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Irradiation hardening and embrittlement in high-Cr oxide dispersion strengthened steels

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ABSTRACT

The effects of neutron irradiation on the mechanical properties of high-Cr oxide dispersion strengthened (ODS) ferritic steels were observed. The materials used were produced by varying the Cr content from 14 to 22 wt% while maintaining the yittria contents within the range of 0.36–0.38 wt%. The ODS steels were irradiated in JMTR at 300, 420 and 550 °C to 5.56×10^{20} n/cm^2 (>1 MeV). Charpy impact tests were performed between −150 °C and room temperature. The upper shelf energy (USE) of all ODS steel samples were reduced after irradiation. However, the reduction became smaller as the irradiation temperature increased. When the ODS steels were irradiated at 300 and 420 °C, the ductile-to-brittle transition temperature increased significantly. Tensile tests of the ODS steels irradiated at 300 °C were performed at room temperature with a strain rate of 6.7×10^{-4} s^{-1}. The yield stress and tensile strength of the irradiated ODS alloys increased significantly, and the level of irradiation hardening increased with the Cr content. Anisotropy was not observed in the yield stress and tensile strength but was observed in the elongation.

1. Introduction

Oxide dispersion-strengthened (ODS) ferritic/martensitic steels have been developed as a fuel cladding material for sodium-cooled fast breeder reactors (SFRs) [1,2]. Due to the dispersion of oxide particles, the ODS steels show high-strength at high temperatures [3]. In terms of the irradiation effects on the mechanical properties, recent irradiation experiments clearly showed that the ODS steels are highly resistant to irradiation embrittlement at temperatures between 300 and 500 °C up to 15 dpa [4]. The ODS steels developed as SFR fuel cladding material contain at most 12% chromium. It is well known that the level of corrosion resistance in high-temperature water diminishes significantly as the chromium concentration decreases to less than 13% [5]. High-Cr ODS steels are noted for their improved corrosion resistance properties in super critical pressurized water (SCPW) [5,6]. However, irradiation embrittlement caused by Fe–Cr phase decomposition under neutron irradiation can be a critical issue for high-Cr steels. The objective of this study is to investigate the effect of neutron irradiation on the mechanical properties of the high-Cr ferritic ODS steels.

2. Experimental procedure

The materials used were five types of ODS steels (K1–K5) that were produced by varying the Cr content from 14 to 22 wt% while maintaining the yittria content in the range of 0.36–0.38 wt%. The main chemical compositions of the K1, K2, K3, K4 and K5 steels are 19Cr, 14Cr–4Al, 16Cr–4Al, 19Cr–4Al and 22Cr–4Al, respectively. The chemical composition of each type of steel is summarized in detail in Table 1. All specimens were irradiated with fast neutron fluence of 5.56×10^{20} n/cm^2 (>1 MeV) in the Japan Materials Testing Reactor (JMTR) at temperatures of 300, 420 and 550 °C. The total irradiation time was approximately 2600 h.

Charpy impact tests were performed using sub-size Charpy v-notch specimens. The dimensions of the sub-size specimens were $1.5 \times 1.5 \times 20$ mm^3, including a 0.3 mm V-notch with a 30° angle. All of the specimens were tested at temperatures ranging from 120 K to room temperature using a specially designed electrically controlled hydraulic machine for instrumented Charpy tests.

Tensile tests were performed using SS-J miniature specimens. The gauge section of the SS-J miniature specimens was $1.2 \times 5.0 \times 0.25$ mm^3. As they are manufactured by extrusion, ODS steels show anisotropy in their mechanical properties because the grains are heavily elongated as a result of the extrusion process. In order to investigate the anisotropy of the tensile properties, specimens were cut out from the extruded bars in the radial and longitudinal directions, as shown in Fig. 1.

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Table 1
The chemical composition of the ODS steels used in this study.

I.D	C	Cr	W	Al	Ti	N	Y	Y_2O_3
K1	0.05	18.37	0.29	<0.01	0.28	0.014	0.29	0.368
K2	0.04	13.64	1.65	4.12	0.28	0.009	0.30	0.381
K3	0.08	16.00	1.82	4.59	0.28	0.006	0.29	0.368
K4	0.09	18.85	1.83	4.61	0.28	0.005	0.29	0.368
K5	0.10	22.05	1.80	4.55	0.27	0.005	0.28	0.356

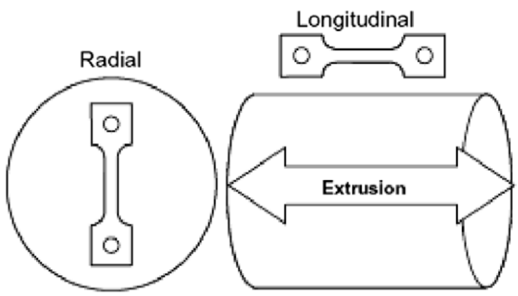

Fig. 1. Cutting scheme of the specimens used in tensile tests.

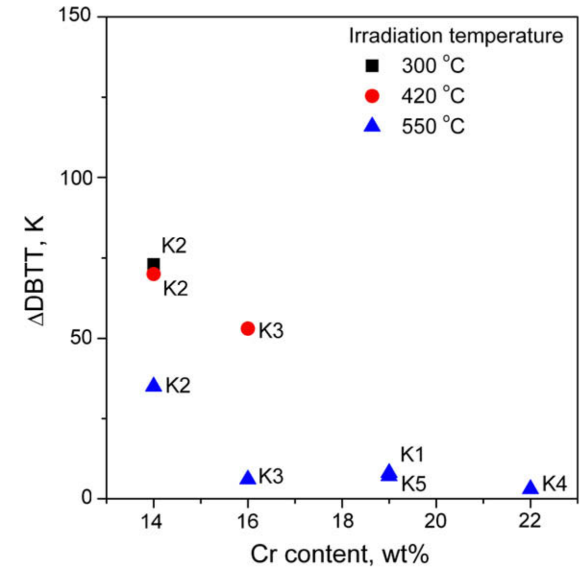

Fig. 3. The change in the DBTT as a function of the Cr content for irradiated ODS steels.

3. Results and discussion

3.1. Ductile-to-brittle transition behavior

Charpy impact energy curves of 14Cr–4Al (K2) ODS steel as a function of the test temperature after irradiation at 300, 420, 550 °C are shown in Fig. 2. The upper shelf energies (USE) of the irradiated samples were lower than these values of unirradiated samples, and the ductile–brittle transition temperatures (DBTT) were higher. For the samples irradiated at 300 °C, USE was reduced and DBTT increased significantly compared with unirradiated samples; however, the USE reduction and the increase in the DBTT became smaller when the irradiation temperature increased. The ΔDBTT of irradiated ODS steels with various Cr contents are shown in Fig. 3. For the 14Cr–4Al ODS steel (K2), the ΔDBTT increased by 73, 70 and 35 K at irradiation temperatures of 300, 420 and 550 °C, respectively. The ΔDBTT for irradiation at 550 °C was 50% less than it was for ΔDBTT at 300 °C. No differences were shown for irradiation temperatures between 300 and 420 °C. Additionally, the

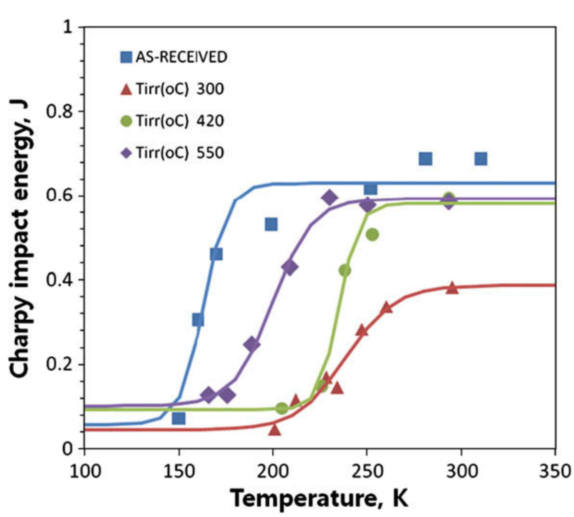

Fig. 2. Effect of the irradiation temperature on the Charpy impact energy of the ODS steels as a function of the test temperature for irradiated 14Cr–4Al (K2) ODS steel.

ΔDBTT in 16Cr–4Al ODS steel (K3), when irradiated at 420 °C, increased significantly. When the Cr content is greater than 16 wt% in the ODS steels, USE was not observed at irradiation temperatures of 300 and 420 °C; hence, it appears that the DBTT of ODS steels with Cr contents greater than 16 wt% are above room temperature. This suggests that neutron irradiation significantly affects the DBTT of ODS steels below irradiation temperatures of 420 °C. Although the DBTT was observed in all specimens at an irradiation temperature of 550 °C, changes in the DBTT did not depend on the Cr content, with the exception of the 14Cr–4Al ODS steel (K2). In the case of K2, ΔDBTT was very large because the DBTT of unirradiated samples was much lower than that of the other ODS steels. Thus, if the DBTT of K2 is excluded, the change in the DBTT appears to be only scarcely affected by neutron irradiation at an irradiation temperature of 550 °C.

3.2. Irradiation hardening

A tensile test was performed to observe the irradiation-induced changes in the tensile properties. The specimens irradiated at 300 °C and up to 5.56×10^{20} n/cm^2 (>1 MeV) were used in tensile test. The tensile test was performed at room temperature at a strain rate of 6.7×10^{-4} s^{-1}.

Irradiation-induced hardening was evaluated by comparing the tensile properties of irradiated specimens with those of the as-received specimens. Fig. 4 shows the changes in the tensile properties (yield stress, UTS and elongation) of the K2, K3, K4 and K5 samples, which have various Cr contents and an Al content of 4 wt%. The hardening increased as the Cr content increased such that the change in the yield stress and UTS increased as the Cr content increased. However, elongation changes were independent of the Cr content. In TEM observations of irradiated ODS steels, it was reported that irradiation hardening is caused by the formation of dislocation loops and phase precipitates [7]. The dislocation loops are formed by neutron irradiation and the formation of α' phase precipitates is affected by the Cr content and by aging during irradiation. Therefore, it is thought that the increase in the degree of irradiation hardening as the Cr content increases results from the formation of the α' phase precipitates. However, in unirradiated ODS steels, it is known that the formation of α' phase precipitates does not appear at 300 °C. This suggests that neutron irradiation promotes the formation of the α' phase.

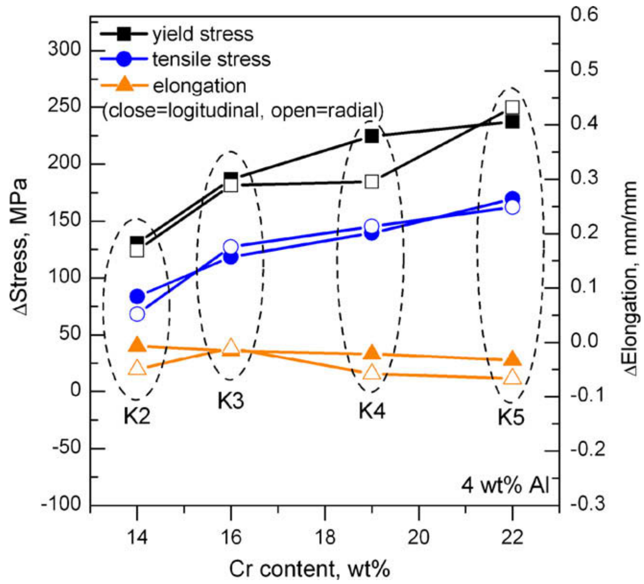

Fig. 4. Change in the yield stress, ultimate tensile strength and elongation versus the Cr concentration after irradiation. Tensile tests were performed at room temperature at a strain rate of $6.7 \times 10^{-4}\ s^{-1}$.

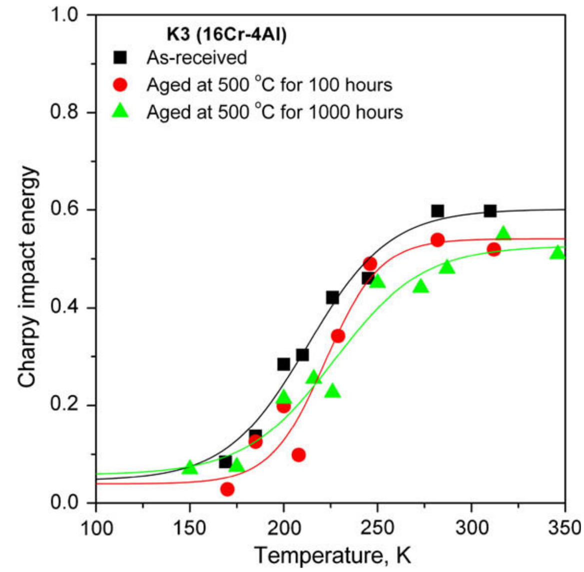

Fig. 6. Effects of a thermal aging treatment on the Charpy impact energy of the 16Cr–4Al ODS steels as a function of the test temperature for samples aged at 500 °C.

Fig. 5 shows the changes in the tensile properties of K1 and K4. The figure shows effect of the Al content on irradiation hardening. In contrast to Cr, the Al content did not affect the irradiation hardening of high-Cr ferritic ODS steels. According to Cho [8], when high-Cr ferritic ODS steels are irradiated at 290 °C to 9×10^{18} n/cm^2 (>1 MeV) for 1400 h, irradiation hardening is independent of the Cr content, although the hardening is significant and is even enhanced by the addition of Al. The findings of Cho contrast with those of this study. Compared with the irradiation condition in Cho's study, the irradiation time and dose used in this study were higher. Therefore, it is thought that irradiation hardening is affected by the irradiation time and dose even when the irradiation temperature is held constant. It should be noted that no effects of irradiation on the total tensile strain were observed, as shown in Figs. 4 and 5. This confirms that high-Cr ODS steels show irradiation hardening with no loss of elongation, as observed for the 9Cr and 12Cr ODS steels [9]. For high-Cr ODS steels, no anisotropy was observed in the tensile strength, although it was observed in total tensile strain. Specimens whose axis is parallel to the extrusion direction of the bars showed the largest degree of elongation, while those in the radial direction showed the least elongation for all of the ODS steels tested here.

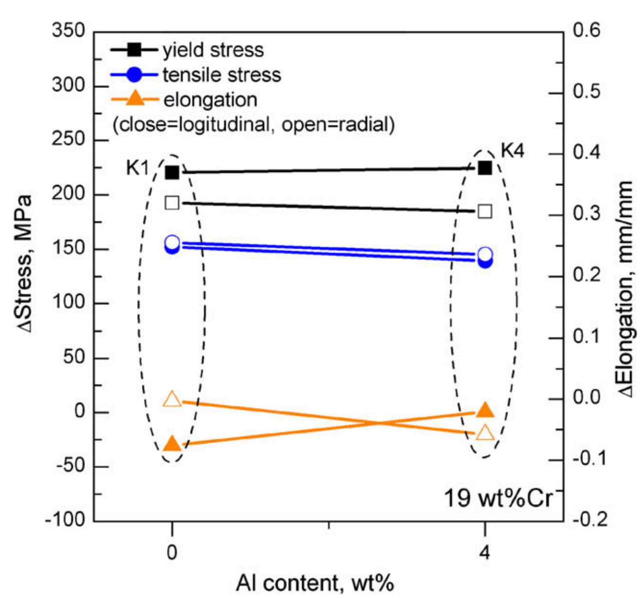

Fig. 5. Change in the yield stress, ultimate tensile strength and elongation versus the Al concentration after irradiation. Tensile tests were performed at room temperature at a strain rate of $6.7 \times 10^{-4}\ s^{-1}$.

3.3. The effect of thermal aging on irradiated ODS steels

It is well known that thermal aging of high-Cr ferritic steels can result in the formation of coherent particles of α' (Cr-rich ferrite) with an increase in the yield and tensile strength and a reduction in ductility [10] during the service at temperatures between 400 and 550 °C. Therefore, the effect of thermal aging on the hardening or embrittlement of irradiated ODS steel should be considered. It was reported that significant embrittlement associated with hardening was observed in some high-Cr ODS steels after thermal aging treatments at temperatures ranging from 430 to 475 °C [11]. On the other hand, this hardening was not observed in the high-Cr ODS steels in this study that were aged at 500 °C, as shown in Fig. 6. These results suggest that the degree of the thermal embrittlement of high-Cr ODS steels increases significantly below 500 °C.

Consequently, the hardening and embrittlement of high-Cr ODS steels irradiated at 550 °C is scarcely affected by irradiation or by thermal aging. On the other hand, the degree of hardening and embrittlement will increase by irradiation and thermal aging when high-Cr ODS steels are irradiated at 420 °C.

4. Conclusion

The effects of neutron irradiation on the Charpy impact energy and tensile properties of five high-Cr ODS steels were investigated after JMTR irradiation at 300, 420 and 550 °C. The main results are as follows:

(1) The reduction in the USE and the increase in the DBTT decreased as the irradiation temperature increased. Below an irradiation temperature of 420 °C, the degree of

irradiation-induced embrittlement increased. On the other hand, the degree of irradiation embrittlement was reduced significantly above temperatures of 550 °C.

(2) From the result of a tensile test of high-Cr ODS steels irradiated at 300 °C, the yield stress and UTS was increased by irradiation hardening. With an increase of the Cr content, the degree of irradiation hardening in terms of the increase in the yield stress and UTS of the high-Cr ODS steels irradiated at 300 °C increased with the Cr content. However, the addition of Al did not affect the change in the stress levels.

(3) The hardening and embrittlement of high-Cr ODS steels irradiated at 550 °C is scarcely affected by irradiation or thermal aging. On the other hand, the degree of hardening and embrittlement are increased by irradiation and thermal aging when high-Cr ODS steels are irradiated at 420 °C.

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