Journal of Nuclear Materials 296 (2001) 119–128

journal of nuclear materials

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The mechanical properties of 316L/304L stainless steels, Alloy 718 and Mod 9Cr–1Mo after irradiation in a spallation environment

S.A. Maloy [a,*], M.R. James [a], G. Willcutt [a], W.F. Sommer [a], M. Sokolov [b], L.L. Snead [b], M.L. Hamilton [c], F. Garner [c]

[a] Los Alamos National Laboratory, MS-H809, Los Alamos, NM 87545, USA
[b] Oak Ridge National Laboratory, Metals and Ceramics Division, Oak Ridge, TN 37831-6151, USA
[c] Pacific Northwest National Laboratory, Richland, WA 99352, USA

Abstract

The Accelerator Production of Tritium (APT) project proposes to use a 1.0 GeV, 100 mA proton beam to produce neutrons via spallation reactions in a tungsten target. The neutrons are multiplied and moderated in a lead/aluminum/water blanket and then captured in ^{3}He to form tritium. The materials in the target and blanket region are exposed to protons and neutrons with energies into the GeV range. The effect of irradiation on the tensile and fracture toughness properties of candidate APT materials, 316L and 304L stainless steel (annealed), modified (Mod) 9Cr–1Mo steel, and Alloy 718 (precipitation hardened), was measured on tensile and fracture toughness specimens irradiated at the Los Alamos Neutron Science Center accelerator, which operates at an energy of 800 MeV and a current of 1 mA. The irradiation temperatures ranged from 50°C to 164°C, prototypic of those expected in the APT target/blanket. The maximum achieved proton fluence was 4.5×10^{21} p/cm^2 for the materials in the center of the beam. This maximum exposure translates to a dpa of 12 and the generation of 10 000 appm H and 1000 appm He for the Type 304L stainless steel tensile specimens. Specimens were tested at the irradiation temperature of 50–164°C. Less than 1 dpa of exposure reduced the uniform elongation of the Alloy 718 (precipitation hardened) and Mod 9Cr–1Mo to less than 2%. This same dose reduced the fracture toughness by 50%. Approximately 4 dpa of exposure was required to reduce the uniform elongation of the austenitic stainless steels (304L and 316L) to less than 2%. The yield stress of the austenitic steels increased to more than twice its non-irradiated value after less than 1 dpa. The fracture toughness reduced significantly by 4 dpa to $\sim$100 MPa m$^{1/2}$. These results are discussed and compared with results of similar materials irradiated in fission reactor environments.

1. Introduction

The US Department of Energy is investigating the use of an accelerator to produce tritium for national defense purposes. The Accelerator Production of Tritium, APT, project design uses a 1.0 GeV, 100 mA proton beam to produce neutrons through the spallation of a tungsten target. The spallation neutrons are moderated and multiplied in a surrounding lead/ aluminum/ water blanket and captured by ^{3}He gas to produce tritium. Therefore, the structural materials in the APT target and blanket systems are exposed to a high energy proton and spallation neutron flux at energies up to 1.0 GeV [1]. This exposure causes displacement damage and the production and implantation of large quantities of hydrogen and helium in structural materials. The energy of this proton/neutron flux is high compared to the particle energies in a fission reactor. Thus the amounts of hydrogen and helium produced per dpa of exposure in the APT will be significantly greater than the amount of gas produced in a typical fission reactor. The temperature of the APT target/blanket, 50–160°C, is also significantly lower than most fission reactor

* Corresponding author. Tel.: +1-505 667 9784; fax: +1-505 667 2787.
E-mail address: maloy@lanl.gov (S.A. Maloy).

0022-3115/01/$ - see front matter
PII: S0022-3115(01)00514-1

environments. The combination of high particle energies, enhanced gas production and low operating temperatures may make irradiation induced degradation of the APT target/blanket materials differ significantly from the degradation previously experienced in fission reactors.

The effects of high energy proton and neutron irradiation on the mechanical properties of APT target and blanket structural materials were determined in an APT experiment which exposed mechanical test specimens to the 800 MeV, 1 mA Gaussian proton beam (where $2\sigma = 3$ cm) at the Los Alamos Neutron Science Center (LANSCE) [2]. Specimens were held in and near the proton beam in stainless steel envelopes to allow them to be irradiated without direct exposure to the cooling water. Because of the small size and high intensity of the Gaussian beam, specimens had to be small and thin to obtain a uniform flux and achieve prototypic APT temperatures (50–160°C).

Various mechanical properties including tensile, fracture toughness, etc., were measured on specimens after irradiation. This paper summarizes the effect of high energy proton and neutron irradiation on the tensile and fracture toughness properties of Alloy 718, 316L and 304L and Mod 9Cr–1Mo. Each of these alloys is a candidate structural material for the APT target and blanket.

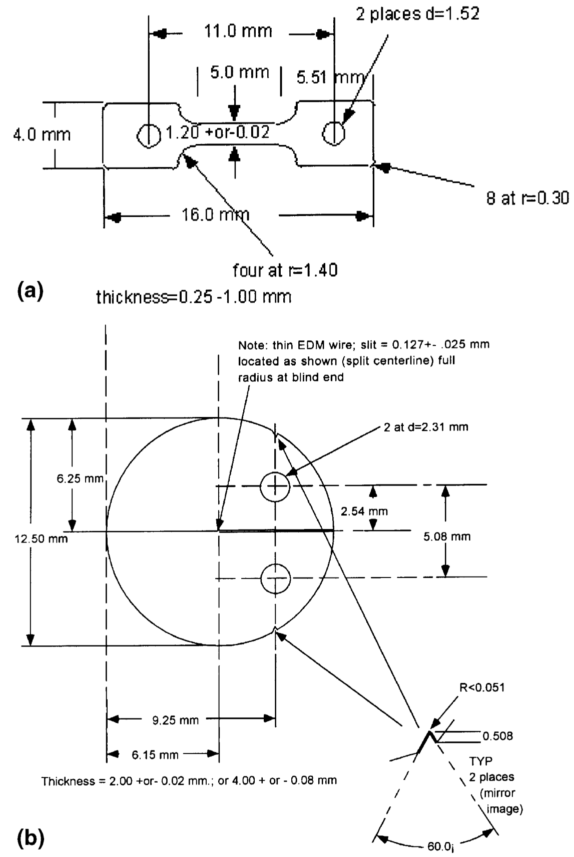

Fig. 1. Schematic showing (a) the dimensions of the S-1 tensile specimen and (b) the dimensions of the sub-size disk compact tension specimen used for measuring the mechanical properties of materials after irradiation in a spallation environment.

2. Experimental

The S-1 tensile specimens, Fig. 1(a), were electro-discharge machined (EDM'd) from two thicknesses of sheet material, 0.25 and 0.75–1.0 mm. Subsize compact tension specimens (Fig. 1(b)) were EDM'd from 2 to 4 mm thick sheet material. Variations in specimen thickness coupled with the thickness effects on energy implantation and heat transfer provided a technique to irradiate materials to the same flux at different temperatures. The compositions of the different heats of material are shown in Table 1. The Mod 9Cr–1Mo specimens were machined from the same heat of material regardless of specimen thicknesses. The compact tension specimens were cut with one set having the crack parallel to the rolling direction and a second set having the crack perpendicular to the rolling direction. The as-machined Alloy 718 and Mod 9Cr–1Mo specimens were heat treated by first separately wrapping the specimens of each material in Nb foil packets and placing them in a quartz tube with a titanium ampoule which was back-filled with argon and sealed. The Alloy 718 specimens were annealed at 1065°C for 30 min and air cooled. This was followed by aging at 760°C for 10 h, furnace cooling to 650°C and holding for a 20 h total furnace time, and finally air cooling. The Mod 9Cr–1Mo specimens were normalized at 1038°C for 1 h and air cooled followed by a temper at 760°C for 1 h and air cooling. The 304L and 316L materials were received in the annealed form, and no further heat treatment was performed. The Vickers hardness measurements at a 500 g load and grain size measured for each heat of material are shown in Table 2. The largest difference in hardness observed was between the two different heats of 304L stainless steel (174 kg/mm^2 at 0.75 mm thick and 159 kg/mm^2 at 0.25 mm thick).

The tensile and fracture toughness specimens were irradiated for six months at the LANSCE accelerator. After irradiation, the dose for each specimen was determined from analysis of pure metal activation foils placed next to specimens during irradiation. Details of the dose determination can be found in [3]. The fluence was first calculated using the Los Alamos High Energy Transport (LAHET) Code System [4,5] Then, the gamma spectra measured from the activation foils were used with the STAYSL2 code to adjust the calculated fluences. A mathematical fit of the position dependence of

Table 1
Elemental composition of materials used in APT irradiation in wt%

Material	Lot	Al	C	Cr	Cu	Fe	Mn	Mo	Ni	P	S	Si	Ti	Others
718 0.25 mm	E618	0.48	0.04	18.13	0.08	Bal	0.13	3.06	53.58	0.008	0.001	0.11	1.03	Nb + Ta-4.98
718 0.75 mm	L426	0.54	0.05	18.13	0.05	Bal	0.21	3.01	52.7	0.005	0.002	0.13	1.06	Nb + Ta-5.07; Co-0.4; B-0.004
718 2, 4 mm	G417	0.47	0.04	17.71	0.15	Bal	0.12	3.00	54.79	0.013	0.001	0.13	0.98	Nb-4.98; Co-0.19; B-0.002
316L 0.25 mm	E835		0.019	17.26	0.26	Bal	1.75	2.57	12.16	0.022	0.006	0.65		
316L 0.75 mm	L406		0.022	16.05		Bal	1.82	2.08	10.11	0.022	0.0002	0.48		
316L 2, 4 mm	D306		0.010	17.33	0.18	Bal	1.61	2.09	10.62	0.024	0.019	0.43		Co-0.21; N-0.060
304L 0.25 mm	K953		0.020	18.23	0.38	Bal	1.77	0.33	9.68	0.026	0.002	0.54		
304L 0.75 mm	L404		0.060	18.19	0.4	Bal	1.86	0.34	8.14	0.030	0.0003	0.48		
304L 2, 4 mm	K861		0.013	18.15	0.23	Bal	1.80	0.18	8.35	0.025	0.010	0.43		Co-0.17, N-0.085
Mod 9Cr–1Mo	10148	0.002	0.089	9.24	0.08	Bal	0.47	0.96	0.16	0.021	0.006	0.28	0.002	V-0.21; Nb-0.054; Co-0.019; N-0.035; O-0.008

fluence was used to determine the fluence at each specimen position. The temperature for each specimen was determined by calculations that used measurements from thermocouples placed near each specimen to determine gap resistances between samples and cover plates. The details of this temperature measurement can be found in [6]. The calculated irradiation temperature varied from 50°C to 164°C depending on specimen thickness and the location of the specimen with respect to center of the proton beam.

The irradiated specimens were tensile tested in air (except for the 250°C and 400°C tests on Mod 9Cr–1Mo) using an Instron machine equipped with a high temperature furnace. Except where noted, materials were tested at the irradiation temperature. Specimens were tested at an initial strain rate of 10^{-4}/s. Load and displacement were measured and converted to stress and strain. The stress/strain curve for each specimen was corrected for machine compliance. The compliance-corrected stress/strain curves were used to determine 0.2% offset yield stress, ultimate stress, uniform elongation and total elongation. Fracture surfaces of selected tensile specimens were examined using a scanning electron microscope (SEM). These SEM micrographs were used to determine reduction of area by measuring the fracture surface area in relation to the area of the gage before testing. The fracture toughness was determined after irradiation by performing J-integral tests using an MTS testing machine in a hot cell. Procedures for testing followed ASTM E1737-96.

3. Results

3.1. Tensile testing

The tensile properties of Alloy 718 in the precipitation hardened condition, 316L and 304L in the annealed condition, and Mod 9Cr–1Mo in the tempered condition were determined after irradiation to a maximum dose of 13 dpa. The thickness of the samples were either 0.25 or 0.75 mm thick. Representative stress/strain curves of irradiated Alloy 718, given in Fig. 2, show that the work hardening rate is essentially zero after only 0.09 dpa of exposure and does not recover as dose increases. The yield strength, shown in Fig. 3, increases with dose up to 1 dpa and then gradually decreases out to 12 dpa. On the other hand, the uniform elongation drops almost immediately to less than 2% after 0.5 dpa and remains low for irradiation to 12 dpa.

Representative stress/strain curves for 316L/304L stainless steel specimens irradiated and tested at 50°C are shown in Fig. 4. These curves show irradiation-induced increases in yield strength, losses in ductility and reductions in work hardening capacity. Test data, for all

Table 2
Hardness and grain size of materials used in APT irradiation

Material	Thickness (mm)	Micro-hardness (kg/mm^2)	Grain size (μm)
Alloy 718 (precipitation hardened)	4.0	486	65
	2.0	473	55
	1.0	458	30
	0.25	465	47
Mod 9Cr–1Mo (tempered)	4.0	293	27
	2.0	295	22
	0.75	283	21
	0.25	278	19
304L (annealed)	4.0	171	55
	2.0	175	65
	0.75	174	18
	0.25	159	15
316L (annealed)	4.0	159	32
	2.0	157	32
	0.75	149	21
	0.25	153	16

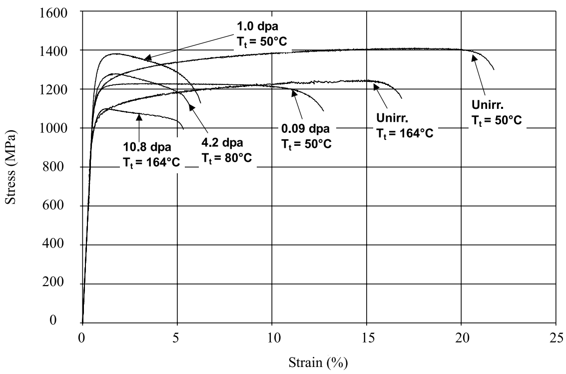

Fig. 2. A representative plot of stress/strain curves measured on Alloy 718 in the precipitation hardened condition after irradiation in a spallation environment.

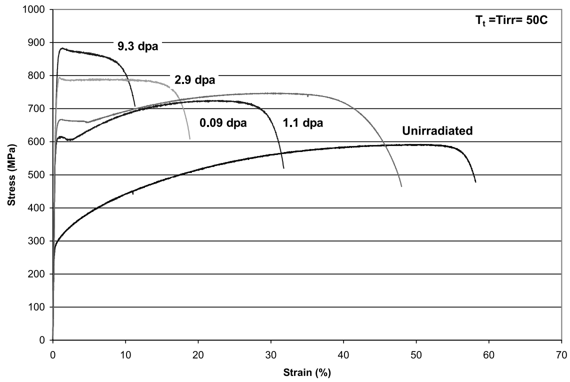

Fig. 4. A representative plot showing stress/strain curves measured on annealed 316L stainless steel after irradiation in a spallation environment.

the 304L/316L stainless steel specimens, given in Fig. 5, show a sharp irradiation-induced increase in yield strength. The irradiation-induced changes in uniform elongation are more complex, as shown in Fig. 5. The non-irradiated uniform elongation ranges from 40% to 60%, and sharp decreases in uniform elongation to less than 20% are observed after 0.01 dpa leading to a more gradual decrease out to 3–4 dpa where the uniform elongation decreases rapidly to less than 1%.

The effect of test temperature on the irradiated tensile properties of 316L/304L is shown in Fig. 6(a). Specimens irradiated to the same dose between 1 and 4 dpa were tested at increasing test temperature from 20°C to 164°C. Uniform elongation shows a significant decrease with increasing test temperature between 20°C and 164°C. Fig. 6(b) shows the effect of test temperature on the stress/strain curve for materials irradiated to 9 dpa. Although the uniform elongation is the same at each temperature, there is a significant difference in the stress/strain curves after maximum load is reached.

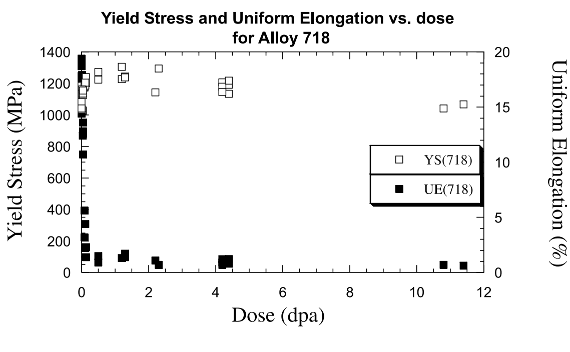

Fig. 3. Graph showing the change in 0.2% offset yield stress and uniform elongation in Alloy 718 after irradiation in a spallation environment ($T_t = T_{irr} = 50$–160°C).

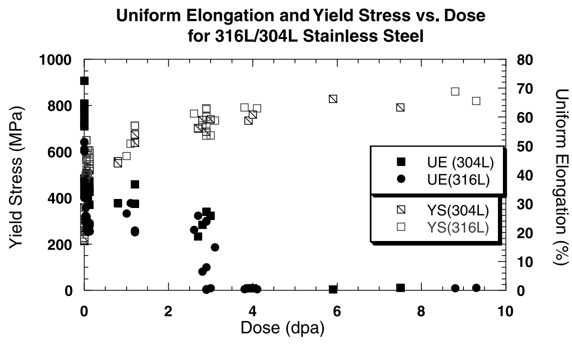

Fig. 5. A graph showing the change in uniform elongation and 0.2% offset yield stress with dose after irradiation in a spallation environment ($T_t = T_{irr} = 50$–160°C).

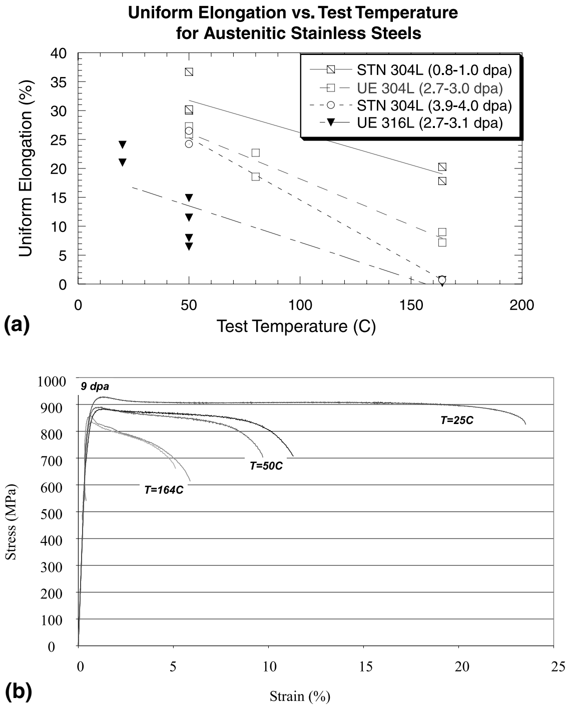

Fig. 6. (a) Graph showing the decrease in uniform elongation or strain-to-necking with increasing test temperature after irradiation to 1–4 dpa in a spallation environment and (b) stress/strain curves showing the effect of test temperature after irradiation to 9 dpa in a spallation environment.

Representative stress/strain curves for Mod 9Cr–1Mo after irradiation are shown in Fig. 7. As observed for Alloy 718, the work hardening rate goes to zero after greater than 0.05 dpa of exposure. The data, for all tests performed on Mod 9Cr–1Mo at temperatures from 50°C to 164°C, are plotted in Fig. 8 and show that the yield stress sharply increases with dose and the uniform elongation drops precipitously to less than 1% after slightly more than 0.1 dpa of exposure.

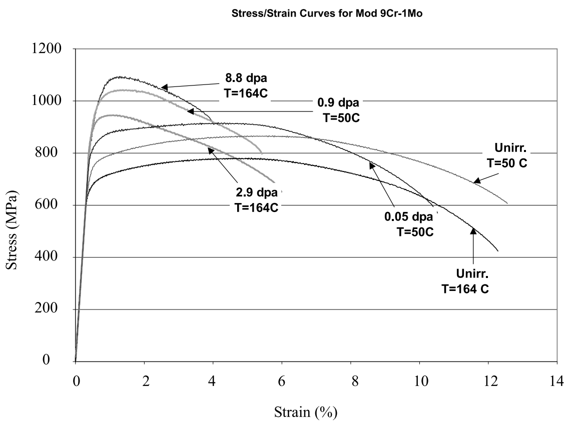

Fig. 7. A representative plot showing stress/strain curves for Mod 9Cr–1Mo after irradiation in a spallation environment.

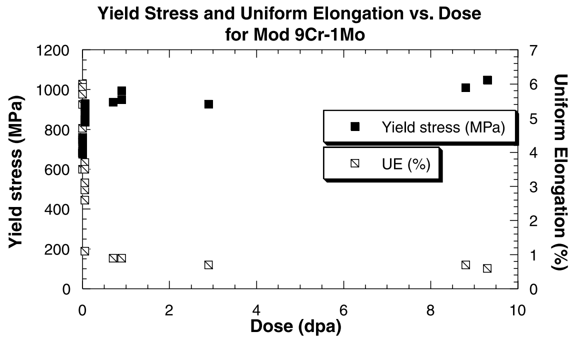

Fig. 8. A graph showing the change in 0.2% offset yield stress and uniform elongation with increasing dose after irradiation in a spallation environment ($T_t = T_{irr} = 50$–160°C).

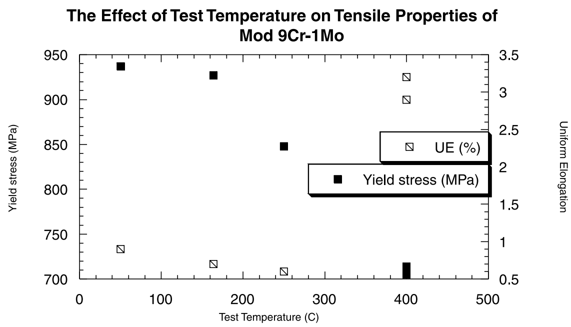

Fig. 9. A graph showing the change in 0.2% offset yield stress and uniform elongation with increasing test temperature for specimens irradiated to 3 dpa in a spallation environment ($T_{irr} = 35$–50°C).

The effect of test temperature was also evaluated on the irradiated tensile properties of Mod 9Cr–1Mo as shown in Fig. 9. Because Mod 9Cr–1Mo is also a candidate material for the Accelerator Transmutation of Waste program in which the target operates at higher temperatures, test temperatures up to 400°C in argon were investigated. The results show that uniform elongation is low (<1%) and does not significantly increase until the test temperature is raised to 400°C. A significant decrease of yield stress is also observed when testing at 400°C.

3.2. SEM analysis

The fracture surfaces of selected Alloy 718 and 316L and 304L austenitic stainless steel tensile specimens were

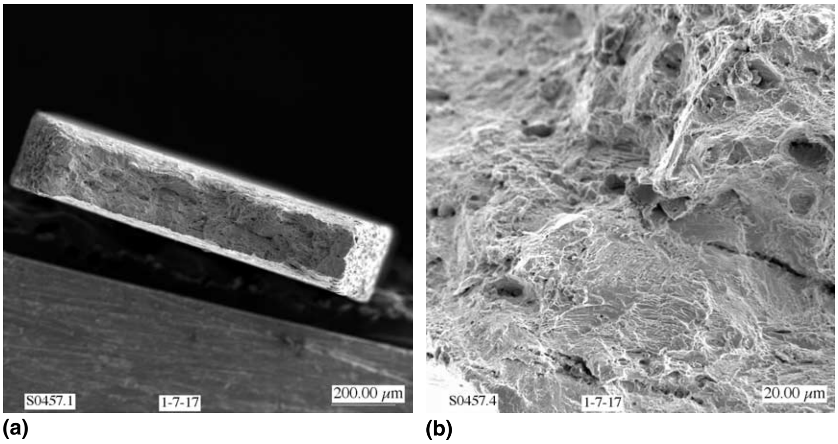

Fig. 10. Scanning Electron Micrographs showing the fracture surface of an Alloy 718 tensile specimen after irradiation to 11 dpa in a spallation environment ($T_t = T_{irr} = 50$–160°C).

observed using scanning electron microscopy. Fig. 10 shows the fracture surface of an Alloy 718 specimen irradiated to 11 dpa. The fracture still appears ductile although the fracture is quite localized, i.e., the necked/fracture region is confined to a very small portion of the gauge section of the specimen. Reduction of area was also measured on this specimen and on others at lower doses. The results are shown in Fig. 11. Although significant reductions in uniform elongation are apparent with increasing dose, very little change in reduction of area was observed. The reduction of area measurements were insensitive to test temperature (between 50°C and 164°C) as well.

The fracture surface of a 316L stainless steel specimen irradiated to 9 dpa is shown in Fig. 12. Fracture is also quite localized and the appearance is more ductile than that observed for Alloy 718. Reduction of area was also measured on this specimen and on other 316L and 304L stainless steel specimens. The results shown in Fig. 10 reveal very little change in reduction of area for both stainless steels with increasing dose (and temperature between 50°C and 164°C) while the change in uniform elongation with dose and temperature is quite large.

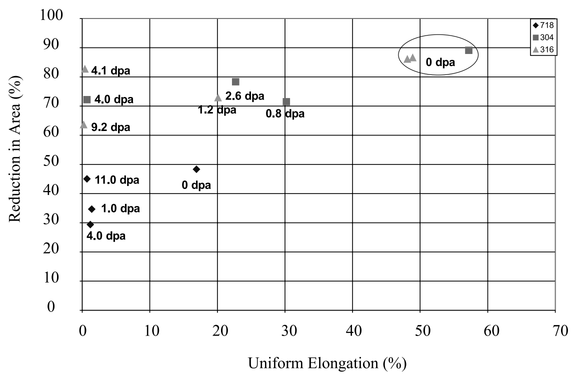

Fig. 11. Graph showing reduction of area plotted vs. uniform elongation for precipitation hardened Alloy 718 and annealed 304L and 316L stainless steel ($T_t = T_{irr} = 50$–160°C).

3.3. Fracture toughness

The change in fracture toughness with dose for precipitation hardened Alloy 718 and annealed 316L and 304L stainless steel is shown in Fig. 13. The toughness for Alloy 718 dramatically decreases with increasing dose to half its non-irradiated value after only 2 dpa. The change in toughness is more gradual for the austenitic stainless steels tending to saturate at less than 100 MPa $m^{1/2}$ after 4–6 dpa of exposure. The change in fracture toughness with dose for Mod 9Cr–1Mo is shown in Fig. 14. Significant decreases in fracture toughness are observed although there is large scatter in the results. The highest toughnesses are observed for the crack perpendicular to the rolling direction for doses up to 3 dpa.

4. Discussion

4.1. Alloy 718

The mechanical properties of Alloy 718 in the precipitation hardened condition show two salient effects with irradiation in a high energy proton and neutron flux. First, the yield stress increases slightly while the uniform elongation and fracture toughness decrease dramatically after exposure to 0.5 and 2 dpa, respectively. Second, the yield stress gradually decreases with increasing dose up to 12 dpa.

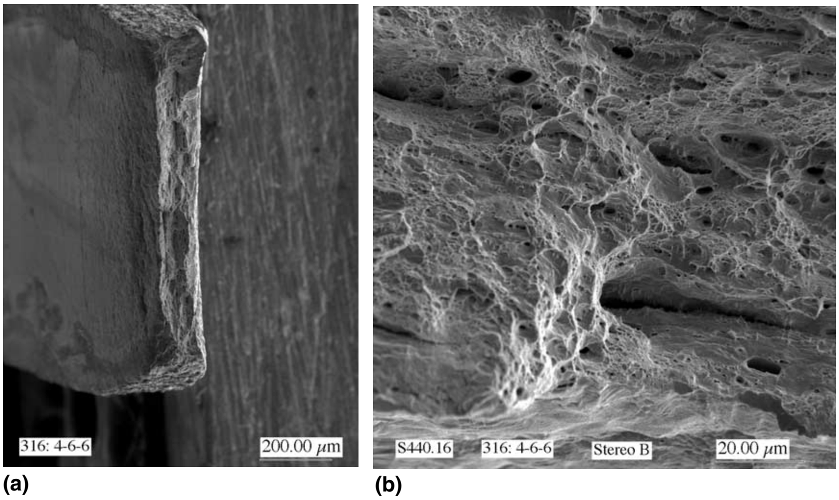

Fig. 12. Scanning Electron Micrographs showing the fracture surface of a 316L tensile specimen after irradiation to 9 dpa in a spallation environment $T_t = 164°C$, $T_{irr} = 72°C$.

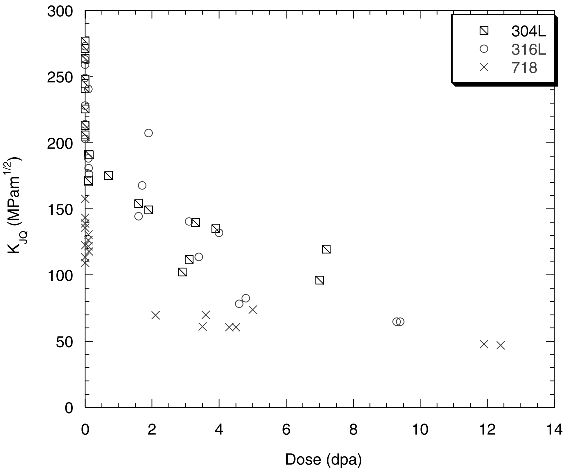

Fig. 13. Graph showing the variation of fracture toughness with dose for precipitation hardened Alloy 718 and annealed 316L and 304L stainless steel after irradiation in a spallation neutron spectrum ($T_t = T_{irr} = 50$–160°C).

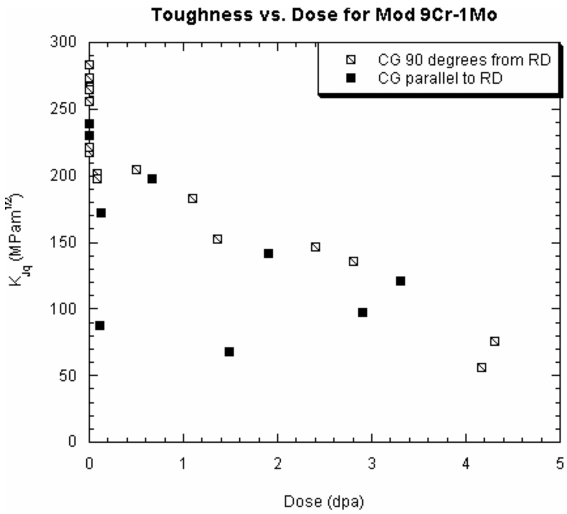

Fig. 14. Graph plotting fracture toughness vs. dose for Mod 9Cr–1Mo after irradiation in a spallation neutron spectrum ($T_t = T_{irr} = 50$–160°C).

The dramatic decrease in uniform elongation and fracture toughness with increasing dose can be explained by the increase in the density of irradiation-induced defects with increasing dose, which hardens the material and increases its strength, producing a concomitant decrease in ductility and toughness. Black spot damage and Frank loop formation has been observed by Sencer et al. [7] in Alloy 718. Such damage is similar to what has been observed for irradiation of Alloy 718 in a fission reactor [8]. In addition, comparison of the change in uniform elongation with dose obtained in this study to that measured after irradiation in the High Flux Isotope Reactor (HFIR) by Farrell et al. [9] shows very little difference in the results (Fig. 15).

The decrease in the yield stress with increasing dose above 1 dpa may be explained by a second microstructural change observed by Sencer et al. [7] In TEM studies, Sencer observed that the superlattice reflections for the strengthening precipitates, γ'/γ'', disappear after irradiation to doses greater than 0.5 dpa in a high energy proton and neutron flux. Since the irradiation temperature is very low, making complete dissolution of the precipitates unlikely, he interprets the loss of these

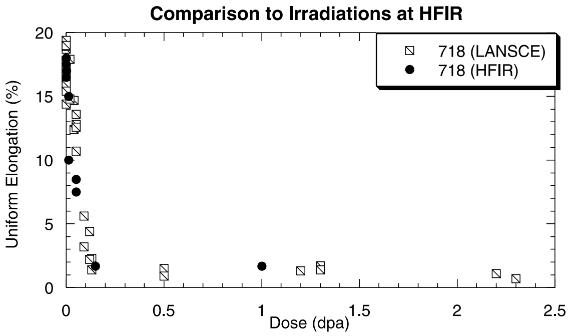

Fig. 15. A graph comparing the uniform elongation measured on Alloy 718 in tension after irradiation in an accelerator to similar doses irradiated in the High Flux Isotope Reactor ($T_t = T_{irr} = 20$–50°C).

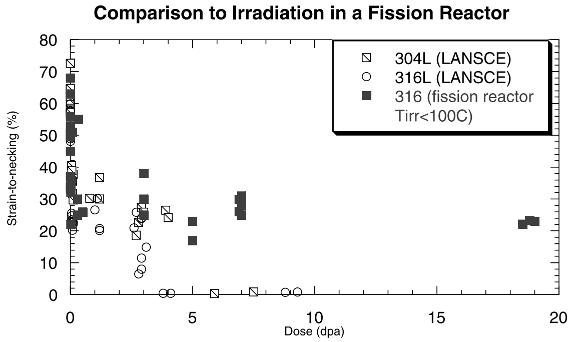

Fig. 16. A graph comparing the strain-to-necking measured on 300 series stainless steels after irradiation in the LANSCE accelerator to those measured after irradiation in a fission reactor [11] at low temperatures (<100°C) ($T_t = T_{irr} = 50$–80°C) for LANSCE data).

reflections, and the decrease in yield stress, as the results of irradiation-enhanced disordering of the hardening precipitates.

The fracture surfaces on the Alloy 718 specimens reveal very little change in fracture appearance and in measured reduction of area values with increasing dose. On the other hand, the uniform elongation drops significantly. The fracture still appears ductile but the localization of the neck in the tensile specimens, and the shape of the stress/strain curve, suggests a very localized type of plastic flow, resulting in a small uniform elongation. Such strain localization has commonly been caused by both hydrogen and irradiation embrittlement.

4.2. 316L/304L Stainless steel

The response of the mechanical properties of austenitic stainless steels, 316L and 304L, to irradiation in a high energy proton and neutron flux is a more gradual increase in yield stress concomitant with a decrease in uniform elongation and fracture toughness with increasing dose, (Figs. 5 and 13). These changes occurring at low dose are attributed to increases in the density of irradiation-induced defects (black spot damage and Frank loops) with increasing dose as observed by Sencer et al. [7]. With increasing dose above 3 dpa, the uniform elongation dramatically decreases. Comparison of measured values of ductility, in this case, strain to necking, for LANSCE-irradiated vs fission neutron-irradiated 316 stainless steel at 20–100°C, does not show the same significant ductility decrease at 3 dpa, Fig. 16. Strain to necking values equal uniform elongation values except in very few cases where the maximum load does not coincide with the maximum elongation before necking occurs. Analyses by Oliver et al. [10] show that in addition to the displacement damage in these alloys, they also retain up to 2000 appm He and 2500 appm H after irradiation in a high energy proton/spallation neutron spectrum. Such large amounts of retained gas may contribute to the observed differences in tensile properties.

A second effect is that increasing the test temperature from 20°C to 164°C results in decreases in the measured uniform elongations (and strain-to-necking) in the 304L and 316L stainless steels for specimens irradiated to doses between 1 and 4 dpa. Fig. 6(b) shows at higher doses, when a change in uniform elongation is not observed with increasing temperature, there is still a significant change in the shape of the stress/strain curve. A similar effect has been observed by Pawel et al. [11] in 316 stainless steel where a minimum uniform elongation (strain-to-necking) is observed for irradiations/testing performed around 300°C. It appears that data reported herein suggest that a minimum may appear at a lower temperature for irradiation in a spallation environment. However, more tests would be required at higher irradiation and test temperatures in a high energy proton/spallation neutron environment to confirm this effect.

The fracture surfaces on the 316L/304L stainless steel specimens still show a ductile appearance after irradiation to 9 dpa. Reduction of area also remains high at 9 dpa. Therefore, the low uniform elongation results from very localized fracture in a ductile mode.

4.3. Mod 9Cr–1Mo

The effect of increasing proton and neutron flux on the mechanical properties of Mod 9Cr–1Mo in the tempered condition is to decrease the uniform elongation and fracture toughness while increasing the yield stress. This can be attributed to the microstructural changes observed by numerous investigators as well as Sencer [12] which show an increase in black spot and Frank loop damage with increasing dose. Our results compare well with the results of Spatig et al. [13] for F82H who measured a $\Delta\sigma$ of 210 MPa at 25°C after a dose of 0.16–0.26 dpa for irradiation in a 590 MeV proton beam while we observe a $\Delta\sigma$ of 200–250 MPa after a dose of 0.12 dpa at 50°C. In addition, large scatter was observed in the measured fracture toughness values with increasing dose. Because the test temperature is very close to the brittle-to-ductile transition temperature (which will increase with increasing dose), such scatter is not surprising.

The effect of increasing test temperature on the tensile properties after irradiation to 3 dpa shows that the uniform elongation increases while the yield stress decreases as the temperature is raised from 250°C to 400°C. This could be caused by annealing of radiation-induced defect clusters with increasing temperature.

5. Conclusions

The mechanical properties measured on Alloy 718 in the precipitation hardened condition, annealed 316L and 304L stainless steel and Mod 9Cr–1Mo in the tempered condition show the following effects after irradiation in a high energy proton/spallation neutron environment.

5.1. Alloy 718

1. The uniform elongation drops to less than 2% after only 0.5 dpa of exposure.
2. The yield stress increases slightly at low doses and then decreases with dose above 1 dpa of exposure.
3. The reduction of area shows very little change with increasing dose to 12 dpa.
4. The fracture toughness drops to less than 80 MPa $m^{1/2}$ after 2 dpa exposure.

5.2. 316L/304L Stainless steel

1. The uniform elongation gradually decreases with increasing dose but then abruptly drops at 3 dpa. Such an effect is not seen for irradiation in a fission spectrum and may be related to high retained helium and hydrogen in the material.
2. The yield stress increases with increasing dose to more than two times its unirradiated value.
3. For specimens irradiated to 1–3 dpa, increasing test temperature from 20°C to 160°C results in decreasing uniform elongation.
4. The reduction of area does not significantly change with increasing dose to 9 dpa.
5. The toughness gradually decreases to less than 120 MPa $m^{1/2}$ after 5 dpa exposure.

5.3. Mod 9Cr–1Mo

1. The yield stress increases to over 900 MPa (30% increase) while the uniform elongation decreases to less than 2% after only 0.5 dpa exposure.
2. A slight increase (to over 3%) in uniform elongation is observed along with a decrease in yield stress when the test temperature is raised to 400°C.
3. The toughness drops quicker for the crack growth parallel to the rolling direction than observed for the crack growth perpendicular to the rolling direction.

Acknowledgements

The authors would like to thank Manny Lopez and Toby Romero of LANL, Gary Whiting of PNNL and Wayne Bolinger and Gary Parks of ORNL for their expertise in handling and testing these tensile and fracture toughness specimens using manipulators in hot cells.

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