton beam where 2σ equals 3 cm, which impinges on the experimental assembly. Specimens were irradiated for six months. Dose for each specimen was determined from analysis of pure metal activation foils placed next to specimens during irradiation. [9] Further details on dose estimation can be found elsewhere. [10,11] Irradiation temperatures which were passively controlled, varied from 35°C to 67°C for the specimens in the present study. Details of the temperature measurement can be found in Reference 12. Doses, irradiation temperatures, and estimated helium & hydrogen content of the T91 steel specimens have been previously reported in Reference 13. Doses, irradiation temperatures, and estimated helium & hydrogen content for the 316L SS specimens are in Tables 2 and 3.

Table 2. **Estimated helium content, estimated hydrogen content, and tensile properties of the control and irradiated 316L SS tensile specimens**

ID	dose (dpa)	T_{irr} (°C)	Est. He (appm)	Est. H (appm)	T_{test} (°C)	YS (MPa)	UTS (MPa)	UE (%)	TE (%)
316-9*	–	–	–	–	50°	255	598	51.4	76.3
316-10*	–	–	–	–	50°	259	593	51.3	72.6
316-14*	–	–	–	–	300°	204	456	34.9	50.9
316-15*	–	–	–	–	300°	220	458	29.9	44.6
24-6-7	2.7	65	160	1 370	50°	711	787	23.9	38.0
24-6-8	2.9	65	170	1 470	50°	699	791	25.9	46.8
24-6-3	2.5	52	150	1 300	300°	552	595	12.8	25.8
24-6-4	2.7	52	160	1 400	300°	571	591	12.1	25.5

* Unirradiated control specimens

Table 3. **Estimated helium content, estimated hydrogen content, shear punch properties, and estimated tensile properties of the control and irradiated 316L SS TEM specimens**

ID	dose (dpa)	T_{irr} (°C)	Est. He (appm)	Est. H (appm)	T_{test} (°C)	Shr. YS (MPa)	Shr. UTS (MPa)	Pred. YS (MPa)	Pred. UTS (MPa)	Pred. UE (%)
316-1SP*	–	–	–	–	300°	120	378	234	559	48
316-2SP*	–	–	–	–	300°	105	378	205	559	56
316-3SP*	–	–	–	–	300°	105	374	205	554	55
316-4SP*	–	–	–	–	300°	110	374	214	554	53
4-2-4	1.5	40	100	870	300°	270	415	527	614	9.8
4-2-25	1.5	40	100	870	300°	285	435	556	644	9.4
4-2-8	9.7	67	700	5 960	300°	380	488	741	722	0.9
4-2-29	9.7	67	700	5 960	300°	390	498	761	737	0.6

* Unirradiated control specimens

Test method

The tensile test method has been previously described in Reference 13. For the specimens tensile tested at 400°C (only the unirradiated specimens), 500°C, and 600°C, the length of time that specimens were above 90% of the test temperature (in Kelvin) prior to the onset of tensile testing was about 2 hours. All other specimens tested at temperatures between 50°C and 300°C were above 90% of the test temperature for about 1-1.5 hours prior to the start of a tensile test. From the tensile traces, 0.2% offset yield strength (YS), ultimate tensile strength (UTS), engineering uniform elongation (UE), and total elongation (TE) were measured.

Shear punch tests were performed on TEM disks using a special fixture in a screw-driven Instron test frame. [14] The fixture used in these tests has a 1.00 mm diameter punch and a 1.04 mm diameter receiving die which results in a clearance of about 25 µm. The punch length was about 18 mm. The crosshead speed was set to 0.13 mm/s. Displacement was measured at the crosshead. The yield load was taken as the point of deviation from linear loading on a load versus displacement trace, and the ultimate load was taken at the peak load on a trace. For the purposes of calculating a stress, an idealised shear deformation condition is assumed to exist, and from this assumption, a shear stress can be calculated from $\tau = P/2\pi rt$, where "P" is the load on the punch, "r" is the average of the punch and receiving die radii, and "t" is the thickness of the TEM disk. A specimen was held at 90% to 100% of the test temperature (in Kelvin) for about 1 hour prior to starting a shear punch test.

Tensile properties were estimated from the shear yield strength and shear ultimate strength using previously published data from shear punch tests and tensile tests on unirradiated materials. [14,15] The yield strength correlation and the ultimate strength correlation were determined by combining the data in ures 8 and 9 of Reference 14, and fitting a straight line through the origin. The resulting correlations are $\sigma_y = 1.95\tau_y$ and $\sigma_m = 1.48\tau_m$. The uniform elongation correlation is the one reported in Reference 15. Note that it is a correlation to predict true uniform elongation. The correlation is $\varepsilon_u = 2.26n_\tau - 0.15$, where n_τ is found from the ratio of shear ultimate strength to shear yield strength. For purposes of comparison to the tensile UE data, these predicted true uniform elongation values were converted to engineering uniform elongation values.

Results and discussion

T91 Steel

The tabulated tensile properties and tensile traces of the T91 steel can be found in Reference 13. Examination of the unirradiated material tensile property trends as a function of temperature in Figure 1 show that YS, UTS, and UE all decrease with increasing tensile test temperature which is typical for similar materials. [8,16] TE decreases until about 400°C and then increases with increasing tensile test temperature.

Figure 1. **Tensile properties of unirradiated Mod 9Cr-1Mo as a function of tensile test temperature**

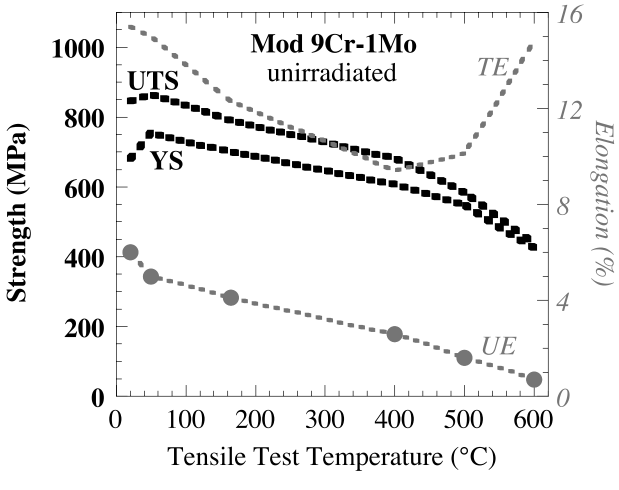
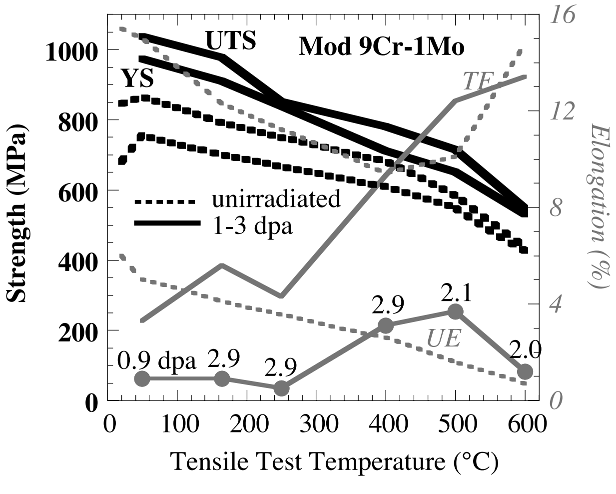


Figure 2. **Tensile properties of Mod 9Cr-1Mo plotted as a function of tensile test temperature for unirradiated material and material irradiated to doses from 1-3 dpa** (Irradiation temperature varied from 34-46°C)



Shown in Figure 2 are the tensile properties of the 1-3 dpa specimens as a function of tensile test temperature. The YS and UTS of the irradiated material show the same general temperature dependence as the unirradiated material, but the values for the irradiated material are greater than that of the unirradiated material with the difference between the irradiated values and unirradiated values decreasing as the tensile test temperature increases. The difference in YS between irradiated and unirradiated (ΔYS) is about 250 MPa at 50°C and decreases to about 100 MPa at 600°C. Within this range of temperatures, the hold times are too short to significantly affect the pre-irradiation microstructure, so the most likely cause for this decrease in ΔYS is annealing of the radiation-induced defects that were produced at the low irradiation temperature. Transmission electron microscopy observations of the irradiated specimens annealed for one hour at 500°C showed no obvious changes in the microstructure compared to unannealed irradiated specimens as illustrated in Figure 3. In Figure 4, the microstructure is compared to the microstructure of a 9 dpa specimen with no post-irradiation anneal. It can be seen that the microstructures are qualitatively similar. Based on this observation, it is likely that irradiation-induced defects not visible by transmission electron microscopy are coarsening and probably dissolving or annihilating. The trends and the magnitude of the UE and the TE for the irradiated material are much different than for the unirradiated material. The UE for the irradiated material hovers at about 1% or less for the tests performed at 50°C, 164°C, and 250°C, and then increases beyond the values for the unirradiated material to a peak at 500°C which is followed by a decline at 600°C to a value approximately equal to that of the unirradiated material. TE of the irradiated material starts out well below that of the unirradiated material, but increases with tensile test temperature, eventually surpassing the values for the unirradiated material at 500°C and then declining at 600°C to a value slightly below that of the unirradiated material. For this material, it is thought the increase in yield strength is so great that it is impossible for the material to become any stronger by work-hardening, and thus the ultimate strength is only slightly greater than the yield strength, and the material quickly begins to neck. [13] This type of temperature dependence where the elongation of the irradiated material surpasses the elongation of the unirradiated material has also been observed in a 12Cr ferritic-martensitic steel (DIN 1.4914) that was neutron irradiated to 1 dpa at less than 100°C. [17] It has also been observed in ferritic-martensitic steels that have been neutron-irradiated to relatively high doses (10 dpa to 59 dpa) at elevated temperature. [18-20] It is thought to be due to a synergistic interaction between the remnant irradiation-induced defects and the behaviour of dislocations in this material at temperatures between 400°C and 600°C. [13] At temperatures below 300°C, the high yield strength and low uniform elongation observed in the irradiated materials is thought to be due to the irradiation-induced formation of a fine dispersion of small, shearable defects. [3,7,21] The tensile properties of the T91 specimens irradiated to 9 dpa are shown in Figure 5. The tensile traces and tabulated data can be found in Reference 13. Compared to the 1-3 dpa tensile properties, at 9 dpa, the YS and UTS are increased while the UE and TE are decreased, but the general trends appear to be similar to the 1-3 dpa specimens.

The tensile properties as a function of dose are shown in Figure 6 for tensile test temperatures of 164°C and 500°C. The tensile properties at 164°C follow the usual trend where the YS and UTS increase with increasing dose while the UE and TE are strongly reduced by irradiation. At 500°C, the YS and UTS increase with increasing dose. However, the UE and TE initially increase at about 2 dpa and then decline by 9 dpa indicating that the UE and TE may reach a maximum as a function of dose for these irradiation conditions and tensile test conditions.

Figure 3. **Microstructure in underfocus of T91 that was proton-irradiated at 35-67°C to 1.4 dpa and then given a one hour post-irradiation anneal at 500°C. There is no evidence of bubbles in this through-focus series of images**

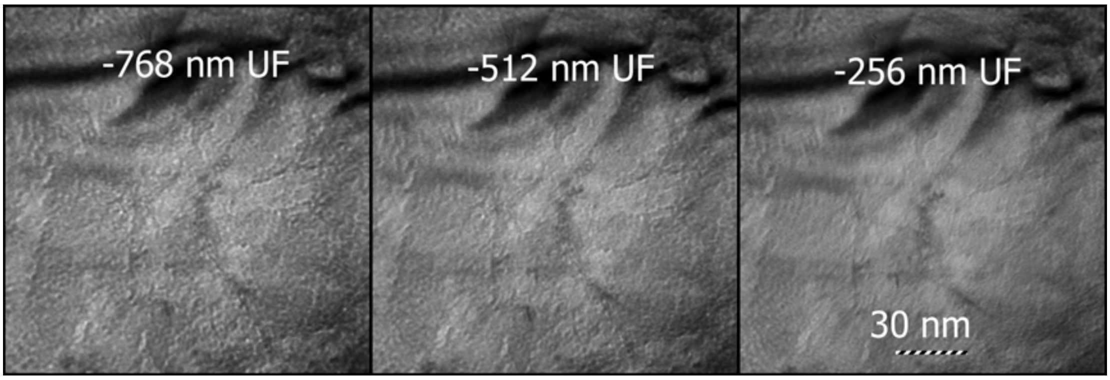


Figure 4. Microstructure of T91 after 1.4 dpa with a post-irradiation anneal compared to the microstructure of T91 after 9 dpa with no post-irradiation anneal. Microstructures are similar in appearance with neither showing any evidence of resolvable bubbles

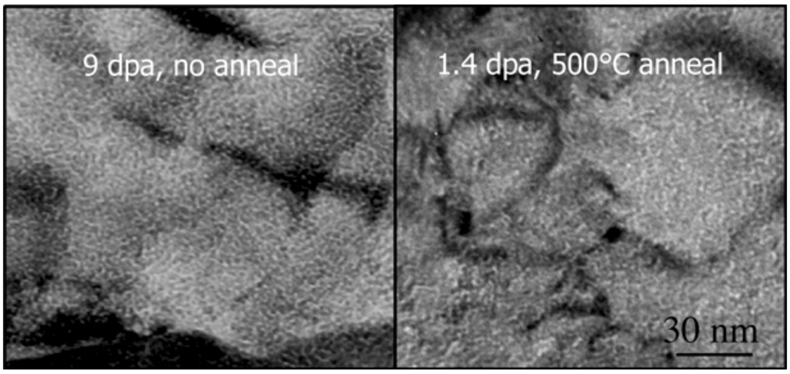
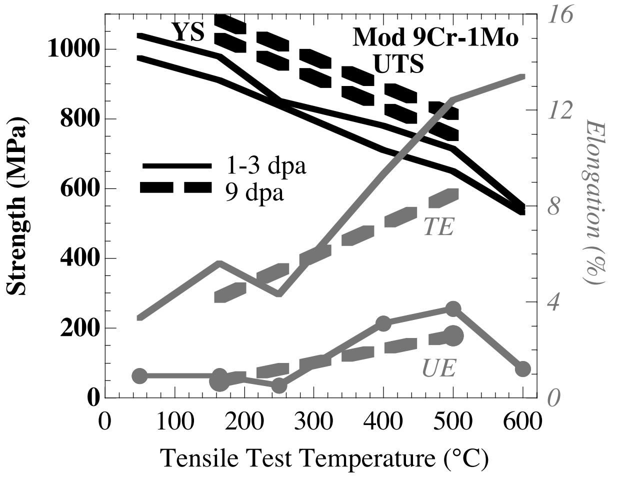
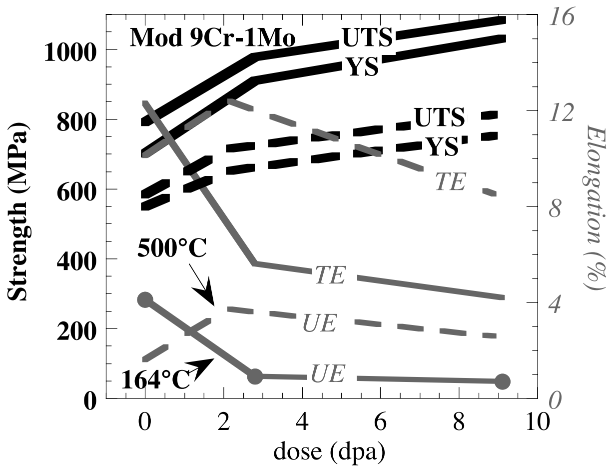


Figure 5. **Tensile properties of Mod 9Cr-1Mo plotted as a function of tensile test temperature for material irradiated to 1-3 dpa or about 9 dpa.**



Irradiation temperature of 9 dpa material was between 65 and 67°C

Figure 6. **Tensile properties of T91 as a function of dose at 164°C and at 500°C**



As there is little data on the tensile properties of ferritic-martensitic steels that have been irradiated in spallation environment at an elevated temperature, one of the objectives of the present study was to evaluate ferritic-martensitic steels for use in a spallation radiation environment at elevated temperature using materials that had been irradiated in a spallation environment at lower temperatures. The irradiation-induced defects produced in ferritic-martensitic steels at 35-67°C are considerably higher in density and smaller in size than those which are produced at higher temperatures, and thus it is possible that materials irradiated at 35-67°C and then tested at higher temperatures would not have the same tensile properties as materials both irradiated and tensile tested at elevated temperature. To assess the limitations of the present experiment for evaluating material performance at elevated temperatures, trends in the literature on tensile tests of ferritic-martensitic steels that were neutron-irradiated and tensile tested at the same temperature were examined. These literature trends are as follows: relative to unirradiated materials, irradiation and tensile testing of ferritic-martensitic steels in the temperature range from 30°C to 350°C results in large increases in yield and ultimate strength, and depending on the irradiation/tensile test temperature, causes moderate to extreme reductions in uniform elongation. [4-8] The extreme reductions in uniform elongation are due to a lack of work-hardening ability that promotes early plastic instability. [3,7,21] For irradiation/tensile test temperatures from 450-600°C (and probably above), the data in the literature show that neutron irradiation has little, if any, effect on the tensile properties of ferritic-martensitic steels, [22] because at these temperatures, significant irradiation damage which can affect tensile properties does not accumulate in ferritic-martensitic steels. These literature trends can be compared to the results from the tests presented here. At tensile test temperatures from 30°C to 250°C, the tensile properties of the Mod 9Cr-1Mo steel presented here largely follow the trends in the literature for ferritic-martensitic steels irradiated and tested at 30°C to 250°C. For temperatures from 500°C to 600°C, the tensile data presented here do not follow the literature trends on neutron-irradiated ferritic-martensitic steels that were irradiated and tensile tested at temperatures from 500°C to 600°C. The YS and UTS reported here for the proton-irradiated material are about 20% greater than the values for the unirradiated material, and the UE and TE of the irradiated material are equal to or higher than that of the unirradiated material. This is in contrast to the trends in the literature where there was often no effect of irradiation on tensile properties of ferritic-martensitic steels that were neutron-irradiated to doses of at least 15 dpa and tensile tested at 500-600°C. The fact that the proton-irradiated material does not have the same tensile properties at 500-600°C as unirradiated materials is likely due to residual irradiation-induced defects present after the post-irradiation two-hour anneal.

Figure 7. **a) Shear punch properties**

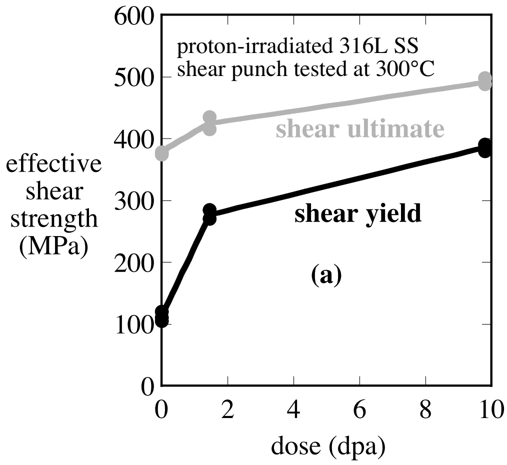
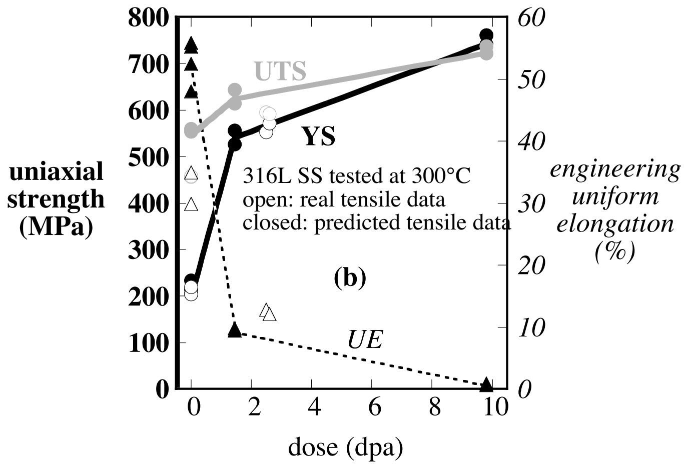


Figure 7. **b) Predicted tensile properties and real tensile properties of 316L SS that was proton-irradiated at 35°C to 67°C and then tensile tested at 300°C**



316L SS

The tensile properties of the unirradiated 316L SS tensile tested at 50°C and 300°C are shown in Table 2. Changing from a 50°C test temperature to a 300°C test temperature causes a 17% drop in YS and a 37% drop in UE.

The tensile properties for the 316L SS tensile specimens irradiated to ~3 dpa are also shown in Table 2. Changing from a 50°C test temperature to a 300°C test temperature causes a 20% drop in YS and a 50% drop in UE. These changes are similar to those observed in the unirradiated material. Thus, it appears that tensile test temperature does have a very large impact on the tensile properties of 316L SS, and this trend is not affected by the microstructure. Reports in the literature on the tensile properties of irradiated 316 stainless steels where irradiations have been conducted at the tensile test temperature have shown that at a temperature of about 330°C, there is a large loss in strain-hardening capacity. [23,24] The data presented here suggest that while the irradiation-induced microstructure plays a large role in the tensile behaviour, it may be that the tensile test temperature itself plays an even larger role in the tensile behaviour, especially the UE, at ~330°C.

The shear yield strength, shear ultimate strength, and the estimated tensile properties obtained from the shear punch tests are shown in Table 3 for the shear punch tests which were performed at 300°C. The shear punch properties are plotted as a function of dose in Figure 7a. The data suggest that about 2/3 of the observed total hardening occurs within the first 2 dpa. The estimated tensile properties along with the measured tensile properties at 300°C are plotted as a function of dose in Figure 7b. The tensile properties predicted from the shear punch tests at 300°C on the unirradiated material and from the material tested at around 2 dpa are in good agreement with the actual tensile properties measured at 300°C. From the shear punch tests, it appears that after 10 dpa, the yield strength and ultimate strength will converge and the uniform elongation will drop to nearly zero. Together, these are strong indicators that after 10 dpa, flow localisation and loss of strain-hardening capacity occurs in this material at 300°C. Thus, it appears that even microstructures formed during irradiation at temperatures below 100°C can cause flow localisation and loss of strain-hardening capability when the material is tested near 330°C.

Summary and conclusions

T91 Steel

Tensile tests were performed at temperatures ranging from 20°C to 600°C on unirradiated Mod 9Cr-1Mo and Mod 9Cr-1Mo irradiated in a spallation environment at temperatures ranging from 35°C to 67°C. The effect of tensile test temperature on the unirradiated material was to decrease the yield strength, ultimate strength, and uniform elongation as test temperature increased. The effect of increasing tensile test temperature on irradiated material relative to unirradiated material was to increase the yield strength and ultimate strength over the unirradiated material throughout the temperature range. Uniform elongation of the irradiated material dropped to values of 1% or less at tensile test temperatures from 50°C to 250°C whereas between 400°C and 500°C, the uniform elongation was observed to increase steadily and surpass the observed uniform elongation of the unirradiated material at 500°C. At 600°C, the uniform elongation of the irradiated material had decreased to match that of the unirradiated material. It is thought that the improved elongation of the Mod 9Cr-1Mo at 500°C is due to a synergistic interaction between the remnant irradiation-induced defects and the behaviour of dislocations in this material at 500°C.

For the Mod 9Cr-1Mo that was proton-irradiated at 37-67°C and tensile tested at temperatures ranging from 50°C to 250°C, the tensile properties as a function of tensile test temperature follow the same trends as those reported in the literature on materials that were neutron-irradiated and tensile tested at the same temperature. For the Mod 9Cr-1Mo that was tensile tested at temperatures ranging from 400°C to 600°C, the YS and UTS were greater than the values for the unirradiated Mod 9Cr-1Mo, and the UE and TE were either greater than or equal to the values for the unirradiated Mod 9Cr-1Mo. In this temperature range, for neutron-irradiation experiments where the irradiation temperature and the tensile test temperature were the same, the tensile properties of ferritic-martensitic steels irradiated to doses as high as 15 dpa were generally observed to be approximately equal to that of the unirradiated material. This is due to the radiation resistance of these materials in this temperature range. Thus, it appears that for the present study, the defects introduced by proton-irradiation at 37-67°C are still present in some form after the two hours post-irradiation anneals performed prior to tensile testing.

316L Stainless steel

Tensile tests and shear punch tests were performed at 300°C on 316L SS irradiated up to about 10 dpa at temperatures ranging from 35°C to 67°C. The data suggest that the majority of the strengthening occurs within the first 2 dpa. After 2 dpa the material still has good ductility, but after 10 dpa, flow localisation and loss of strain-hardening capacity are apparent at 300°C. These results suggest that since this material was irradiated at less than 100°C, flow localisation and loss of strain-hardening capacity which have been observed in other 316L stainless steels that were irradiated and tensile tested at temperatures near 300°C, are not purely a product of the microstructure produced at about 300°C but also a product of the test temperature itself.

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