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Tensile properties of the ferritic martensitic steel F82H after irradiation in a spallation target

Z. Tong [a,b], Y. Dai [a,*]

[a] *Paul Scherrer Institut, 5232 Villigen PSI, Switzerland*
[b] *China Institute of Atomic Energy, 102413 Beijing, PR China*

ABSTRACT

Ferritic/Martensitic (FM) steel, F82H, was irradiated up to a displacement dose of 20 dpa (displacement per atom) at temperatures ranging from 510 to 1075 K in the third experiment of the SINQ Target Irradiation Program (STIP-III). Tensile testing was performed at 295 and 723 K. The tensile test results demonstrate that not only the specimen irradiated in the low temperature regime (<~675 K) but also those irradiated at elevated temperatures $\geqslant$710 K show significant hardening effect. After annealing at 873 K for 2 h the irradiated specimens still persist great hardening, which is usually not observed in FM steels after neutron irradiation at low temperatures and annealing at 873 K. The hardening observed in the specimens is believed to be due to the high-density He-bubbles formed in the specimens.

1. Introduction

Reduced-activation ferritic/martensitic steels have been developed as candidate structural materials for applications in advanced fusion [1,2] and fission reactors [3]. The F82H steel is one of such steels intensively studied in the fusion materials program because of its relatively low shifts in the ductile-to-brittle-transition temperature after neutron irradiation [4]. The F82H steel is also selected as one of main materials irradiated and studied in the SINQ (the Swiss Spallation Source) target irradiation program (STIP) [5,6] due to the promising applications of FM steels in advanced high power spallation targets [7,8]. A lot of work has been done in recent years on studying the microstructure and mechanical properties of the F82H steel after irradiation (e.g. [9–14]), mostly at low temperatures (<~675 K). In this work the tensile properties of the F82H steel irradiated in STIP-III in a temperature range of 510 – 1075 K to displacement doses of 8 – 20 dpa have been studied.

2. Experimental

The F82H steel (IEA Heat 9741) used in this study was provided through the Fusion Technology Materials Group of CRPP-EPFL, Switzerland. Its main composition is: 7.65 Cr, 2 W, 0.16 Mn, 0.16 V, 0.02 Ta, 0.11 Si and 0.09 C in wt% and Fe for the balance. The plate was normalized at 1313 K for 38 min and tempered at 1023 K for 1 h. The specimens were miniature type with a gauge section of 5 mm × 1 mm × 0.4 mm.

The STIP-III irradiation was effectively performed for about 68 weeks during 2002 and 2003 in SINQ target-5. As shown in [5,6] the temperature history of a STIP irradiation is usually rather complicated due to a large variation of the proton beam current (about ± 15% around an averaged value) and a lot of beam trips (about 50 times per day with a duration of 1 min or longer) during two years. This results in a similar magnitude of temperature variation in the specimens, because they are mainly heated by the proton beam. In STIP-III case, the situation was even worse. The maximum temperature of the tensile specimens used in this work was about 500 K in the first week, and then increased to about 625 K in the 7 weeks that followed. In the 9th week, it increased greatly to about 1025 K due to a 15% increase of proton beam current and some unclear reasons. It remained at this level or even slightly higher (~1075 K) afterwards for about 51 weeks. During the last 7 weeks of the irradiation it was about 940 K as the proton beam current at the target was reduced by about 20%. The temperature of each specimen was roughly proportional to the maximum value at a ratio depending on the energy deposition in the specimen. The specimens tested in this work are listed in Table 1, in which the temperature values are averaged ones calculated with the ANSYS code [5,6]. As described above it should be noted that the temperature variation is around ± 15%, plus a lot of low temperature trips.

The irradiation displacement dose of the specimens is between 8 and 20 dpa and the corresponding helium concentration is between 500 and 1800 appm, which was evaluated in the same way as described in the previous papers (e.g. [14]). The main irradiation parameters of the specimens are listed in Table 1.

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[*] Corresponding author. Tel.: +41 56 310 4171; fax: +41 56 310 4529.
E-mail address: yong.dai@psi.ch (Y. Dai).

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Table 1
Irradiation parameters and tensile properties of the F82H specimens. Displacement dose (*D*), yield stress (YS), ultimate tensile strength (UTS), strain-to-necking (STN) and total elongation (TE).

Specimen ID	$T_{irr.}$* (K)	*D* (dpa)	[He] (appm)	$T_{ann.}$ (K)	T_{test} (K)	YS (MPa)	UTS (MPa)	STN (%)	TE (%)
–	–	–	–	–	295	485	640	6.7	17.2
–	–	–	–	–	723	412	438	1.5	10.3
D01	510	8.8	615	–	*295*	*958*	*959*	*0.52*	*7.01*
D03	510	8.8	615	873	295	734	820	6.92	13.96
D04	700	13	985	–	295	865	920	4.43	8.91
D16	730	13	985	873	295	780	885	8.18	11.75
D09	900	17.4	1420	–	295	783	783	0.39	0.87
D21	940	17.4	1420	873	295	694	876	4.08	8.97
D12	1025	20.2	1710	–	295	701	701	0.35	0.35
D05	760	13	985	–	723	616	637	1.73	6.22
D08	930	17.4	1420	–	723	660	677	1.25	3.02
D20	1010	17.4	1420	873	723	562	562	0.61	2.7
D24	1075	20.2	1710	–	723	611	627	0.76	2.5

* Averaged temperature during the effective irradiation time period.

Tensile tests were performed on a 2 kN MTS mechanical testing machine equipped with a video-extensometer so that the displacement could be directly measured from the gauge section. The tests were mostly conducted at room temperature (295 K) and 723 K at a nominal strain rate of $1 \times 10^{-3}\,s^{-1}$.

In order to study the recovery of mechanical properties after annealing and helium (He) effect on hardening, some specimens were tested after annealing at 873 K for 2 h. The fracture surfaces of some specimens tested at 295 K were observed using the scanning electron microscopy (SEM) to identify the fracture mode.

3. Results

The results of tests performed at 295 and 723 K are presented in Fig. 1(a) and (b), respectively. Both figures include results of the irradiated specimens with and without (as-irradiated) annealing at 873 K. As shown in Fig. 1(a), the tests performed at 295 K indicate that the specimens at different displacement doses having great differences in deformation behavior, although all of them showing significant hardening as compared to the unirradiated specimen. The specimen of 8.8 dpa shows prompt necking after yielding, almost without any work hardening and uniform elongation, while the 13 dpa sample obviously displays a recovery of work hardening and uniform elongation. The other two specimens of higher displacement doses, 17.4 and 20.2 dpa, are completely different from the previous two specimens of lower displacement doses, showing a very brittle fracture without any plastic deformation.

The results of tests conducted at 723 K (Fig. 1(b)) are different from that of tests at 295 K. The main differences are in the strength and more important, in the ductility of the specimens of 17.4 and 20.2 dpa.

The tensile curves of the specimens after annealing at 873 K are also included in Fig. 1. Comparing to the stress – strain curves of the as-irradiated specimens, the results illustrate a decrease in yield stress and recovery of ductility after annealing at 873 K and tested at 295 K. The single annealed specimen tested at 723 K shows a decrease in yield stress but no recovery in ductility.

The values of yield stress (YS), ultimate tensile strength (UTS), strain-to-necking (STN) and total elongation (TE) of the specimens evaluated from the corresponding tensile stress – strain curves are listed in Table 1 as well.

The fracture surfaces of the specimens at different displacement doses tested at 295 K were observed using SEM. Fig. 2(a) presents a view of the fracture surface of the 8.8 dpa specimen, which indicates a fully ductile and transgranular fracture mode. Large dimples and small secondary cracks can be observed. With displacement dose increasing to 13 dpa, a numerous of secondary cracks can be observed on the fracture surface of the specimen (Fig. 2(b)). The appearance of the fracture surface is ductile in general (Fig. 2(b)), but the dimples are very shallow, which suggests a reduced

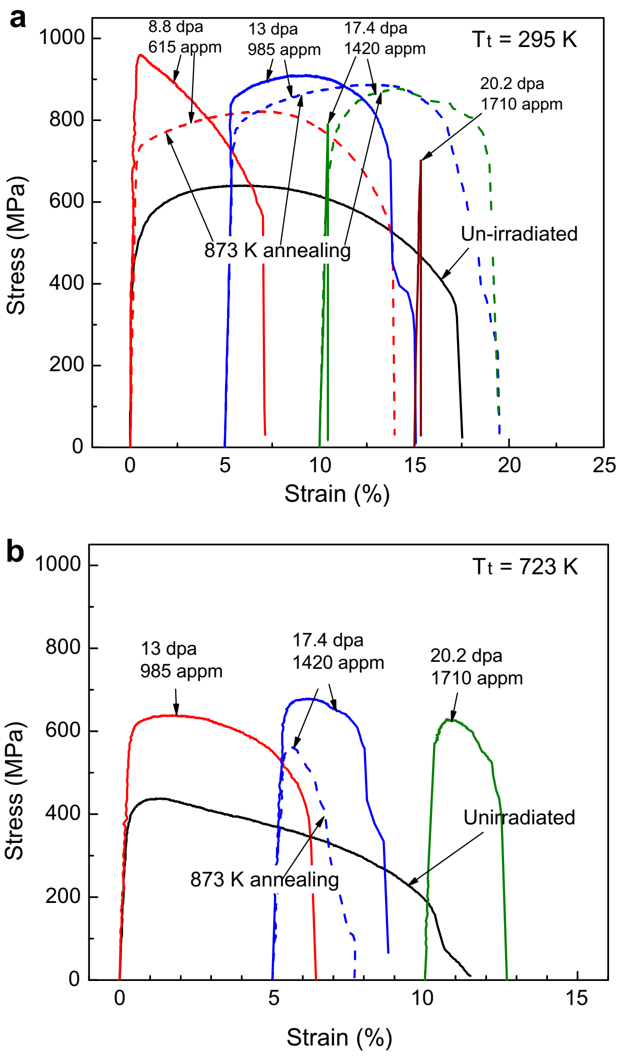

Fig. 1. Tensile stress – strain curves of the F82H specimens tested at (a) 295 K and (b) 723 K.

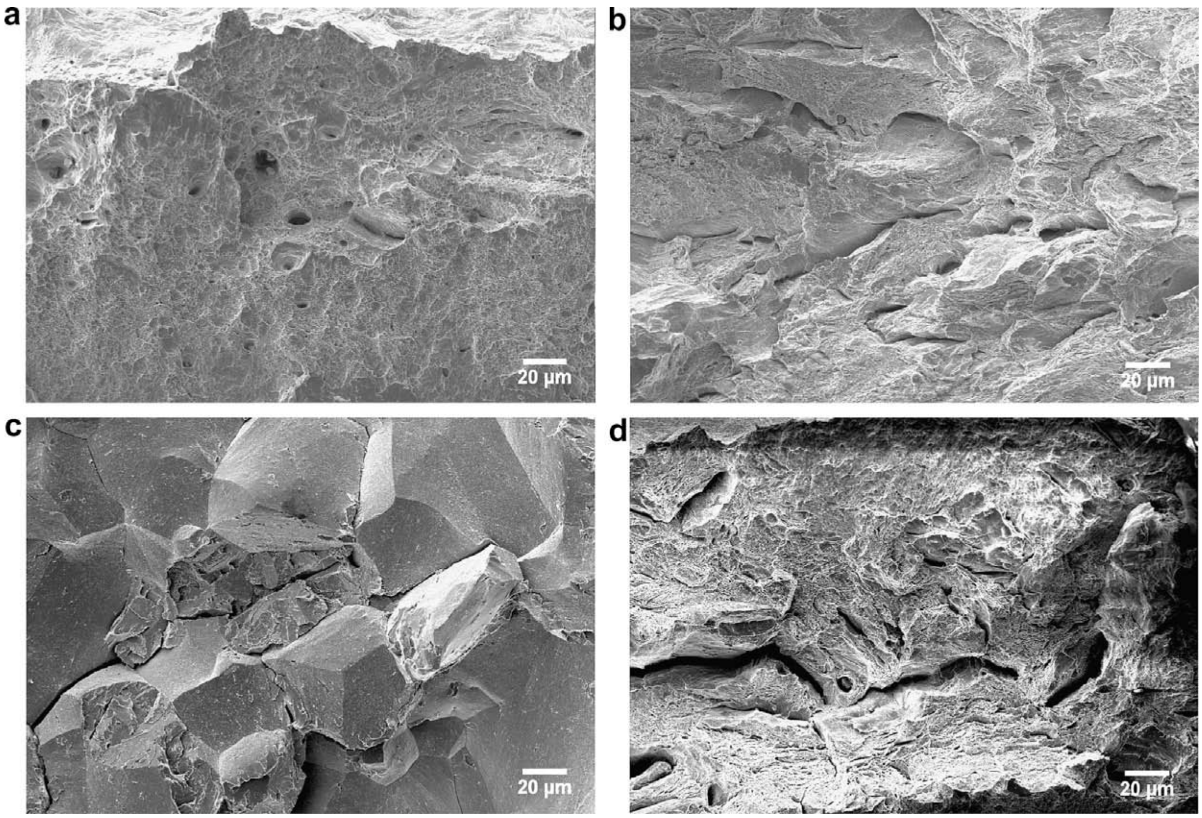

Fig. 2. SEM micrographs showing fracture surfaces of the F82H specimens at: (a) 8.8 dpa , (b)13 dpa, (c) 17.4 dpa, and (d) 8.8 dpa after annealing at 873 K for 2 h, after testing at 295 K.

ductility as compared to the previous case. In addition, some cleavage zones are also found. A complete change of fracture mode is observed in the specimen of 17.4 dpa (Fig. 2(c)). The fracture surface lies strictly perpendicular to the tensile axis and displays a fully brittle and mostly intergranular appearance. Fig. 2(d) shows the fracture surface of the annealed specimen of 13 dpa. No obvious difference between the as-irradiated specimen (Fig. 2(b)) and the annealed one (Fig. 2(d)) can be identified.

4. Discussion

4.1. Tensile properties of the specimens

Fig. 1 demonstrates that the different specimens have quite different behaviors, which can be essentially understood as a consequence of the different irradiation and testing conditions.

The reason for the difference between two specimens of lower displacement doses tested at 295 K can be attributed to the effect of irradiation temperature. In Table 1, one can see that the averaged irradiation temperatures of these two specimens were about 510 and 700 K. It is well known that FM steels demonstrate significant hardening and embrittlement, especially losing the uniform elongation, after irradiation in the low temperature regime ($<\sim$675 K), e.g. as those shown in [9,11,14]. The hardening can be attributed to the irradiation-induced defect clusters and dislocation loops [11], while the mechanism for losing uniform elongation is clear yet and might be understood as a result of localized deformation in specimens irradiated and tested at lower temperatures [15]. At higher temperatures $>\sim$723 K, usually the tensile properties of FM steels do not change much after neutron irradiation [9,16], which can be explained as no or very few of dislocation loops are formed [17]. In the case of spallation irradiation, it could be different due a very high He production rate. In the present case, the He contents in the specimens of 17.4 dpa and 20.2 dpa are very high, 1420 and 1710 appm, respectively. The He-induced ductile brittle transition temperature shift [18] is expected to be more than 500 K. Thus, a very brittle fracture of such thin specimens at 295 K is understandable, even considering a thin structure is favorable for ductile fracture. However, at 723 K, the deformation of these thin specimens should be no more in a brittle regime and the ductility can recover slightly.

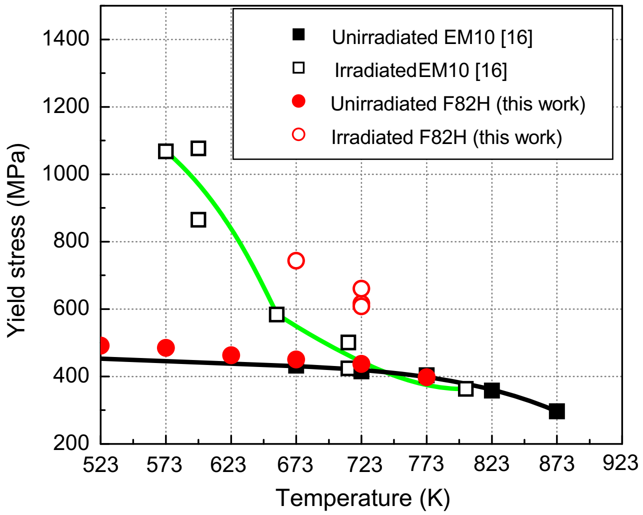

Fig. 3. A comparison of hardening in 8-9Cr martensitic steels after irradiation in the Phénix reactor under a fission neutron spectrum and in the sinq spallation target under a proton and neutron mixed spectrum.

4.2. Hardening effect of helium

As discussed above, the hardening effect of neutron irradiation in FM steels disappears at temperatures above around 723 K, as shown in Fig. 3 for the EM10 (9Cr–1Mo) steel irradiated in the Phénix reactor [16]. However, it is different for the F82H steel irradiated with proton and neutron mixture flux in SINQ targets. In Figs. 1 and 3 one can see that the irradiation hardening effect can be clearly observed from those specimens irradiated at $\geqslant$700 K and tested at 675 and 723 K. Microstructural investigations show that small He-bubbles of 1 – 2 nm in diameter with a high-density from 3 to $5 \times 10^{23}\ \mathrm{m}^{-3}$ were formed in the specimens, although dislocation loops were seldom observed [19]. It is, therefore, believed that the hardening of these specimens is mainly attributed to the high-density He-bubbles, because such small He-bubbles may produce a significant hardening [20].

The hardening effect of He-bubbles is also demonstrated by the results of the annealed specimens. In general, annealing of FM steels irradiated in low temperature regime can annihilate the defect clusters or dislocation loops induced by irradiation [21], and therefore, can reduce the irradiation-induced hardening and embrittlement effects. Kimura et al. [22] showed that the tensile properties of the JLM steels irradiated at 646 K to 10 dpa in a fission reactor could be fully recovered by annealing at 873 K. This suggests that annealing at 873 K can fully remove the dislocation loops produced by the neutron irradiation. However, the present results indicate that the annealing at 873 K has only limited recovery effect, even for the specimen irradiated at about 510 K. In Fig. 1 it can be seen that although the ductility recovers greatly, the hardening still remains at high level after annealing, in particular for those specimens irradiated at higher temperatures $\geqslant$710 K. Since the dislocation loops are annihilated by the annealing, the rest hardening can be essentially attributed to the He-bubbles left after annealing, which agrees with the results of the as-irradiated specimens.

An interesting point should be noted that the specimens irradiated at higher temperatures or after annealing at 873 K can persist not only significant hardening but also a great ductility as well. The deformation mechanisms in such a case are under studying and will be discussed elsewhere [23].

Despite of a large amount of hydrogen produced in the specimens, since the irradiation temperatures of these specimens were $\geqslant$525 K, the remaining hydrogen in the specimens should be very low [24]. The brittle fracture observed in the specimens of 17.4 and 20.2 dpa should not be related to the small amount of hydrogen content.

5. Conclusion

The F82H steel irradiation in STIP III (SINQ target-5) to displacement doses between 8.8 and 20.2 dpa has been performed at temperatures ranging from 510 to 1075 K. Tensile tests have been performed at 295 and 723 K in both as-irradiated and annealed conditions. The results demonstrate that

(a) Not only the specimens irradiated in the low temperature regime, but also those irradiated at higher temperatures above $\geqslant$710 K show significant hardening effect at both 295 and 723 K.
(b) After annealing at 873 K for the irradiated specimens still show considerable hardening which is normally not observed in FM steels after neutron irradiation at low temperatures and annealing at similar temperatures.
(c) The hardening observed in the specimens should be attributed to the hardening effect of high-density He-bubbles.
(d) The two specimens irradiated to 17.4 and 20.2 dpa broke in the elastic regime without any necking at 295 K, which is believed to be due to the He-induced embrittlement effect.

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