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Journal of Nuclear Materials 283–287 (2000) 1144–1147

journal of nuclear materials

www.elsevier.nl/locate/jnucmat

Microstructural development of neutron irradiated W–Re alloys

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Abstract

Tungsten (W) alloys are candidate materials to be used as high-heat-flux materials in fusion reactors. In our previous work, W–26 wt% Re showed drastic hardening and embrittlement after the neutron irradiation. In this study, to clarify the irradiation hardening and embrittlement behavior of W–26 wt% Re, from the viewpoint of microstructural development, the microstructure observation of the neutron irradiated W–26 wt% Re was carried out using transmission electron microscope (TEM). The specimens were irradiated at the materials open test assembly of the fast flux test facility (FFTF/MOTA-2A cycle 11) up to $\sim 1 \times 10^{27}$ n/m^2, ($E_n > 0.1$ MeV). The irradiation temperatures were 646, 679, 792, 873 and 1073 K. In all neutron irradiated W–26 wt% Re samples, sigma-phase precipitates and chi-phase precipitates were observed, while in the thermally aged specimen, only sigma-phase precipitates were observed. Irradiation effects on microstructural development are discussed.

1. Introduction

Tungsten (W) is one of the candidates for high-heat-flux materials in fusion reactors, because of its high melting point, high thermal conductivity, high resistance to erosion and sputtering [1–4]. The addition of rhenium (Re) to W increases the recrystallization temperature and ductility [5,6]. Under fusion irradiation conditions, transmutation by 14 MeV neutron irradiation is one of the major concerns. In the STARFIRE studies, 4 at.% Re and 0.5 at.% osmium (Os) form after neutron irradiation of pure W at 10 dpa [7]. Thus, a study of the mechanical properties and physical properties of W–Re alloys after neutron irradiation is required to predict transmutation effects on irradiated W. In a previous study of neutron irradiated W–Re alloys, drastic radiation-induced embrittlement and hardening by precipitation were reported [8–12]. In the case of other refractory metals such as molybdenum (Mo), we have reported that irradiation embrittlement of Mo and Mo–Re can be suppressed by pre-irradiation heat treatment [13,14]. The aim of this work is to study the influence of pre-irradiation thermal treatment and irradiation temperature on the microstructural development of W–26 wt% Re after neutron irradiation.

2. Experimental

A sheet of powder metallurgical W–26 wt% Re alloy was supplied by the Plansee. This was hot-worked and cold-worked by the supplier. Chemical composition of the material is listed in Table 1. A disk specimen (thickness: 0.15 mm, diameter 3.0 mm) was punched out from the sheet, and two different pre-irradiation heat treatments were carried out in vacuum. These were; a recrystallization treatment (grain size: 2.0 μm) at 1873 K for 1 h, and a stress relief treatment (grain size: 0.6 μm) at 1573 K for 1 h. The specimens were irradiated in the materials open test assembly of the fast flux test facility (FFTF/MOTA-2A cycle 11) up to $\sim 1 \times 10^{27}$ n/m^2 ($E_n > 0.1$ MeV), in a helium-filled capsule. The irradia-

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PII: S0022-3115(00)00290-7

Table 1
Chemical composition of W–26 wt% Re

W	Re										
74±1	26±1 (wt%)										
H	C	O	N								
5	30	30	10 (wt.ppm)								
Ag	Al	As	Ba	Ca	Cd	Co	Cr	Cu	Fe	K	Mg
5	15	5	10	10	10	10	10	10	30	10	5
Mn	Mo	Na	Nb	Ni	Pb	S	Si	T	Ti	Zn	Zr
5	100	10	10	20	10	5	20	10	10	5	10 (wt.ppm)

tion conditions are presented in Table 2 [15]. To study the effect of thermal aging during irradiation on microstructure, the recrystallized W–26Re were thermally aged at 673, 873, 1073 K for 1 month in an evacuated and shielded quartz tube.

After irradiation, thin specimens for microstructural observation were prepared by electro-polishing. The polishing solution was 2% NaOH with water (0.5 mol/l). Polishing was carried out at 85 V and 1.2 A at a temperature of 278 K. The microstructure of the specimens was observed using a transmission electron microscope (JEOL 2000FX) at 200 kV. Vickers hardness testing was carried out at room temperature at a load of 1.96 N for 20 s. X-ray diffraction (JEOL, JDX3530, 40 kV, 40 mA) was then used to detect the precipitates in the specimen.

Table 2
Irradiation condition

Irradiation temperature (K)	Fluence ($E_n > 0.1$ MeV) ($\times 10^{26}$ n/m^2)	Irradiation damage (dpa)[a]
646±5	2.0	2
679±5	7.5	9
792±5	9.5	11
873±5	9.5	11
1073±5	3.2	4

[a] Calculated as $E_d = 90$ eV.

3. Results and discussion

Fig. 1(a) shows a typical microstructure of W–26Re after neutron irradiation to 11 dpa at 873 K. No voids, no dislocations, no dislocation loops, but two types of fine and dense precipitates were observed in all irradiated specimens. The precipitates were classified to: (1) equiaxed-type precipitates and (2) platelet-type precipitates. As is shown in Fig. 1(a), the platelet-type precipitates were also observed at the grain boundaries. However it is only in the case when the grain boundary is parallel to the (1 1 0) plane of matrix. Figs. 1(b) and (c) show the dark field images of the precipitates with diffraction patterns. Fig. 2 shows the diffraction pattern from (0 0 1) and (1 1 1) planes of the specimen. The diffraction patterns and the dark field images indicate that the equiaxed type precipitates are sigma-phase with a CrFe structure, and the platelet type precipitates are chi-phase with a α-Mn structure [16]. As it is shown in Fig. 2(b), streaks on the diffraction pattern were observed. It indicates that the chi-phase precipitates are sheet-type precipitates on the (0 1 1) plane of the matrix.

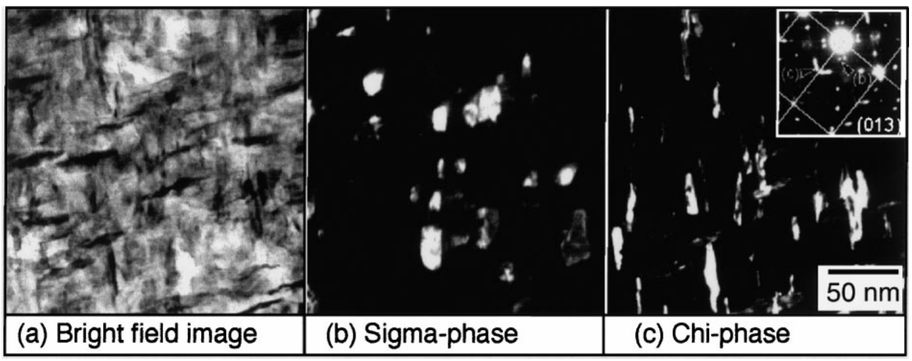

Fig. 1. TEM micrographs of stress-relieved W–26Re after neutron irradiation: (a) bright field image; (b), (c) dark field images of the precipitates with the diffraction pattern.

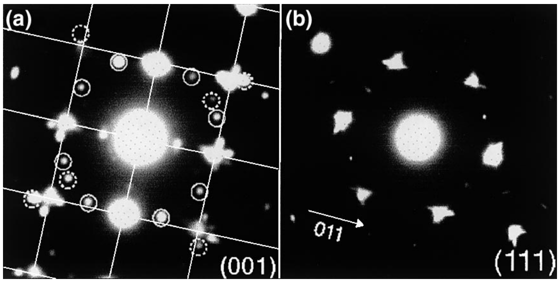

Fig. 2. Diffraction patterns from recrystallized and neutron-irradiated W–26Re: (a) the lines are drawn through the matrix (W) spots. Diffraction spots from the sigma-phase precipitates, diffraction spots from the chi-phase precipitates. The other spots might be double diffraction patterns; (b) The streaks indicate that there are thin plate like precipitates on the (0 1 1) plane of W.

Fig. 3 shows the effect of irradiation temperature on the size of the chi-phase precipitates. The size means the length of the long axis of the platelet precipitates. The size of the chi-phase precipitates increased with the irradiation temperature, and the number density, which was in the same range of about 10^{22} m^{-3}, did not depend on the irradiation temperature. The effect of pre-irradiation heat treatment on precipitation behavior of chi-phase was not clearly observed. Also the effect of radiation damage on precipitation behavior was not clearly observed in the range of 2–11 dpa. The image of sigma-phase was not as clear as that of chi-phase, so the densities are not as certain as that for the chi-phase. Nevertheless, it is concluded that the size of sigma-phase precipitates increased with an increase in irradiation temperature (5–15 nm).

Vickers hardness of recrystallized specimen increased slightly after thermal aging for 1 month. For example, the increments of Vickers hardness are 38 for 1073 K, 42 for 873 K and 33 for 673 K, respectively.

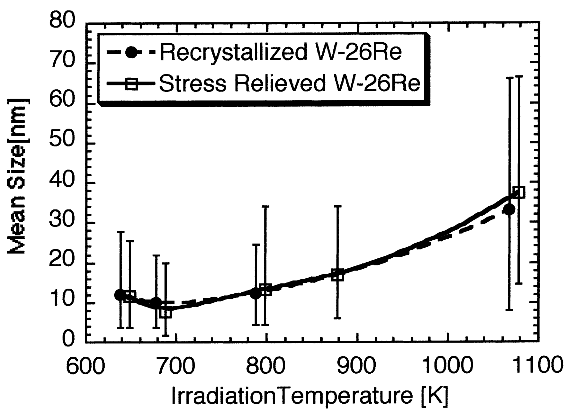

Fig. 3. Effect of irradiation temperature on the size of the chi-phase precipitates.

Hardness of as-recrystallized sample is 466. The corresponding TEM micrograph for the specimen thermally aged at 1073 K for 1 month is shown in Fig. 4. Equiaxed type precipitates were observed in this specimen. No precipitates were observed in the specimens thermally aged at 673 and 873 K. The size of the precipitates (50 nm) in Fig. 4 was greater, and the density (4.5×10^{22} m^{-3}) was lower than that was observed in irradiated specimens. From the X-ray diffraction results for the thermally aged specimen, the precipitates appear to be sigma-phase, with a CrFe structure and a lattice parameter of $a = 0.939$ nm and $c = 0.489$ nm, respectively.

In our previous work, all the W–26Re irradiated specimens in FFTF/MOTA-2A showed irradiation induced embrittlement and hardening [9]. The microstructural observation of this work suggests that the hardening and the embrittlement may be due to precipitation of sigma- and chi- phases. The sigma- and chi-phases have hardness values of 1400 and 500 Hv, respectively [17]. These precipitates are brittle, so they would initiate points of cracking, and if they are on the grain boundary, they would weaken the grain boundary strength. In addition, they would harden the matrix. Especially the chi phase is on the (0 1 1) plane, so it would be a strong obstacle for dislocation gliding on the (1 1 0) plane in W [17]. In the results of the thermal aging experiment, only the sigma-phase precipitates were formed. Therefore chi-phase precipitates can be considered as radiation induced precipitates.

The results of this work suggest that a reduction in precipitation in W–Re alloys is required for satisfactory use in fusion reactors with an optimum Re content that may be less than 26 wt%. To predict irradiation embrittlement by the irradiation induced precipitates, Re

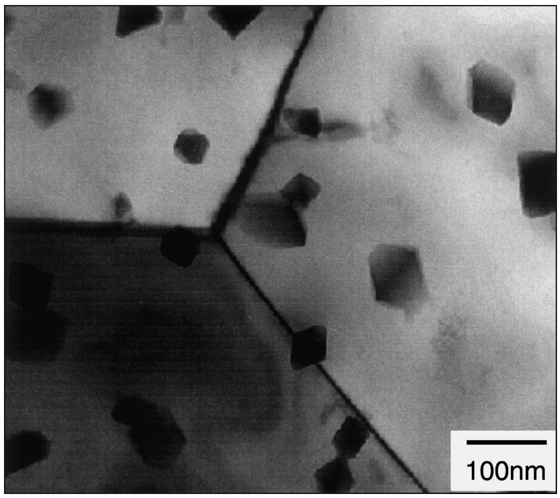

Fig. 4. TEM micrograph of recrystallized W–26Re that was thermally aged at 1073 K for 1 month.

and Os contents produced by transmutation under irradiation should be estimated, and considered in the design of the alloy. An additional study of the effects on Re content in W–Re alloys after neutron irradiation is required to predict the transmutation effects of W on microstructural development and irradiation induced embrittlement.

4. Summary

The microstructure of neutron irradiated W–26Re was studied in this work. Sigma-phase and chi-phase precipitates were observed in all specimens after neutron irradiation. The fine and dense chi-phase precipitates appear to be formed as irradiation induced precipitation with large irradiation hardening and embrittlement of W–26Re.

Acknowledgements

Authors are grateful to Mr Y. Takahashi and M. Satou, Radio-Isotope Laboratory of the Faculty of Engineering, of Tohoku University, for their help in radioactive materials experiments. This work was supported by the JUPITER program (Japan–USA Program of Irradiation Test for Fusion Research) and by a grant-in-aid for Scientific Research from the Ministry of Education, Science and Culture, Japan.

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