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Effects of neutron irradiation on the tensile properties of high-Cr oxide dispersion strengthened ferritic steels

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Abstract

The effects of neutron irradiation on the tensile properties of oxide dispersion strengthened (ODS) ferritic steels have been investigated. Various ODS steels containing 14–22 wt% Cr and 4.5 wt% Al or no Al were produced by mechanical alloying. In order to investigate their susceptibility to hardening and embrittlement induced by neutron irradiation, the ODS steels were irradiated in JMTR at 290, 400 and 600 °C up to doses of 0.01, 0.3 and 0.75 dpa, respectively. Tensile tests were performed at RT before and after irradiation. Microstructure observations were conducted in TEM. It was found that high-Cr-ODS steels show a significant hardening after irradiation at 290 and 400 °C, while no effect was observed after irradiation at 600 °C. The irradiation hardening, however, is not accompanied by a reduction of total elongation. After irradiation at 400 °C the measured hardening increases with Cr concentration, while it is almost constant versus Cr concentration after irradiation at 290 and 600 °C.

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1. Introduction

Oxide dispersion strengthened (ODS) ferritic/martensitic steels have been developed as fuel cladding material for sodium cooled fast breeder reactors (SFRs) [1,2]. Because of the dispersion of oxide particles, the ODS steels show high-strength at high-temperatures [3]. As for 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 [4,5].

The ODS steels developed as SFR fuel cladding material contain at most 12% chromium. It is well known that the corrosion resistance in high-temperature water diminishes significantly with decreasing chromium concentration below 13% [6–12]. In order to improve corrosion resistance in super critical pressurized water (SCPW), high-Cr-ODS steels have been developed [7–13]. However irradiation embrittlement caused by the Fe–Cr phase decomposition under neutron irradiation is a critical issue for high-Cr steels. The goal of this study is to develop ODS ferritic steels, which are resistant to corrosion in SCPW and exhibit a high resistance to neutron irradiation embrittlement and high-temperature strength, by means of alloy design.

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Table 1
Chemical composition of the materials used in this work (wt%)

Materials	C	Si	Mn	P	S	Cr	W	Al	Ti	N	Y	Y_2O_3
19Cr (K1)	0.05	0.041	0.06	<0.005	0.002	18.37	0.29	<0.01	0.28	0.014	0.29	0.368
14Cr–4Al (K2)	0.04	0.033	0.06	<0.005	0.002	13.64	1.65	4.12	0.28	0.009	0.30	0.381
16Cr–4Al (K3)	0.08	0.033	0.06	<0.005	0.002	16.00	1.82	4.59	0.28	0.006	0.29	0.368
19Cr–4Al (K4)	0.09	0.039	0.06	<0.005	0.002	18.85	1.83	4.61	0.28	0.005	0.29	0.368
22Cr–4Al (K5)	0.10	0.039	0.07	<0.005	0.002	22.05	1.80	4.55	0.27	0.005	0.28	0.356

In this work, the effects of neutron irradiation on the mechanical properties of high-Cr-ODS steels have been investigated to evaluate their performance under neutron irradiation.

2. Experimental

2.1. Materials

The materials used were ODS ferritic steels [1–3]. The chemical compositions are reported in Table 1. The details of the fabrication process of the ODS steels are given in previous papers [1–3]. These ODS steels were developed to investigate the effect of Cr and Al on the corrosion resistance and mechanical properties. The Cr concentration ranged from 14% to 22%. Most of the steels, except the K1 ODS steel, contained 4.5% Al. All the ODS steels were finally heat treated at 1323 K for 1 h, then air-cooled.

3. Neutron irradiation experiments

All the specimens were irradiated with neutrons in the Japan Materials Testing Reactor (JMTR). Irradiations were carried out at three different temperatures of 290, 400 and 600 °C, and up to three different doses reaching a maximum value of 5×10^{20} n/cm^2 (0.75 dpa), as reported in Table 2.

Table 2
Irradiation conditions with JMTR in this work

Capsule ID	Temperature (°C)	Time (h)	Irradiation dose
04M-16U	290	1403.23	9×10^{18} n/cm^2 (0.01 dpa)
03M-69U	400	2708.9	2×10^{20} n/cm^2 (0.3 dpa)
	600	2708.9	5×10^{20} n/cm^2 (0.75 dpa)

4. Tensile tests and microstructure analysis

As post-irradiation examination of mechanical properties, tensile tests were performed on SS-J miniature specimens. The gauge section of SS-J miniature specimens was $1.2 \times 5.0 \times 0.25$ mm^3. When manufactured by extrusion, the ODS steels show anisotropy in their mechanical properties, because grains are heavily elongated as a result of the extrusion process. In order to investigate the anisotropy of tensile properties, specimens were cut out from the extruded bars in the radial and longitudinal directions, as shown in Fig. 1. Microstructure observations were carried out using a transmission electron microscope (TEM) operating at 200 kV.

5. Results and discussion

5.1. Irradiation hardening

The tensile test results are shown in Fig. 2, where the irradiation-induced changes in yield stress, $\Delta\sigma_y$, and ultimate tensile strength, $\Delta\sigma_U$, are reported versus the irradiation temperature. Tensile tests were carried out at room temperature. The hardening is the largest after irradiation at 290 °C. With increasing irradiation temperature, the hardening decreases. The addition of Al causes an increase in the hardening.

Fig. 3 shows the changes in the ultimate tensile strength and total strain versus the Cr concentra-

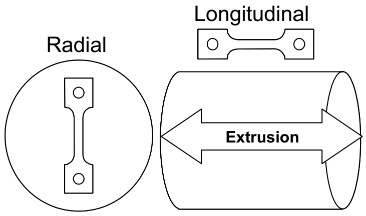
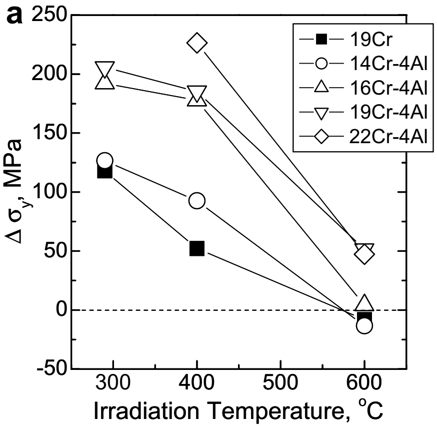


Fig. 1. Cutting scheme of the specimens used in tensile tests.



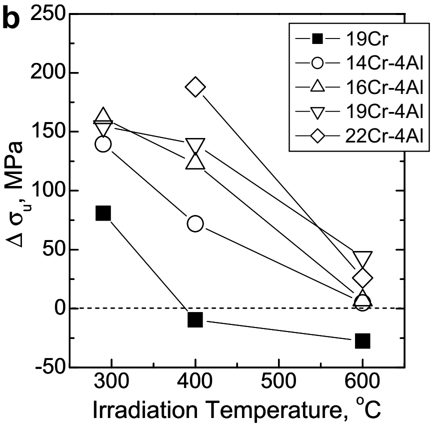
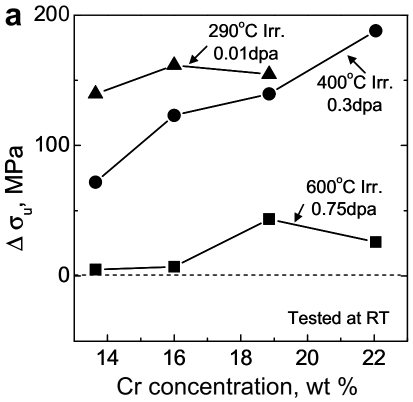


Fig. 2. Dependence of the changes in (a) yield stress: $\Delta\sigma_y$ and (b) ultimate tensile strength: $\Delta\sigma_u$, on the irradiation temperature. Tensile tests were carried out at room temperature at a strain rate of $6.7 \times 10^{-4}\ \mathrm{s}^{-1}$.



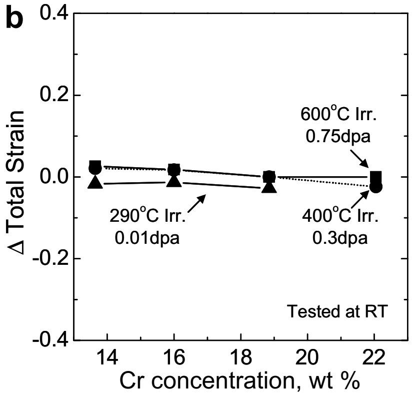


Fig. 3. Changes in (a) ultimate tensile strength: $\Delta\sigma_u$ and (b) total strain versus Cr concentration after irradiation. Tensile tests were performed at room temperature at a strain rate of $6.7 \times 10^{-4}\ \mathrm{s}^{-1}$.

tions in the ODS steels. After irradiation at 290 °C, the irradiation hardening appears independent of Cr concentration, although the hardening is significant. However, after irradiation at 400 °C, the irradiation hardening increases with Cr concentration. The K5 (22Cr–4Al) ODS shows a hardening twice that of the K2 (14Cr–4Al) ODS steel. On the other hand, after irradiation at 600 °C, almost no irradiation hardening was observed. It should be noticed that no irradiation effect on the total tensile strain was observed, as shown in Fig. 3(b). It was then confirmed that high-Cr-ODS steels show irradiation hardening accompanied by no loss of elongation, as observed for 9Cr- and 12Cr-ODS steels [4,5].

6. Anisotropy in tensile properties

For high-Cr-ODS steels, no anisotropy was observed in tensile strength but was observed in total tensile strain. The specimens whose axis is parallel to the longitudinal direction of the extruded bars showed the largest strain, and those in radial direction showed the smallest strain, for all the ODS steels. In order to evaluate the anisotropy in tensile properties, we defined the degree of anisotropy, D_{AN} as follows:

$$D_{\mathrm{AN}} = 1 - \frac{\text{total strain in radial direction}}{\text{total strain in longitudinal direction}}. \qquad (1)$$

A large D_{AN} stands for large anisotropy. For example, if the degree of anisotropy is 1, then the material is completely anisotropic. As shown in Fig. 4, the K1 (19Cr) and K5 (22Cr–4Al) ODS steels show relatively severe anisotropy after irradiation. However most of the ODS steels containing Al, except K5, do not show severe anisotropy, even after irradiation. It can be concluded that Al addition decreases the anisotropy in total tensile strain of the ODS steels, especially in the unirradiated state.

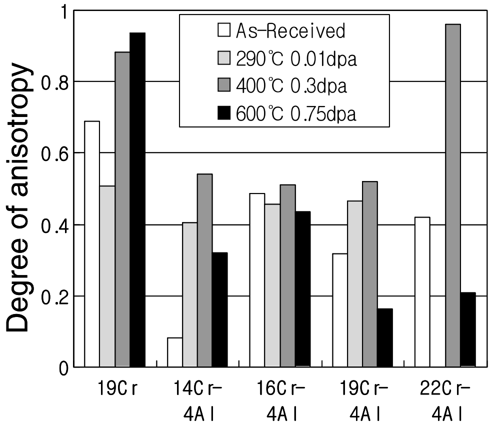


Fig. 4. Degree of anisotropy ratio in total tensile strain before and after irradiation.

7. TEM observations

After irradiation at 290 °C to 0.01 dpa, many dislocation loops were observed in the K3 (16Cr–4Al) ODS steel, as shown in Fig. 5. The average loop size was about 12 nm and the number density was $6.1 \times 10^{21}\ \mathrm{m}^{-3}$. From these numerical data, the irradiation hardening ($\Delta\sigma_{\mathrm{y\,cal}}$) was evaluated by using the Orowan equation [14]. The obtained $\Delta\sigma_{\mathrm{y\,cal}}$, due to dislocation loops, was about 126 MPa. This value is smaller than the experimental value ($\Delta\sigma_{\mathrm{y\,exp}}$: 192 MPa) reported in Fig. 2, nevertheless it can be concluded that irradiation hardening at 290 °C is mostly due to the formation of dislocation loops.

Fig. 6(a) shows the TEM image of the K4 (19Cr–4Al) ODS steel after irradiation at 400 °C to 0.3 dpa. A number of spherical precipitates, which are thought to be α′ phase precipitates, were observed. Similar α′ precipitates were observed in specimens aged at 500 °C for 1000 h (Fig. 6(b)), although they are larger than those observed after irradiation at 400 °C. Because of the Cr concentration dependence of the irradiation hardening, it is considered that the irradiation hardening at 400 °C is due to the formation of α′ phase as in the case of aging hardening at 500 °C.

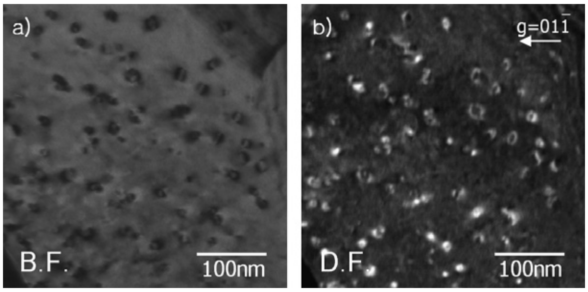


Fig. 5. Dislocation loops after irradiation at 290 °C for about 1400 h to 0.01 dpa in the K3 (16Cr–4Al) ODS steel: (a) bright field image and (b) dark field image.

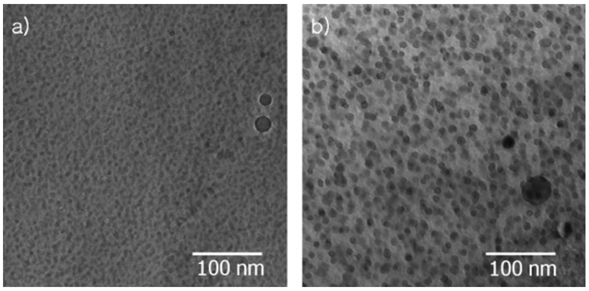


Fig. 6. α′ phase precipitates after (a) irradiation at 400 °C for about 2700 h to 0.3 dpa in the K4 (19Cr–4Al) ODS steel and (b) after thermal aging at 500 °C for 1000 h also in the K4 ODS steel.

8. Conclusions

The effects of neutron irradiation on the tensile properties and microstructure of high-Cr-ODS steels were investigated after JMTR irradiation at 290, 400 and 600 °C to doses of 0.01, 0.3 and 0.75 dpa, respectively. The main obtained results are the following:

1. High-Cr-ODS steels show a significant hardening after the irradiation at 290 and 400 °C, while no effect is observed after irradiation at 600 °C. The significant irradiation hardening, however, is not accompanied by a reduction of total elongation.
2. Irradiation hardening at 290 °C is mainly due to the formation of dislocation loops.
3. After irradiation at 400 °C, the measured hardening increase with Cr concentration. The irradiation hardening at 400 °C is considered to be due to the formation of α′ phase precipitates.
4. After irradiation at 600 °C, no effects of irradiation were observed for high-Cr-ODS steels, indicating a good irradiation resistance. Further irradiation experiments up to much higher doses are necessary to evaluate the irradiation resistance of the ODS steels.

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