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Dependence of impact properties on irradiation temperature in reduced-activation martensitic steels

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Abstract

Ductile–brittle transition (DBT) behavior of 9%Cr-2%W reduced-activation martensitic (RAM) steels has been investigated following neutron irradiation in the fast flux test facility, materials open test facility (FFTF/MOTA) at different temperatures. Both the irradiations at 663 and 733 K cause an increase in DBT temperature, while the irradiation at 663 K induces the hardening and the softening at 733 K. Microstructural observation by transmission electron microscope (TEM) revealed that small dislocation loops existed in the specimen irradiated at 663 K and no such a loop, but relatively large M_6C carbides and Laves phase were formed by the irradiation at 733 K. There appears to be a linear dependence between ΔDBTT and $\Delta\sigma_Y$ in neutron irradiated RAM steels when irradiation induces the hardening. Irradiation embrittlement accompanied by the softening is considered to be due to reduction of cleavage fracture stress caused by the irradiation-induced recovery of the martensitic structure, namely decrease in dislocation density and formation of large precipitates.

1. Introduction

It is well known that neutron irradiation hardening of 9%Cr-2%W RAM steels [1–5] is generally much smaller than that of austenitic steels [6–8] and vanadium alloys [9–11]. The martensitic structure of reduced-activation ferritic steels, which consists of a number of dislocations, sub-grain boundaries and fine carbides, is considered to be adequate to fusion structural materials, since it is expected that those microstructures provide a number of trapping sites for point defects and suppress the growth of defect clusters, resulting in a higher resistance to the irradiation. Previous work [12] clearly showed that neutron irradiation hardening of 9Cr–2W martensitic steels was mainly dependent on the irradiation temperature at fluences above 10 dpa, and the FFTF irradiation induced hardening only below 700 K and the irradiation effect turned to softening above it.

Irradiation embrittlement has been often correlated to irradiation hardening [13,14] in light water pressure vessel steels (LW–PVS), where the irradiation-induced increase in yield stress ($\Delta\sigma_Y$) appears to be proportional to the increase in ductile-brittle transition temperature (ΔDBTT), and this correlation is very useful to estimate the irradiation embrittlement of the steels. It is also required to investigate the $\Delta\sigma_Y$-ΔDBTT relationship of RAM steels for fusion application, since the irradiation condition of RAM steel, such as temperature and dose, is expected to be quite different from LW-PVS. This is especially true when we consider that the operation temperature of RAM steels for fusion applications is in the range between 573 and 823 K where the irradiation induces both hardening and softening.

In this research, impact properties of RAM steels have been investigated following neutron irradiation at different irradiation temperatures in the FFTF and JMTR (Japanese Material Test Reactor) in order to make clear the difference in the mechanism of irradiation embrittlement in the steels irradiated at different temperatures.

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2. Experimental procedure

The materials used are low activation 9Cr–2W single phase martensitic steels with a small change in the minor alloying elements; JLM-0 and JLM-1 contain no boron and 30wt.ppm B, respectively. Miniaturized tensile (4 mm × 16 ml × 0.25 mt) and Charpy V-notched (CVN) impact (1.5 mm × 1.5 mm × 20 ml) specimens and disk shape (3 mϕ × 0.25 mt) specimens for microstructural observation by TEM were fabricated. Neutron irradiations were performed in the FFTF/MOTA-2A at 663 K (24 dpa) and 733 K (22 dpa) and JMTR at 333 K (0.0063 dpa). Impact tests were carried out in an instrumented drop tower type impact testing machine in Oarai Brunch of Institute for Materials Research, Tohoku University. A specimen was set in a cold bath filled with the appropriate solution, such as ethanol or isopentane, which was cooled by liquid nitrogen and held at a desired constant temperature for 15 min before testing. After impact testing, fracture surfaces were observed by scanning electron microscope (SEM). Tensile tests were done at a cross head speed of 0.2 mm/min at room temperature. The TEM disk specimens were jet thinned and microstructure examinations were performed with JEM-1200EX and 2000FX which were equipped with energy dispersive X-ray spectrometers (EDS).

In order to make clear the effects of thermal aging, those tests were also carried out for thermal control specimens which were prepared by aging specimens at exactly the same operating conditions, such as temperature and time period, with the FFTF/MOTA-2A irradiation.

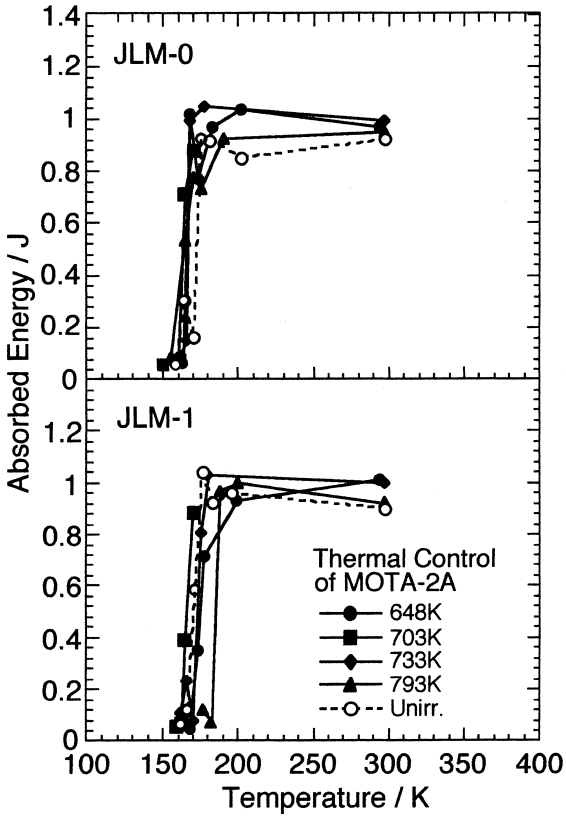

Fig. 1. Ductile–brittle transition behavior of the control specimens for FFTF/MOTA-2A irradiations. The specimens were aged at exactly the same conditions of the thermal history of MOTA-2A. The DBT curves of control specimen at each temperature is very similar to that of unirradiated specimens.

3. Results and discussion

3.1. Impact properties

Ductile-brittle transition (DBT) behavior of the thermal control specimens of JLM-0 and JLM-1 steel is shown in Fig. 1, indicating that in both the steels the DBT behavior is almost the same among unirradiated and thermal control specimens aged at temperatures between 648 and 733 K. This indicates that there is no effect of thermal aging on the impact properties of RAM steels at those temperatures. Fig. 2 shows the effect of FFTF/MOTA irradiations at 663 K (22 dpa) and 733 K (24 dpa) on the DBT behavior of the steels. The ΔDBTT depends on irradiation temperature; in both the steels the irradiation at 663 K induces much larger ΔDBTT (30–40 K) than the irradiation at 733 K (15 K), while the absorbed energy at room temperature is not influenced by the irradiations. The JLM-1 steel, which contains 30 wtppm of boron, shows smaller ΔDBTT than JLM-0 after the irradiation at 663 K, suggesting that helium transmuted from natural boron, which is estimated to be about 8 atppm [15,16], does not enhance irradiation embrittlement but even suppresses it. Observation of brittle fracture surfaces by SEM revealed that all the specimen showed a cleavage fracture mode, and there was no significant difference among those of thermal control specimens and both the specimens irradiated at 663 and 733 K.

Although irradiation dose is much smaller for the specimen irradiated in JMTR, namely 0.0063 dpa, relatively large ΔDBTTs are observed for JLM-6 and JLM-9 steel irradiated in a leaky capsule which allows specimens to be irradiated at coolant temperature, about 333 K, as shown in Fig. 3.

Dependence of ΔDBTT of RAM steels on irradiation temperature is shown in Fig. 4 with the data of previous works on 9Cr–1Mo steel [17]. The bracketed numbers indicate the dose in dpa. The ΔDBTTs of RAM steels are smaller than those of 9Cr–1Mo steels at all the temperatures, suggesting that the former is more resistant to irradiation embrittlement than the later. In the temperature range shown in the figure, irradiation

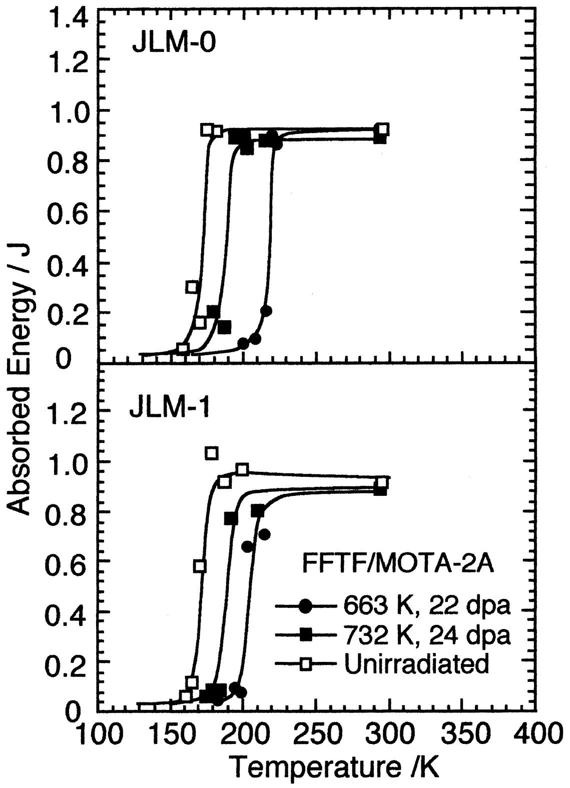

Fig. 2. Effects of FFTF/MOTA irradiation on the DBT behavior of JLM steels.

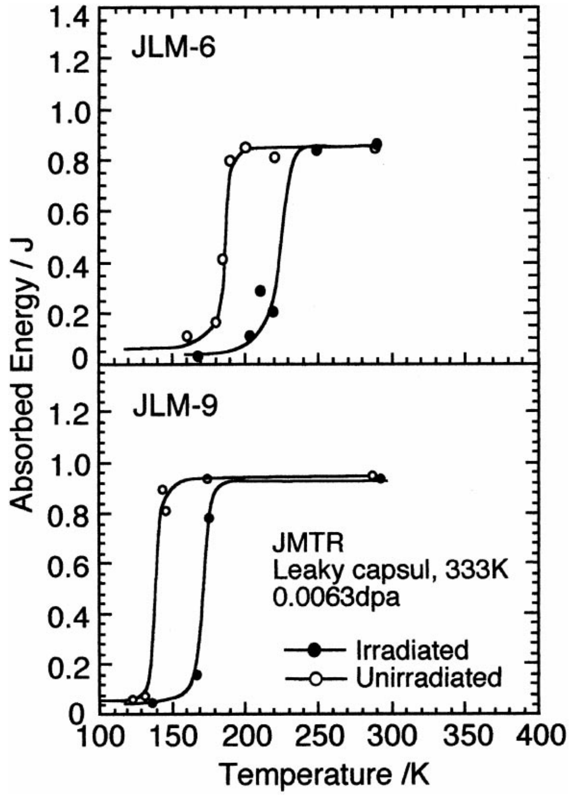

Fig. 3. Effects of JMTR irradiation at coolant temperature, 333 K, on the DBT behavior of JLM steels. Relatively large shift in the DBTT was induced by the irradiation up to only 0.0063 dpa.

always induces an increase in DBTT and the ΔDBTT appears to increase with decreasing irradiation temperature.

3.2. Correlation between irradiation hardening and embrittlement

Previous work clearly showed that FFTF/MOTA irradiation induced hardening and softening below and above 700 K, respectively, and the hardening increased with decreasing temperature, while the softening saturated above 723 K [12]. Fig. 5 shows the relation between ΔDBTT and $\Delta\sigma_Y$ of RAM steels following neutron irradiation together with that of 9Cr–1Mo steel for comparison. It appears that there is a linear dependence between those two when hardening is observed except for data point (b). It is worth to note that almost the same relationship is observed for these two steels irradiated with neutrons of different energy spectra. This suggests that the hardening mechanism is also applicable to the irradiation embrittlement of RAM steels, and the amount of irradiation hardening is considered to be a good monitor of irradiation embrittlement. The data point (b) is far from the linear relationship, indicating that a relatively small embrittlement accompanied by a huge hardening were induced by the irradiation. Since the irradiation hardening or embrittlement is strongly dependent on the irradiation temperature especially around 700 K, the irradiation effects at these temperatures should be investigated in care of the irradiation temperature. Non-hardening embrittlement can be accompanied by the change in the fracture mode. One of authors reported [18,19] that the fracture mode of high manganese reduced-activation ferritic steels was changed from cleavage fracture to intergranular fracture by FFTF/MOTA irradiation at 638 K, which was due to reduction of intergranular fracture stress to below that of cleavage fracture. Since the shift in DBTT by the irradiation at 733 K in this work, which is accompanied by irradiation softening, does not cause any changes in the fracture mode, the shift in DBTT is considered to be due to reduction of cleavage fracture stress. Microstructural observation of the JLM-1 steel (Fig. 6) revealed that a number of dislocation loops and small $M_{23}C_6$ carbides exist in the specimen irradiated at 663 K, while no such a structure but rather large precipitates

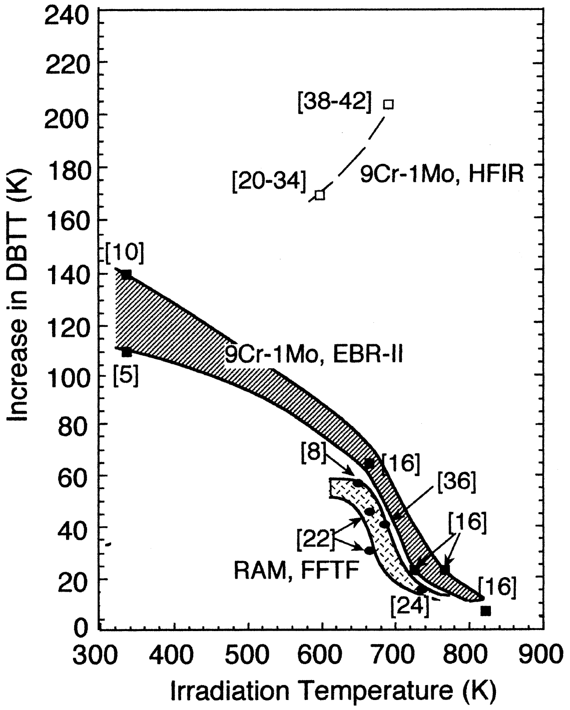

Fig. 4. Dependence of irradiation temperature on the shift in DBTT in ferritic steels. The bracketed numbers indicate the dose in dpa.

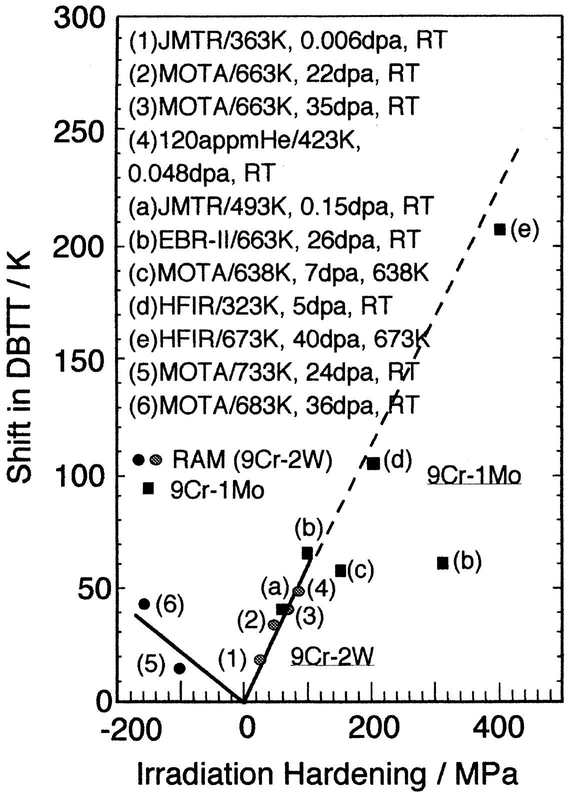

Fig. 5. Relation between irradiation hardening and shift in DBTT in neutron-irradiated JLM steels; MOTA/673 K, 40 dpa, 453 K means that the specimen was irradiated in the FFTF/MOTA at 673 K up to 40 dpa and tensile tested at 453 K.

are observed in the specimen irradiated at 733 K. Examples of EDS spectra obtained for JLM-1 steel following irradiation at both the temperatures are shown in the bottom of Fig. 7. At 663 K, chromium-rich $M_{23}C_6$ carbides were mainly observed, while at 733 K, tantalum-rich M_6C carbides and tungsten-rich Fe_2W Laves phase are also observed besides $M_{23}C_6$ carbides. The martensitic structure was partly recovered by the irradiation, showing the reduction of dislocation density and coarsening of $M_{23}C_6$ carbides. Since no such structural change, namely, a significant reduction of dislocation density and formation of M_6C carbides and Laves phase, was observed in the thermal control specimen at 663 K, it is very likely that the irradiation-induced recovery of martensitic structure accompanied by the precipitation of M_6C carbides and Laves phase is detrimental to the cleavage fracture toughness, and they causes an increase in DBTT accompanied by irradiation softening.

4. Conclusion

Dependence of ΔDBTT on the irradiation temperature has been investigated for the JLM series of steels following FFTF/MOTA-2A and JMTR irradiations. The main results are;

(1) The ΔDBTT of the RAM steels is 40 K at most after the irradiation at 663 K up to 22 dpa, which is much smaller than that of 9Cr–1Mo steel (70 K, < 10 dpa). The neutron irradiation also induced an increase in the DBTT following the irradiation at 733 K which was accompanied by irradiation softening.

(2) There was no significant difference among the brittle fracture mode, which was cleavage of thermal control specimens and the irradiated specimens at 663 and 733 K. Microstructural observation revealed that a number of dislocation loops and small $M_{23}C_6$ carbides existed in the specimens irradiated at 663 K, while at 733 K, no dislocation loops appeared, but irradiation-induced recovery of martensitic structure occurred, which was accompanied by the formation of relatively large M_6C carbides and Laves phase.

(3) It appears that the ΔDBTT is proportional to $\Delta\sigma_Y$ when irradiation induced the hardening, suggesting that the hardening mechanism is applicable after neutron irradiation below 700 K. However, the mechanism of irradiation embrittlement after irradiation above 700 K, where irradiation induced softening, is completely different from the hardening mechanism and could be

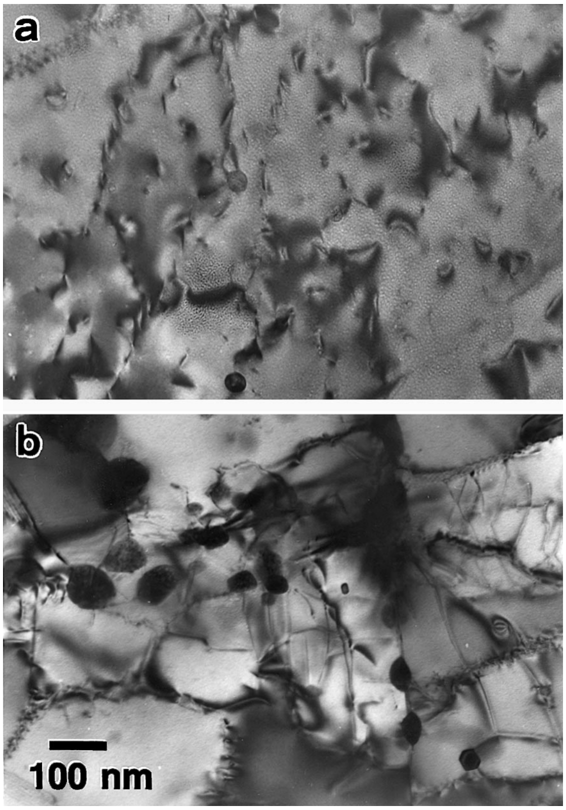

Fig. 6. Microstrucure of JLM-1 steel following FFTF/MOTA irradiation at: (a) 663 K and (b) 733 K.

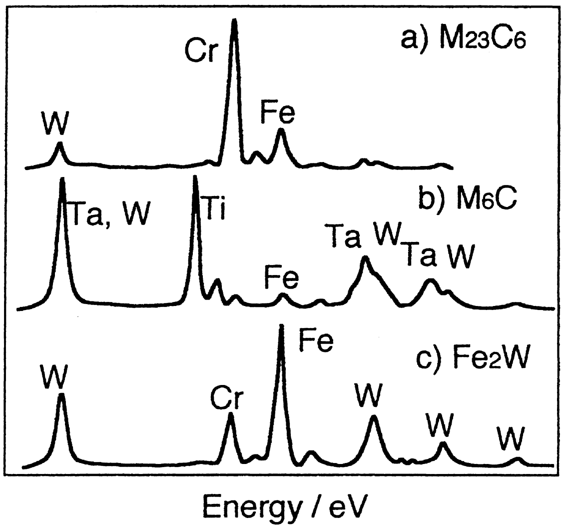

Fig. 7. Examples of EDS spectra obtained for the JLM-1 steel irradiated in the FFTF/MOTA at 733 K. The spectra (a) was the only one observed in the steel irradiated at 663 K.

attributed for the irradiation-induced recovery of the martensitic structure accompanied by the reduction of dislocation density and formation of relatively large M_6C carbides and Laves phase.

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