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Fracture toughness and tensile behavior of ferritic–martensitic steels irradiated at low temperatures

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Abstract

Disk compact tension and sheet tensile specimens of the ferritic-martensitic steels F82H and Sandvik HT-9 were irradiated in the High Flux Isotope Reactor (HFIR) at 90°C and 250°C to neutron doses of 1.5–2.5 dpa. For both steels, radiation hardening was accompanied by a reduction in strain hardening capacity (SHC). When combined with other literature data it is apparent that severe loss of SHC occurs in F82H for irradiation temperatures below ~400°C and in HT-9 for irradiation temperatures below ~250°C. For both alloys, severe loss of SHC does not correlate with brittle behavior during fracture toughness testing.

1. Introduction

For structural applications in fusion energy systems the ferritic-martensitic steels have several advantages based upon their resistance to void swelling, good thermal stress resistance, and well-established commercial production and fabrication technologies. In recent years, the attention of the fusion community has moved away from the conventional 9–12Cr steels (e.g. mod 9Cr–1Mo) towards replacement steels with more attractive activation properties with regard to safety and to long-term waste disposal (e.g. F82H, JLF, and OPTIFER alloys) [1]. Ferritic-martensitic steels undergo radiation-induced hardening during neutron irradiation at temperatures up to ~400°C, followed by a transition to fluence-dependent radiation softening in the region of 400–450°C. Radiation-hardening is often accompanied by a reduction in strain-hardening capacity (SHC) and uniform elongation (ε_u) and an increased propensity for brittle failure under certain combinations of temperature and loading conditions.

Using machined-notch Charpy impact specimens, it has been shown that reduced activation steels, based upon 8–9Cr WVTa, undergo much smaller increases in ductile-to-brittle transition temperature (DBTT) during irradiation at $\leqslant$ 365°C, compared to conventional steels [2,3]. The reasons for the improved behavior are not well understood. As part of an irradiation experiment designed to investigate the effects of low-temperature neutron irradiation on the fracture toughness of austenitic stainless steels [4], a limited number of disk compact tension (DCT) and sheet tensile specimens (SS-3) were included in the HFIR JP17, JP18, and JP19 capsules to allow a comparison of the fracture toughness and tensile properties of the conventional Sandvik HT-9 and the reduced activation F82H alloy irradiated under similar conditions.

2. Experimental

DCT and SS-3 specimens were prepared from HT-9, heat no. 9607R2, and from a 150 kg heat of F82H; compositions and final heat treatments are shown in Table 1. The DCT specimens (12.5 mm dia and 4.63 mm thick) were machined in the T–L orientation so that crack propagation occurred parallel to the rolling direction. Fatigue pre-cracking was at room temperature to a crack length to specimen width ratio (a/W) of approximately 0.5. This was followed by side-grooving on

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Table 1
Alloy composition (wt%) (balance Fe) and heat treatments

	Ni	Cr	Mo	Mn	Si	C	N	V	W	Ta	P	S
HT-9	0.51	12.1	1.04	0.57	0.17	0.20	0.027	0.28	0.45	–	0.016	0.003
F82H	0.05	7.65	–	0.49	0.09	0.093	0.002	0.18	1.98	0.038	0.001	0.001

Heat treatment: HT-9, 1050°C/1 h/AC +780°C/2.5 h/AC; F82H, 1040°C/0.5 h/AC +740°C/1.5 h/AC.

each side to a depth of ~10% of specimen thickness. The SS-3 tensile specimens were 0.76 mm thick with a gage length of 7.6 mm. Irradiation was carried out at two temperatures in the High Flux Isotope Reactor (HFIR) to neutron fluences of $\sim 3.7 \times 10^{21}$ n/cm^2 ($E > 0.1$ MeV) and $\sim 5.5 \times 10^{21}$ n/cm^2 ($E > 0.5$ MeV), resulting in displacement values in the range 1.6–2.3 dpa. In capsules JP 18 and 19, nominally at 90°C, the flat faces of the DCT specimens of HT-9 were in direct contact with the reactor coolant water, and the specimen temperature over the region of crack propagation was calculated to range from 80°C to 100°C (F82H was not included in the 90°C irradiation capsules). Capsule JP17, nominally at 250°C, was a shrouded type with reactor coolant flowing over an aluminum cladding tube containing the specimens; gamma-heating was used to raise the specimen temperature. For this capsule, the temperature in the crack-tip region of the specimen was calculated to range from 250°C to 300°C. The uncertainty in tensile specimen temperature is ±15°C. The experimental errors and specimen variability combine to give an overall uncertainty in yield stress of ±50 MPa.

After irradiation, J-integral resistance curves were determined using an unloading compliance technique in general accordance with the ASTM Standard E 813-89 – Standard Test Method for J_{IC}. Tensile testing was carried out in vacuum at a strain rate of 5.6×10^{-4}/s. Full experimental details have been presented elsewhere [4].

3. Results

For both alloys, radiation hardening is accompanied by reductions in ε_u for both irradiation temperatures. In the unirradiated condition the HT-9 alloy has superior SHC, with ε_u in the range 12–14%; the corresponding values of F82H are much lower (3–5%) (Table 2). Whereas HT-9 retains much of its SHC at 250°C following irradiation at 250°C ($\varepsilon_u \sim 6\%$), the ability to strain harden is completely lost for F82H ($\varepsilon_u \sim 0.3\%$). This behavior is graphically demonstrated by the engineering stress–strain curves shown in Figs. 1 and 2. Because of the very limited test matrix in this experiment, it is useful to include data from other experiments

Table 2
Summary of tensile data for Figs. 2 and 3

Alloy	Dose (dpa)	Irrad. Temp. (°C)	Test Temp. (°C)	σ_y (MPa)	σ_u (MPa)	ε_u (%)	ε_t (%)	Ref.
HT-9	0	–	25	476	696	14.3	24.1	
	0	–	90	416	622	14.1	23.7	
	0	–	250	427	627	12.1	20.8	
	2.7	90	90	903	914	0.4	8.7	
	2.7	90	250	758	770	0.5	9.8	
	2.7	250	250	875	932	6.2	13.4	
	2.7	250	90	962	1000	7.2	13.6	[6]
	2.5	300	RT	960	994	5.5	14.3	
	2.5	300	400	760	835	4.2	12.0	[6]
	10	200	200	1132	1162	0.9	8.9	[5]
	10	400	400	770	888	4.5	10.8	[5]
F82H	0	–	25	573	682	5.4	15.5	
	0	–	90	571	660	4.5	15.3	
	0	–	250	480	550	3.3	13.3	
	2.7	250	90	755	821	8.2	17.7	
	2.7	250	250	852	856	0.3	7.8	
	8	300	300	780	795	0.7	8.3	[5]
	10	200	200	805	805	0.0	8.2	[5]
	10	400	400	445	589	1.7	9.4	[5]

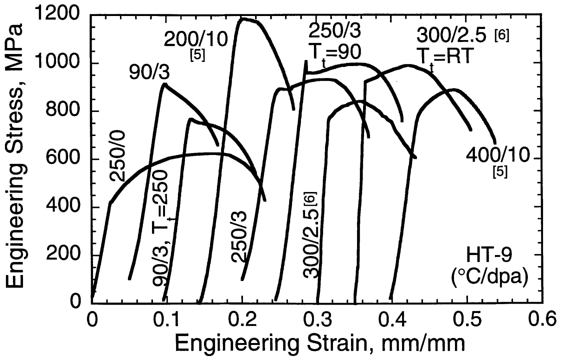

Fig. 1. Stress–strain behavior of HT-9 irradiated at the indicated temperatures and doses (°C/dpa). All tests were carried out at the irradiation temperature except as indicated.

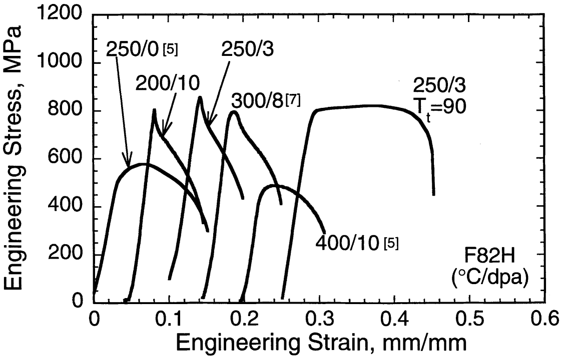

Fig. 2. Stress–strain behavior of F82H irradiated at the indicated temperatures and doses (°C/dpa). All tests were carried out at the irradiation temperature except for one test carried out at 90°C.

that have been reported recently in order to develop a broader picture of the deformation behavior of these alloys following irradiation. Figs. 1 and 2 illustrate the stress–strain behavior from this experiment, together with data at 200°C/10 dpa, from Robertson et al. [5], at 300°C/2.5 dpa from Horsten [6], and at 300°C/8 dpa from Shiba et al. [7]. For these dose levels (2–10 dpa), HT-9 retains SHC for irradiation temperatures of 300 and 250°C. However, SHC is strongly reduced at 200°C and is practically non-existent following irradiation at 90°C. Similar observations of complete loss of SHC have also been reported by Klueh et al. for HT-9 [8] and for 9Cr–1MoVNb [9] irradiated at 50°C and tested at 25°C.

The loss of SHC occurs over a wider range of irradiation temperatures for F82H. Following irradiation at 400°C, ε_u is only 1.5%, and this is reduced to well below 1% as the irradiation temperature is progressively reduced to 300°C, 250°C, and 200°C. Interestingly, the ability to strain harden is substantially restored for a specimen irradiated at 250°C when tested at 90°C.

The unirradiated and irradiated fracture toughness (K_J) data are summarized in Table 3. These data show some differences in irradiation behavior between the two alloys. However, because of the limited number of ferritic-martensitic steel specimens included in this HFIR experiment, it is necessary to view these data in the context of previously reported fracture toughness and tensile data in order to clarify these differences.

In Fig. 3, the fracture toughness data are presented together with the unirradiated data of Lucas et al. [10] for HT-9 and F82H and with the much larger body of data presented by Horsten [6] as part of the IEA collaborative program on ferritic-martensitic steels. In this work, rectangular compact tension specimens of HT-9 approximately 23×28 mm^2 and a thickness of 10–12 mm were irradiated in the High Flux Reactor (HFR) at Petten at ~80°C and 300°C to peak doses of 1.5 and 3.5 dpa, respectively.

The unirradiated ductile initiation fracture toughness of both F82H and HT-9 falls within a range of 200–300 MPa$\sqrt{m}$ over the range RT to 300°C. Lucas et al., have estimated that the unirradiated 100 MPa$\sqrt{m}$ transition temperatures for F82H and HT-9 are −100 and −75°C, respectively [10]. The data of Horsten [6] plotted in Fig. 3 indicate that low dose irradiation at ~80°C increases the

Table 3
Summary of fracture toughness data

Alloy	Dose (dpa)	Irrad. Temp. (°C)	Test. Temp. (°C)	J_Q (kJ/m^2)	K_J (MPa$\sqrt{m}$)	T
HT-9	0	–	22	484	316	104
	0	–	90	470	307	91
	0	–	250	415	283	41
	2.3	90	90	283	238	41
	2.4	250	25	5	31	–
	2.5	250	250	140	164	17
F82H	0	–	250	334	254	122
	2.1	250	25	117	156	–
	1.6	250	250	197	195	22

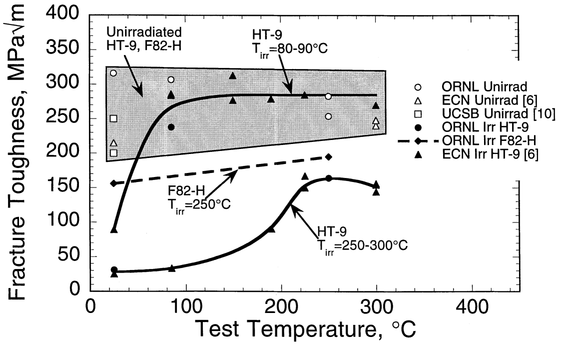

Fig. 3. Fracture toughness (K_J) data from various sources for unirradiated HT-9 and F82H and for HT-9 irradiated to 1.5–2.5 dpa at 80–90°C and 250–300°C, and for F82H irradiated to 1.5–2.1 dpa at ~250°C.

K_J transition temperature for HT-9 by ~100°C, whereas irradiation at ~300°C increases the transition temperature by about 275°C. In addition, the higher irradiation temperature results in a significant reduction in the upper shelf fracture toughness. The data obtained in the ORNL irradiation experiments at ~90°C and ~250°C are fully consistent with these observations.

Our limited data on F82H indicate somewhat better toughness than for HT-9 following low dose irradiation at ~250°C. For the 250°C test temperature both alloys are in the upper shelf regime with K_J values of ~195 MPa$\sqrt{m}$ for F82H and 164 MPa$\sqrt{m}$ for HT-9. At room temperature, F82H is in a cleavage initiation regime with a fracture toughness of 150 MPa$\sqrt{m}$. Under the same testing conditions, HT-9 exhibits lower shelf behavior and a K_J of 31 MPa$\sqrt{m}$.

4. Discussion

Radiation hardening in FCC, BCC, and CPH metal systems is frequently accompanied by a reduction in strain hardening capacity. There is an extensive body of evidence [11] indicating that the inability to strain harden following low temperature irradiation is due to inhomogeneous deformation and the development of coarse slip bands separated by regions of relatively undeformed material. Dislocation channeling occurs when glide dislocations cut through primary obstacles or are able to assimilate favorably oriented segments of loops. Subsequent dislocations move easily through the cleared channel, deformation becomes concentrated in a few widely-spaced slip bands, and the material exhibits small or negative strain hardening. On the other hand, the size and spacing of radiation-induced defects or precipitate particles, in combination with the appropriate test conditions, may be conducive to the by-passing of obstacles by the Orowan looping mechanism [12]. In these circumstances, significant dislocation generation occurs during deformation and the material exhibits positive strain hardening. For HT-9 irradiated at 250°C, 300°C, and 400°C, deformation is homogeneous with significant SHC and uniform strains of 4–7%; this is true for tests at 25°, 90°, and at the irradiation temperature. Following irradiation at 250°C, brittle fracture occurs during fracture toughness testing at RT despite the fact that the tensile deformation is homogeneous with relatively high levels of uniform strain. At lower irradiation temperatures, in the range 90–200°C, the stress–strain behavior reflects the existence of a different microstructural regime in which deformation is dominated by dislocation channeling and uniform strains are <0.5%. However, following irradiation at 90°C, failure occurs by ductile tearing during fracture toughness testing at 90°C. Thus, tensile embrittlement characterized by a very low level of uniform strain, does not correlate with an embrittlement characterized by lower shelf cleavage fracture. In F82H, the microstructural regime in which dislocation channeling is the dominant mode of deformation extends from 210°C to at least 400°C. In this regime uniform strains are low (<1%), with some indication of an improvement at 400°C (1.7%). Following irradiation at 250°C, failure occurs by ductile tearing during fracture toughness testing at 250°C. Thus, as in the case of HT-9, the loss of SHC and uniform strain, induced by dislocation channeling, do not necessarily lead to brittle behavior in a fracture toughness test.

The lack of a correlation between tensile toughness and fracture toughness in the ferritic-martensitic steels is probably related to the dominant role of yield stress. The initiation of a cleavage event in the fracture toughness specimens is determined by the magnitude of the normal tensile stress which has to exceed a critical value (σ_f*) over a finite region ahead of the crack tip. When radiation hardening is of sufficient magnitude for this to occur, cleavage will be initiated regardless of the mode of post-yield deformation behavior. In situations where radiation hardening is less severe and the yield stress is lower, it is possible that inhomogeneous channel deformation could lead to a reduction in the critical fracture stress σ_f* through the initiation of microcracks at dislocation pile-ups.

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References

[1] A. Kohyama, A. Hishinuma, D.S. Gelles, R.L. Klueh, W. Dietz, K. Ehrlich, J. Nucl. Mater. 233–237 (1966) 138.
[2] R.L. Klueh, D.J. Alexander, J. Nucl. Mater. 233–237 (1996) 336.
[3] M. Rieth, B. Dafferner, J. Nucl. Mater. 233–237 (1996) 229.
[4] D.J. Alexander, J.E. Pawel, M.L. Grossbeck, A.F. Rowcliffe, K. Shiba, 17th International Symposium on Effects of Radiation on Materials, ASTM STP 1270, 1996, p. 945.
[5] J.P. Robertson, R.L. Klueh, A. Wakai, A. Hishinuma, presented at 8th Int. Conf. on Fusion Reactor Materials, Sendai, Japan, 1997.
[6] M. Horsten, R.L. Klueh (Ed.), Proceedings of the IEA Working Group Meeting on Ferritic/Martensitic Steels, JET Culham, UK, 24–25 October 1996, ORNL/M-5674.
[7] K. Shiba et al., EuroMat 96; Topical Fonference on Materials and Nuclear Power, October 1996, Bournemouth, UK, pp. 265–272.
[8] R.L. Klueh, J.M. Vitek, M.L. Grossbeck, J. Nucl. Mater. 103&104 (1981) 797.
[9] R.L. Klueh, J.M. Vitek, M.L. Grossbeck, 11th International Symposium on Effects of Radiation on Materials, ASTM STP 782, 1982, p. 648.
[10] G.E. Lucas, G.R. Odette, K. Edsinger, B. Wirth, J.W. Scheckherd, 17th International Symposium on Effects of Radiation on Materials, ASTM STP 1270, 1996, p. 790.
[11] M.S. Wechsler, The Inhomogeneity of Plastic Deformation, American Society for Metals Seminar, 1971, pp. 19–52.
[12] M.F. Ashby, Acta Metall. 14 (1966) 679.