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High temperature tensile testing of modified 9Cr–1Mo after irradiation with high energy protons

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Abstract

This study examines the effect of tensile test temperatures ranging from 50 to 600 °C on the tensile properties of a modified 9Cr–1Mo ferritic steel after high energy proton irradiation at about 35–67 °C to doses from 1 to 3 dpa and 9 dpa. For the specimens irradiated to doses between 1 and 3 dpa, it was observed that the yield strength and ultimate strength decreased monotonically as a function of tensile test temperature, whereas the uniform elongation (UE) 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 UE was observed to be less than the values at 400 and 500 °C. UE of the irradiated material tensile tested at 400–600 °C was observed to be greater than the values for the unirradiated material at the same temperatures. Tensile tests on the 9 dpa specimens followed similar trends.

1. Introduction

Data in the literature on the effects of low temperature irradiation on the tensile properties of ferritic–martensitic steels have been steadily growing over the last five years. These data are from both neutron irradiation experiments [1–5] and high energy proton irradiation experiments [6–11]. In most instances, the data have been generated from tensile tests that were conducted at the irradiation temperature [1–5,7,8]. Conducting the tensile tests at the same temperature as the irradiation temperature has been considered to be a reasonable approach as such data are design relevant. However, in some instances, tensile data in the literature have been generated from tensile tests conducted at room temperature [9,10], or in some instances, the data have been generated from tensile specimens that were irradiated at one temperature but then tensile tested at a variety of temperatures different from the irradiation temperature [1,5,11]. Some possible reasons for these latter approaches may include limitations in irradiation facilities or tensile testing facilities. Specimens for the present study were irradiated at the Los Alamos Neutron Science Center (LANSCE) as part of the accelerator production of tritium (APT) materials irradiation program. In this irradiation program, the irradiation temperature was passively controlled with specimen temperatures for the present study ranging from 35 to 67 °C. Within this limitation, it was necessary to attempt to evaluate the possible tensile behavior of the specimens over a range of temperatures relevant to future in-service accelerator environments. Thus, tensile tests were performed at temperatures ranging from 50 to 600 °C. The possible effects of irradiating at a temperature lower than the tensile test temperature were evaluated by analyzing studies in the literature where irradiations have been performed at a variety of temperatures, and the tensile properties were then evaluated at several different tensile test temperatures [1,5].

2. Experiment

2.1. Material and specimens

The composition of the modified 9Cr–1Mo steel is shown in Table 1. Prior to specimen fabrication, the

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Table 1
Composition of the modified 9Cr–1Mo steel

Modified 9Cr–1Mo (Lot Num. 10148) composition in wt%				
Fe	Cr	Ni	Mo	Mn
Bal	9.24	0.16	0.96	0.47
C	Si	P	S	Cu
0.089	0.28	0.030	0.006	0.08
Ti	Al	V	Nb	Co
0.002	0.002	0.21	0.054	0.019
N	O			
0.035	0.008			

sheet stock was normalized at 1038 °C for 1 h, air cooled, and then tempered at 760 °C for 1 h resulting in a tempered martensite (i.e. ferrite) crystal structure containing dislocations and carbides. S-1 tensile specimens with a 5 mm gage length and 1.2 mm gage width were electro-spark machined from the 0.25 mm thick sheet stock.

2.2. Irradiation conditions

Irradiations were conducted in the LANSCE facility as part of the APT materials irradiation program. 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 [12]. Further details on dose estimation can be found elsewhere [13,14]. Irradiation temperatures which were passively controlled, varied from 35 to 67 °C for the specimens in the present study. Details of the temperature measurement can be found in [15]. Doses and irradiation temperatures are shown in Table 2.

2.3. Test method

Tensile testing was performed in an Instron screw-driven test frame. Specimen gauge dimensions were measured prior to each test. Heat-up was performed with a constant one pound load (15 MPa) maintained on the specimen. For the specimens tensile tested at 400 °C (only the unirradiated specimens), 500, 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 generally about 2 h. All other specimens tested at elevated temperature were above 90% of the

Table 2
Estimated helium content, estimated hydrogen content, and tensile properties of the control and irradiated specimens

ID	Dose (dpa)	T_{irr} (°C)	Est. He (appm)	Est. H. (appm)	T_{test} (°C)	YS (MPa)	UTS (MPa)	UE (%)	TE (%)
9Cr-1[a]	–	–	–	–	20	682	864	6.0	17.1
9Cr-2[a]	–	–	–	–	20	677	828	5.9	13.7
9Cr-3[a]	–	–	–	–	50	760	867	5.4	14.9
9Cr-4[a]	–	–	–	–	50	748	858	4.5	15.0
9Cr-5[a]	–	–	–	–	164	718	797	3.5	12.3
9Cr-6[a]	–	–	–	–	164	681	782	4.7	12.3
9Cr-9[a]	–	–	–	–	400	613	692	2.6	9.9
9Cr-10[a]	–	–	–	–	400	603	671	2.5	8.8
9Cr-11[a]	–	–	–	–	500	555	598	1.9	10.3
9Cr-12[a]	–	–	–	–	500	540	568	1.3	9.9
9Cr-13[a]	–	–	–	–	600	426	438	0.7	14.8
4-3-11	0.9	38	40	320	50	951	1030	0.9	1.4
4-3-12	0.9	38	40	320	50	995	1044	0.9	5.2
23-5-7	2.9	35	180	1590	164	927	946	0.7	5.8
4-3-3	2.8	46	210	1810	250	828	837	0.4	1.8
4-3-4	3.0	46	210	1810	250	848	864	0.6	6.7
4-3-9	2.8	46	200	1730	400	705	772	3.2	9.0
4-3-10	3.0	46	200	1730	400	714	786	2.9	9.5
23-5-5	2.2	34	130	1140	500	660	720	4.1	13.1
23-5-6	2.0	34	120	1040	500	639	708	3.3	11.6
23-5-9	2.0	35	120	1060	600	530	547	1.2	13.4
4-3-7	8.7	67	640	5400	164	1011	1095	0.7	3.7
4-3-8	9.2	67	640	5400	164	1049	1072	0.6	4.6
4-3-5	8.7	65	650	5480	500	752	800	2.0	7.8
4-3-6	9.1	65	650	5480	500	750	825	3.2	9.1

[a] Unirradiated control specimens.

test temperature for about 1–1.5 h prior to the start of a tensile test. All tests were performed at a displacement rate of 0.0021 mm/s, corresponding to an initial strain rate of 4×10^{-4} s^{-1}. All tests were performed using a 454 kg load cell that was accurate to within 0.5 kg. Dimensional measurements of the specimens were made using a calibrated digital micrometer accurate to 0.005 mm. Specimen temperatures during testing were maintained to within ±5 °C.

From the raw tensile traces, 0.2% offset yield strength (YS), ultimate tensile strength (UTS), engineering uniform elongation (UE), and total elongation (TE) were measured. Compliance-corrected test traces are shown in this paper instead of the raw tensile traces. To obtain the compliance-corrected test traces, the elastic strain associated with test machine and specimen compliance were removed from the strain data, and then the elastic compliance of the specimen was added back to the strain data using the known Young's modulus of the material at room temperature.

3. Results and discussion

3.1. Unirradiated materials

The tensile properties are listed in Table 2. Fig. 1 shows the tensile behavior of the unirradiated material for tensile test temperatures ranging from 20 to 600 °C. Examination of the unirradiated material tensile property trends as a function of temperature in Fig. 2 shows that YS, UTS, and UE all decrease with increasing tensile test temperature which is typical for similar materials [5,16]. TE decreases until about 400 °C and then increases with increasing tensile test temperature.

3.2. Irradiated materials

Fig. 3 shows the tensile behavior of Mod 9Cr–1Mo irradiated to between 1 and 3 dpa when tensile tested at temperatures ranging from 50 to 600 °C. Shown in Fig. 4 are the corresponding tensile properties along with the tensile properties of the unirradiated material. The YS and UTS of the irradiated material show the same

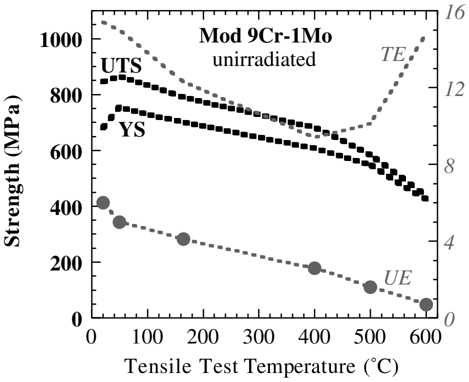

Fig. 2. Tensile properties of unirradiated Mod 9Cr–1Mo as a function of tensile test temperature.

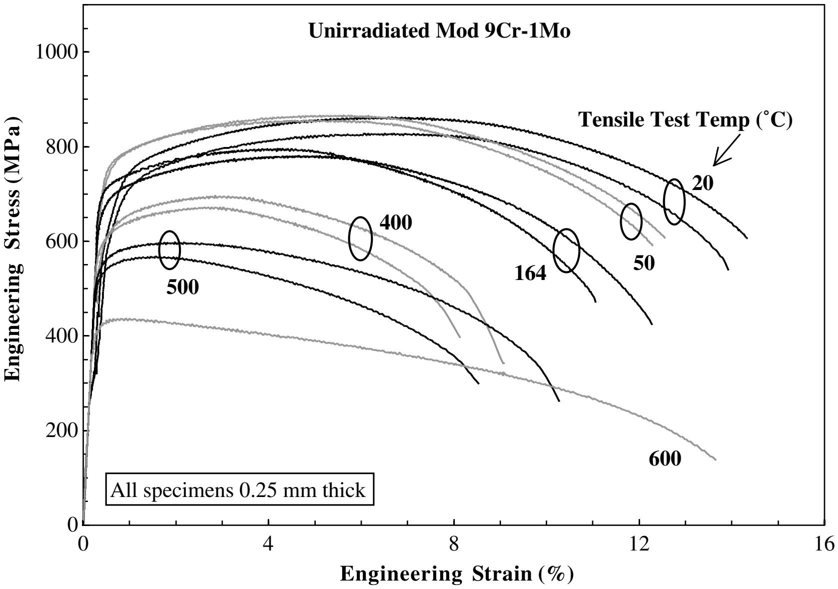

Fig. 1. Engineering stress versus engineering strain traces of unirradiated Mod 9Cr–1Mo tensile tested at temperatures ranging from 20 to 600 °C.

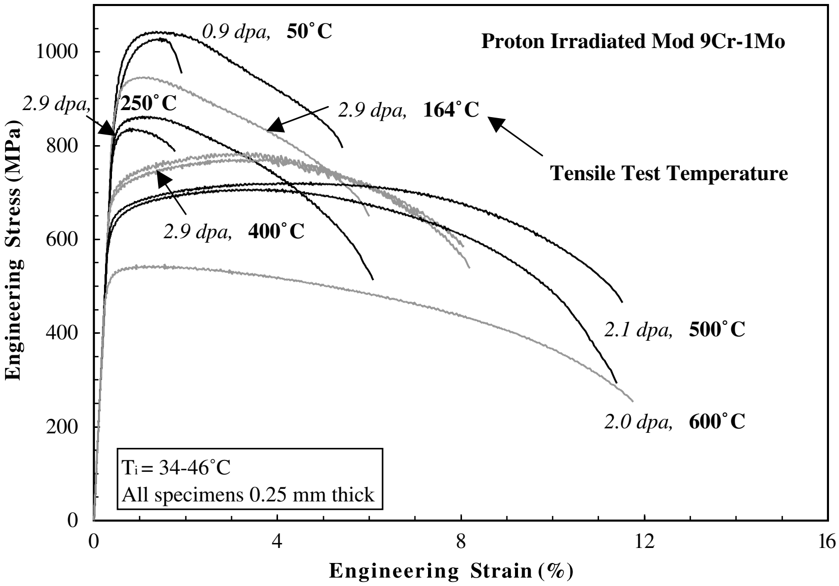

Fig. 3. Engineering stress versus engineering strain traces of Mod 9Cr–1Mo irradiated to doses between 2 and 3 dpa and tensile tested at temperatures between 50 and 600 °C.

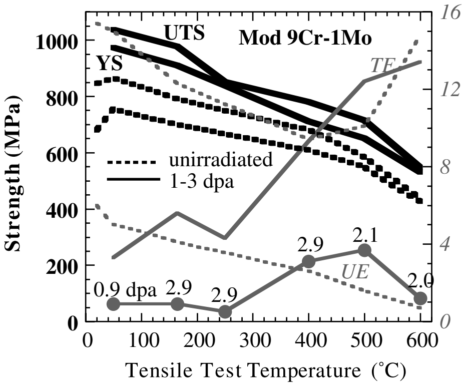

Fig. 4. Tensile properties of Mod 9Cr–1Mo plotted as a function of tensile test temperature for unirradiated material and material irradiated to doses from 1 to 3 dpa. Irradiation temperature varied from 34 to 46 °C.

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 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, 164, 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. Fig. 5 shows the tensile behavior of Mod 9Cr–1Mo after 9 dpa when tensile tested at either 164 or 500 °C. The tensile properties from these tests are shown in Fig. 6. Compared to the 1–3 dpa tensile properties, at 9 dpa, the YS and UTS are increased while the UE and TE are decreased.

The tensile properties as a function of dose are shown in Fig. 7 for tensile test temperatures of 164 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 have begun to 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.

For the 1–3 dpa specimens, the increase in YS over the unirradiated material (ΔYS) is temperature dependent. ΔYS is about 250 MPa at 50 °C and decreases to about 100 MPa at 600 °C. As these temperatures and hold times are too low to significantly affect the pre-irradiation microstructure, the most likely cause for this decrease in ΔYS is annealing of the radiation-induced defects. The effect of post-irradiation anneals on the

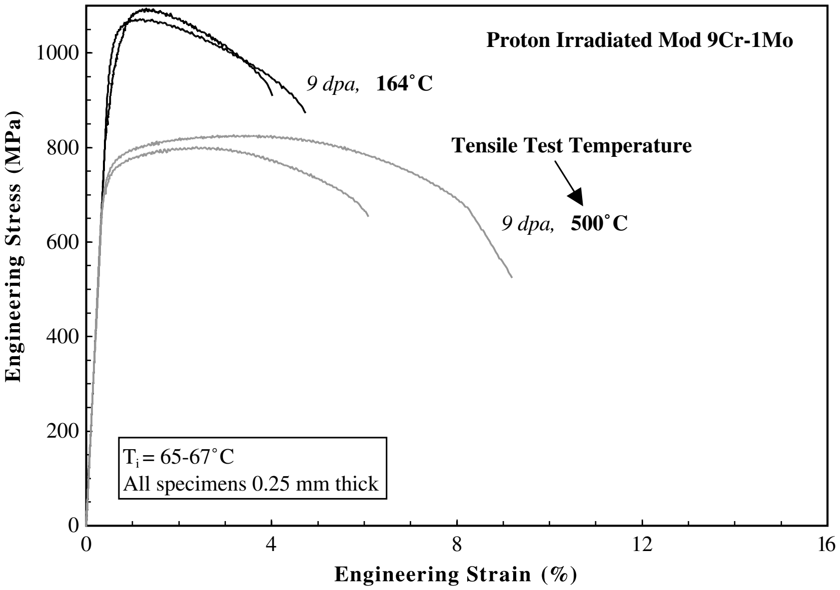

Fig. 5. Engineering stress versus engineering strain traces of Mod 9Cr–1Mo irradiated to a dose of about 9 dpa and tensile tested at either 164 or 500 °C.

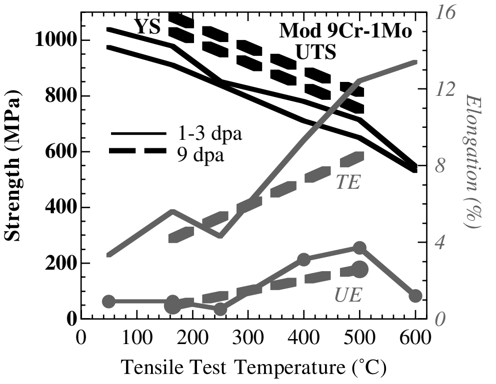

Fig. 6. Tensile properties of Mod 9Cr–1Mo plotted as a function of tensile test temperature for material irradiated to 1–3 dpa or about 9 dpa. Irradiation temperature of 9 dpa material was between 65 and 67 °C.

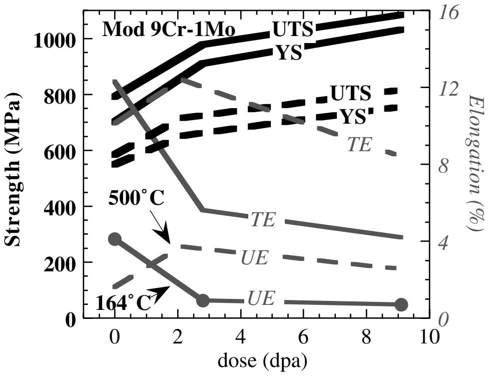

Fig. 7. Tensile properties as a function of dose at 164 and at 500 °C.

mechanical properties of ferritic–martensitic steels has been previously observed by others [17]. Transmission electron microscopy observations of the irradiated specimens annealed for 1 h at 500 °C showed no obvious changes in the microstructure compared to unannealed irradiated specimens. Based on this observation, it is likely that irradiation-induced defects not visible by transmission electron microscopy are coarsening and probably dissolving or annihilating. At 9 dpa, the YS again follows the same temperature dependent trend as the unirradiated material, with ΔYS about equal to 330 MPa at 164 °C and 200 MPa at 500 °C.

At temperatures below 300 °C, the high YS and low UE observed in the irradiated materials is thought to be due to the irradiation-induced formation of a fine dispersion of small, shearable defects [1,3,8]. These defects are thought to very effectively pin dislocations which causes a large increase in YS. These irradiation-induced defects can reduce the UE in at least two different ways. The first way is by flow localization which occurs when a dislocation breaks free and sweeps out a dislocation-free channel by shearing and possibly annihilating all the irradiation-induced defects it encounters [1,3,8]. These cleared channels provide low-resistance paths for subsequent dislocations. Dislocation–dislocation interactions in these channels is minimal resulting in little or no work-hardening occurring as dislocations pass through the channels. Thus deformation is largely confined to these channels, and the material quickly begins to neck in the region where the first channels form. Another method by which low temperature irradiation can cause low UE is simply to cause an increase in YS beyond that which the material can reach by work-hardening. In this scenario, the material simply cannot work-harden after yielding, and plastic instability occurs immediately after yield. As this 9Cr steel has a large number of carbides that would effectively limit the channeling phenomenon, its is likely that the presence of the irradiation-induced defects is simply raising the strength of the material to the point where it cannot become any stronger by work-hardening.

At tensile test temperatures between 400 and 600 °C, the UE of the material irradiated to 1–3 dpa was observed to be greater than or equal to the unirradiated values. TE showed a similar trend. Such a temperature dependence on UE 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 [18]. It has also been observed in ferritic–martensitic steels that have been neutron-irradiated to relatively high doses (10–59 dpa) at elevated temperature [19–21]. Therefore, it appears that this is not unusual behavior for this material. The cause of this behavior appears to be due, in part, to the formation of the irradiation-induced microstructure and its subsequent alteration caused by the 1 h post-irradiation anneal. It is tempting to suggest that coarsening of the microstructure alone accounts for the increase in UE, but this does not fit the trends in the data as it would be reasonable to expect that annealing of the defects produced by irradiation would cause the UE of an irradiated specimen to approach the value for the unirradiated material, but not surpass it. On this basis, it seems that there is something else at work besides annealing of the irradiation-induced defects. Elevated temperatures can have several effects on dislocation behavior during plastic deformation. For instance, elevated temperatures can lead to the activation of additional slip systems, and elevated temperatures can also promote increased annihilation or recombination of dislocations during plastic deformation. Perhaps it is one of these phenomena acting synergistically with the annealed, irradiation-induced defect population that is leading to the increase in UE over the unirradiated material at around 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 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 UE [1,5,9–11]. The extreme reductions in UE are a result of a lack of work-hardening ability that promotes early plastic instability [1,3,8]. For irradiation/tensile test temperatures from 450 to 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 [17], 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 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 30–250 °C. For temperatures from 500 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 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 the tensile properties of ferritic–martensitic steels that were neutron-irradiated 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 2 h post-irradiation anneal.

4. Summary and conclusions

Tensile tests were performed at temperatures ranging from 20 to 600 °C on unirradiated Mod 9Cr–1Mo and Mod 9Cr–1Mo irradiated to 1–3 dpa and 9 dpa in a spallation environment at temperatures ranging from 35 to 67 °C. The effect of tensile test temperature on the unirradiated material was to decrease the YS, ultimate strength, and UE as test temperature increased. Relative to unirradiated material, the effect of increasing tensile test temperature on the material irradiated to between 1 and 3 dpa was to increase the YS and ultimate strength over the unirradiated material throughout the temperature range. UE of the material irradiated to between 1 and 3 dpa dropped to values of 1% or less at tensile test temperatures from 50 to 250 °C whereas between 400 and 500 °C, the UE was observed to increase steadily and surpass the observed UE of the unirradiated material at 500 °C. At 600 °C, the UE of the irradiated material had decreased to match that of the unirradiated material. The tensile properties after 9 dpa showed similar trends. 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 behavior 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 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 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 irradiated ferritic–martensitic steels were mostly 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 2 h post-irradiation anneals performed prior to tensile testing. As helium and hydrogen are present in relatively large quantities after proton-irradiation, it is possible that these remaining defects contain helium and hydrogen.

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