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The change of fracture toughness of martensitic steels after irradiation in SINQ target-3

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Abstract

Small bend specimens of martensitic steels T91, F82H and Optimax were irradiated in SINQ target-3 up to about 9 dpa in a temperature range of 100–250 °C. The single specimen method was used to determine the *J*-integral values. The specimens were tested in a temperature range from room temperature to 250 °C, mostly at irradiation temperatures. The results showed that the fracture toughness of the martensitic steels decreased with increasing irradiation dose. Specimens tested at the irradiation temperatures and 250 °C indicated that the fracture toughness of all three martensitic steels remained above 100 $\mathrm{MPa\,m^{1/2}}$. One T91 specimen irradiated to 4.3 dpa tested at room temperature was totally brittle and broke in the elastic deformation region. The fracture toughness value was only about 28 $\mathrm{MPa\,m^{1/2}}$, within the lower shelf of the fracture toughness. There was no obvious difference in the fracture toughness of the steels after irradiation.

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1. Introduction

Ferritic/martensitic (FM) steels are selected for the beam window materials of liquid metal spallation targets such as MEGAPIE [1] and those in the future accelerator driven systems (ADS). For these targets, the degradation of mechanical properties of the beam window materials under mixed proton/neutron spectrum irradiation is of great concern and not yet well understood. In particular, there is a strong need on fracture toughness data since such data are very limited for mixed spectrum irradiations. This work aims to measure fracture toughness of three kinds of FM steels; F82H, T91 and Optimax, irradiated in SINQ target-3 up to about 9 dpa. The fracture toughness is determined by conducting three points bending tests.

2. Experimental

2.1. Materials and specimen

Materials used in the present work are three kinds of FM steels F82H, T91 and Optimax. All are candidate materials for structural materials for either the first wall of fusion reactors or the liquid metal containers of spallation targets. The F82H steel (IEA Heat 9741) is a low activation martensitic steel for fusion materials study that was produced in Japan. The T91 (9Cr–1MoNbV mod., Heat 30716) steel was obtained from Oak Ridge National Laboratory, USA. The Optimax steel is also a low

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Table 1
Chemical composition of all materials (wt%)

Material	Cr	Ni	Mn	Mo	Ti	V	Si	P	Nb	W	Ta	C	Fe
F82H	7.65	0.02	0.49	0.003	0.004	0.19	0.07	0.003	0.002	1.98	0.03	0.09	Bal.
T91	8.32	0.09	0.48	0.86	0.001	0.2	0.15	0.012	0.06	<0.01	–	0.092	Bal.
Optimax	9.3	0.01	0.6	0.09	0.01	0.24	0.02	0.01	0.01	0.97	–	0.098	Bal.

activation martensitic steel which was developed by the CRPP fusion material group in EPFL, Switzerland. The compositions of the three steels are listed in Table 1.

Due to the limited irradiation volume in the SINQ target, small 3-point bending specimens with a size of 2 × 4 × 20 mm were used, as shown in Fig. 1. The specimens were fatigue pre-cracked before irradiation to a ratio of crack length to specimen width (a/W) of about 0.5 [2].

2.2. Irradiation condition

In the first SINQ target irradiation program (STIP-I), specimens of different kinds of materials and various sizes were packed in 10 rods. The position of these special rods was in the central columns of the target. Detailed information of STIP-I can be found in [2]. Bend samples of F82H, T91 and Optimax steels were included in 3 rods, so a series of irradiation dose and temperatures were achieved. Table 2 gives the irradiation temperatures, irradiation doses and helium contents of the samples. The irradiation dose and helium concentrations were calculated with the MCNPX code. The helium concentrations were further corrected based on the measurements performed on some samples in the same irradiation [3]. The hydrogen content of the samples was also calculated. However, the measurements showed that the hydrogen content decreased rapidly with increasing irradiation temperature leading to a large discrepancy from the calculated

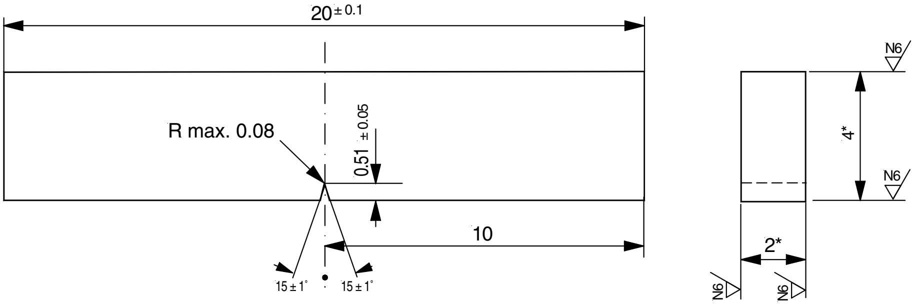

Fig. 1. Specimen geometry of the bend bar samples.

Table 2
Irradiation conditions of bending samples

Materials	Specimen no.	Irrdiation temperature (°Cs)[a]	dpa	He (appm)	Test temperature (°C)
T91	I1	220/260	9.1	532	250
	I4	170/200	7.1	398	250
	I5	95/115	4.3	223	250
	I6	95/115	4.3	223	RT
F82H	P62	220/260	9.1	532	250
	P63	170/200	7.1	398	170
	P65	95/115	4.3	223	100
Optimax	N41	210/245	8.4	460	250
	N43	94/110	4.0	195	100

[a] The irradiation temperature range in the irradiation period of two years.

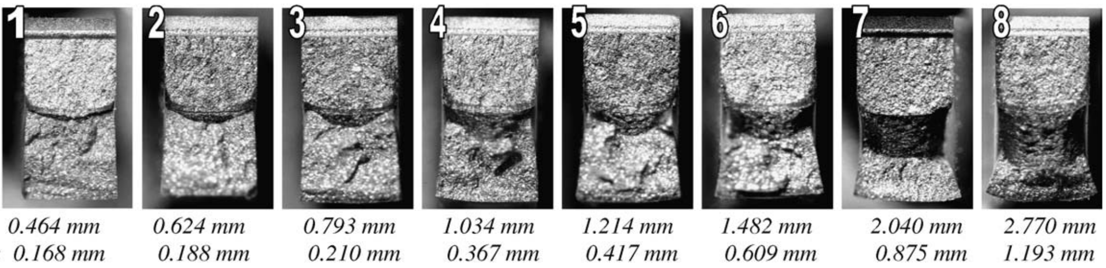

Fig. 2. Crack growth in eight T91 specimens loaded to different displacements. The heat treatment of the specimens is: normalizing at 1050 °C for 2 h, and tempering at 650 °C for 2 h. The bending tests were performed at 25 °C. The values of displacement (Δl) and averaged crack growth (Δa) are indicated in the figure.

values. Therefore, the hydrogen concentration is not given in the table.

2.3. *J*-integral testing

Three-point bending tests were conducted in accordance with ASTM standard E1820–01 [4]. The specimens were tested using an MTS 2 kN machine installed in a semi-hot-cell. Video-extensometer was used to measure the bending displacement. The single-specimen technique using loading–unloading compliance method was applied to determine the *J*-integral resistance (*J*–*R*) curves and *J*-values.

The difficulty associated with application of the single-sample technique is the determination of the crack length of the samples, which is normally assessed using the multi-sample technique. This is particularly true for irradiated samples because the relationship of crack length and bending displacement is different for samples in different irradiation and testing conditions. This issue is approached using T91 un-irradiated samples with different tempering temperatures. It is known that when FM steels are tempered at lower temperatures, they are harder and more brittle, which is similar to the hardening and embrittlement effects induced by irradiation. Therefore, in the present work, un-irradiated T91 steel samples were tempered at 500, 550, 600, 650, 700 and 750 °C to simulate different irradiation conditions.

The relationship of crack-length and bending displacement was determined using the multi-sample technique. A group of samples with the same heat treatment condition were loaded to different displacements, then heat tinted at 300 °C for 30 min and finally broken in liquid nitrogen. An optical microscope was used to determine the initial pre-crack and final crack lengths. Fig. 2 shows an example of the crack length measurement of eight T91 specimens which were tempered at 650 °C for 2 h and tested at 25 °C. The relatively dark area in the middle part of each specimen was produced by the crack propagation during bending test. Samples with different tempering temperatures, 500, 550, 600, 650, 700 and 750 °C, were tested. The relationship between the bending displacement and measured crack growth is shown in Fig. 3.

Tests of the irradiated and un-irradiated samples were performed in a temperature range from room temperature to 250 °C in argon gas. The *J* versus crack growth behaviour was approximated with a best-fit power law relationship. The blunting line was calculated from the material flow properties. The intersection of the power law fit and the 0.2 mm offset line parallel to the blunting line defined J_Q. These J_Q values were converted into equivalent stress intensity K_{JQ} values with the equation

$$K_{JQ} = \sqrt{EJ_Q},$$

where E is Young's modulus.

3. Results and discussion

Before irradiation, T91, F82H and Optimax steels have *J*-values around 310–340 kJ/mm^2. In the test temperature range of 25–250 °C, the *J*-values decrease slightly with increasing test temperature, and give a value about 250–280 kJ/mm^2 for all three materials at 250 °C. Fig. 4 shows the load–displacement curves of irradiated and un-irradiated specimens of T91 steels tested at 250 °C. Due to irradiation hardening and embrittlement, the yielding level increases and the failure occurs by crack propagation, both occurring faster with increasing irradiation dose.

Fig. 5 presents the load–displacement curves of two irradiated T91 specimens of 4.3 and 9.1 dpa

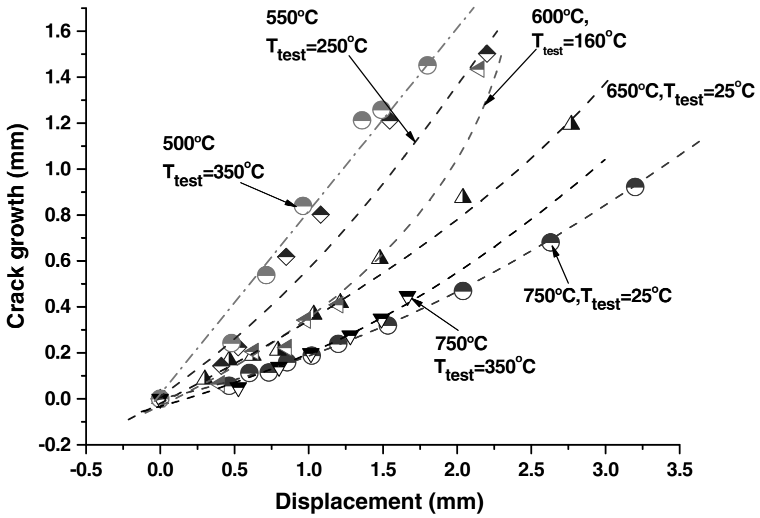

Fig. 3. The relationship between the bending displacement and crack growth of the T91 specimens in different tempering and testing conditions.

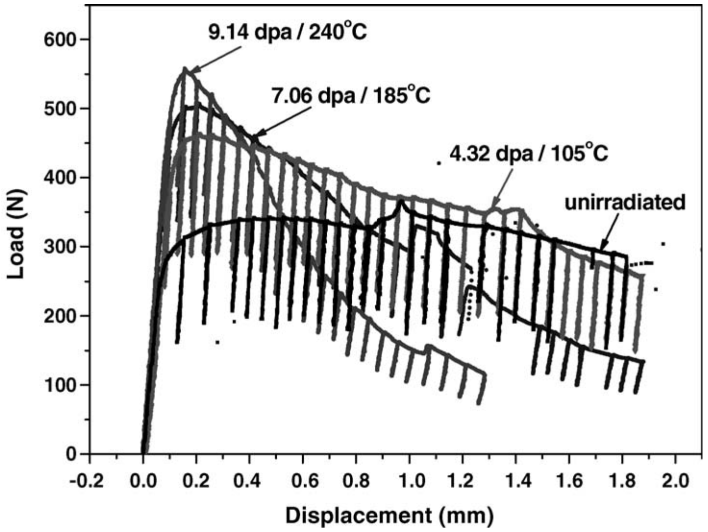

Fig. 4. The load–displacement curves of both irradiated and un-irradiated specimens of T91 steel tested at 250 °C.

tested at 250 °C, and two un-irradiated specimens, one tempered at 650 °C and tested at room temperature and the other tempered at 550 °C and tested at 250 °C. One can see that the curve of the 4.3 dpa specimen is similar to that of 650 °C tempered specimen, and the curve of the 9.1 dpa specimen is similar to that of 550 °C tempered specimens, although the lower-temperature tempered specimens are stronger. In this case, it is believed that the crack propagation in the irradiated specimens should be similar to that in the corresponding tempered specimens. Therefore, the crack growth of the irradiated specimens was derived from Fig. 3. The same methodology was applied to other irradiated specimens for evaluating the crack length values. Due to the high radioactivity of the specimens, it was difficult to measure the crack lengths of the tested irradiated specimens in a hot cell in order to validate the results of such evaluation. Work is underway to develop a SEM-replica technique to measure the crack length, and furthermore to examine the fracture surface of the tested irradiated specimen.

Fig. 6 shows the measured fracture toughness data of the three steels before and after irradiation.

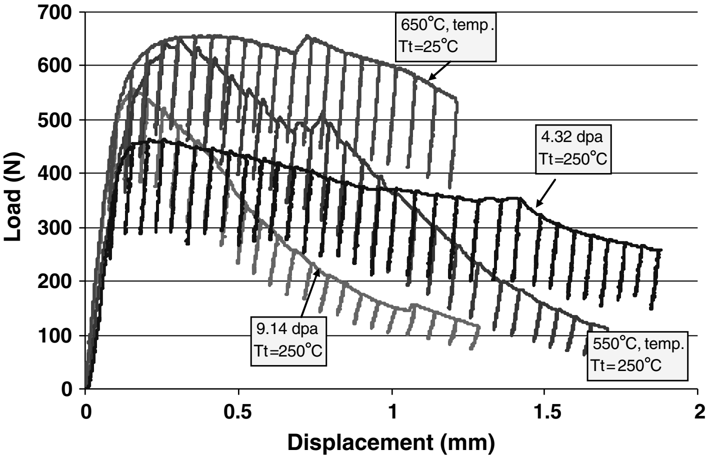

Fig. 5. Comparison between the load–displacement curves of irradiated and un-irradiated T91 specimens tempered at lower temperatures.

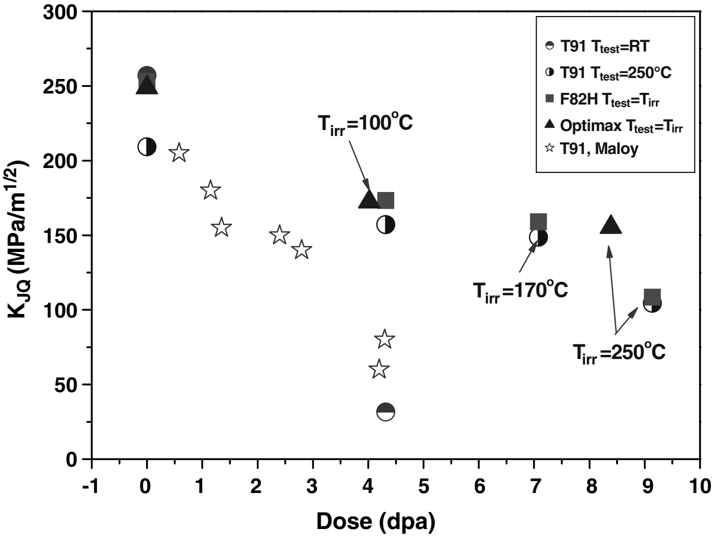

Fig. 6. Fracture toughness of the three FM steels after irradiation in SINQ target-3. The data of T91 steel irradiated in LANSCE with 800 MeV protons and spallation neutrons at ⩽160 °C [8] are included for comparison.

The result shows that in the irradiation temperature range of 100–250 °C the fracture toughness of martensitic steels decreases with increasing irradiation dose. At room temperature, after irradiation to about 4.3 dpa, the T91 specimen was totally brittle and broke in the linear elastic part of test curve. The fracture toughness value was only about 28 MPa m$^{1/2}$, within the lower-shelf region. Except for this specimen, all the other specimens show that the fracture toughness of the three FM steels still remains above 100 MPa m$^{1/2}$ after testing at irradiation temperatures ⩾100 °C (250 °C for the T91 samples).

There are few data available for comparison with the present results. Maloy et al. has reported fracture toughness of T91 steels irradiated at LANSCE with 800 MeV protons up to about 4.3 dpa at 50–160 °C [5]. As shown in Fig. 6, the fracture toughness of LANSCE irradiated specimens is lower than that of present STIP specimens. The reason might be the lower irradiation and testing temperatures and higher helium contents (about a factor of two at similar doses) of the LANSCE specimens.

One of the main objectives of the STIP experiments is to study helium and hydrogen effects on mechanical properties of structural materials.

However, the data obtained in the present work is too limited to make any confident conclusions of helium and hydrogen effects on the degradation of fracture toughness of the three FM steels. Samples irradiated in STIP-II to higher doses and higher helium and hydrogen concentrations will be tested to extend this study.

4. Conclusions

F82H and T91 steels were irradiated in SINQ target-3 to doses up to 9.1 dpa in a temperature range of 100–250 °C. The irradiation reduced the fracture toughness of these martensitic steels. At room temperature, a T91 specimen broke in brittle fracture mode after irradiation to about 4.3 dpa. The fracture toughness value was about 28 $\mathrm{MPa\,m^{1/2}}$, within the lower-shelf of the fracture toughness. However, all other specimens tested at $\geqslant$100 °C showed that the fracture toughness of the steels remained above 100 $\mathrm{MPa\,m^{1/2}}$. There was no obvious difference in the fracture toughness of the three steels after irradiation.

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