Neutron energy spectrum influence on irradiation hardening and microstructural development of tungsten

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Abstract

Neutron irradiation to single crystal pure tungsten was performed in the mixed spectrum High Flux Isotope Reactor (HFIR). To investigate the influences of neutron energy spectrum, the microstructure and irradiation hardening were compared with previous data obtained from the irradiation campaigns in the mixed spectrum Japan Material Testing Reactor (JMTR) and the sodium-cooled fast reactor Joyo. The irradiation temperatures were in the range of ~90 to

~800 °C and fast neutron fluences were 0.02 to $9.00 \times 10^{25} \text{ n/m}^2$ ($E > 0.1 \text{ MeV}$). Post irradiation evaluation included Vickers hardness measurements and transmission electron microscopy. The hardness and microstructure changes exhibited a clear dependence on the neutron energy spectrum. The hardness appeared to increase with increasing thermal neutron flux when fast fluence exceeds $1 \times 10^{25} \text{ n/m}^2$ ($E > 0.1 \text{ MeV}$). Irradiation induced precipitates considered to be χ - and σ -phases were observed in samples irradiated to $>1 \times 10^{25} \text{ n/m}^2$ ($E > 0.1 \text{ MeV}$), which were pronounced at high dose and due to the very high thermal neutron flux of HFIR. Although the irradiation hardening mainly caused by defects clusters in a low dose regime, the transmutation-induced precipitation appeared to impose additional significant hardening of the tungsten.

Keywords

tungsten, neutron irradiation, irradiation hardening, microstructure, neutron spectrum effect, HFIR, JMTR, Joyo

1. Introduction

The plasma facing materials (PFMs) of fusion reactors will be exposed to neutron irradiation over an energy range up to 14.1 MeV. In ITER, tungsten (W) is the primary choice for the divertor PFM [1]. Because tungsten has desirable material properties such as high melting temperature, high thermal conductivity, low sputtering yield, and low tritium retention, it is also a candidate for the PFM in demonstration reactor (DEMO) designs [2,3]. However, the effects of neutron irradiation on these properties of W are largely unknown. Among the critical concerns are irradiation-induced mechanical property changes, in specifically embrittlement [4], which can have a significant impact on design. Therefore, understanding the mechanical property changes and the underlying microstructural processes in W due to neutron irradiation is one of the important subjects for the R&D of fusion materials.

Neutron irradiation causes formation of defect clusters such as voids and dislocation loops in W. These defect clusters are known to cause increases in hardness and yield stress and decreases in elongation for W [5,6]. The irradiation induced precipitation also causes large irradiation hardening and decreased ductility (irradiation embrittlement) in pure W and W-Re alloys [7-9]. In the case of pure W, the transmutation products rhenium (Re) and osmium (Os) will be formed during neutron irradiation, and the segregation of these elements results in irradiation induced precipitates. The production rates of the transmutation elements depends on the flux and the energy spectrum thermal neutrons. For this reason, the extents of spectrum moderation, related to the distance from the first wall and the designs and configurations of the divertor and blanket (e.g. water-cooled compared to gas-cooled) have a large impact on the production rates for transmutant of Re and Os as normalized to the atomic displacement damage [10-13]. Because of this, the microstructure and related mechanical properties will be a function of the material's location within the fusion machine as that is

related with the produced damage and transmutation rate. The objective of this work is to investigate the neutron spectrum dependence of irradiation hardening and microstructural development in pure W.

2. Experimental

High purity (99.999%) single crystal W oriented along [110] was the source material for this work, and this material is denoted as (SX) in this paper. The impurity contents in the as-received material are listed in Table 1 [14]. The neutron irradiation was conducted in the flux trap facility in the High Flux Isotope Reactor (HFIR) at Oak Ridge National Laboratory (ORNL) in the United States. The neutron irradiation conditions are listed in Table 2. The irradiation temperatures were ~90 to ~800 °C and the neutron fluences were 0.02 to 9.00×10^{25} n/m² ($E > 0.1$ MeV). The neutron fluences given in this paper are for fast neutrons as defined $E > 0.1$ MeV. The irradiation temperatures were determined by examining the silicon carbide (SiC) temperature monitors in each irradiation capsule [15]. Following the irradiation, micro-Vickers hardness measurements and microstructure observations were carried out. The Vickers hardness measurements were conducted at room temperature, with an indentation load of 1.96 N (200 gf) and a dwell time of 15 s. The number of measurements was 3–5 for each samples. The Vickers hardness of unirradiated SX sample was HV376. The thin foil samples for microstructure observation were prepared by using a focused ion beam (FIB) device, FEI Quanta 3D DualBeam. The FIB was operated at 30 kV for a rough milling, and at 2 kV for a finish polish. Insignificant damage was found by FIB processing in our previous work [14]. The microstructure of the samples was observed using a Philips CM200-FEG transmission electron microscope (TEM) operating at 200 kV.

In order to investigate the effects of neutron energy spectrum, the previous results of microstructure observation and hardness measurements for W irradiated in the Japan

Materials Testing Reactor (JMTR) and experimental fast reactor Joyo operated by the Japan Atomic Energy Agency (JAEA) are referred to for comparison [9,16-21]. The irradiation conditions of this work and the reference data are plotted in Figure 1. The irradiation temperatures were determined by thermocouples and temperature monitors in JMTR and Joyo, respectively.

Two types of pure W were examined from literature. One of them is a rolled sheet and the other one is an arc-melted button, and these materials are hereafter referred to as (HR) and (AC), respectively. The pure W (HR) samples were annealed at 1400 or 1600 °C as a recrystallization heat treatment before neutron irradiation. Pure W (AC) samples were annealed at 1400 °C for 1 h as a stress relief heat treatment before the neutron irradiation. The conditions for Vickers hardness measurements in referred articles are the same as that of this work. The Vickers hardness of unirradiated HR and AC samples were HV408 and 392, respectively. The samples from the JMTR and Joyo that were used for microstructure observation by TEM were prepared by electro polishing.

3. Results and discussion

3.1. Irradiation hardening

The neutron fluence and irradiation dose dependence of the irradiation hardening of pure W after neutron irradiation in HFIR, JMTR, and Joyo were plotted in Figures 2 (a) and (b), respectively. The data scatter of each plots were negligibly small. Due to the difference in neutron energy spectrum, the fluence/dpa ratio differs for each reactor. The fluence/dpa ratios for HFIR and Joyo were 3.1×10^{25} and 7.9×10^{25} (n/m², $E > 0.1$ MeV)/dpa, respectively. The irradiation hardening (ΔHV) was determined as the difference in Vickers hardness before and after the neutron irradiation. The irradiation hardening of pure W (SX) irradiated in HFIR was approximately $\Delta HV50$ –100 below ~ 0.16 dpa ($\sim 0.5 \times 10^{25}$ n/m²). After ~ 0.6 dpa ($\sim 2 \times$

10^{25} n/m^2), significant irradiation hardening was observed. The magnitude of the irradiation hardening of pure W (SX) irradiated in HFIR increased with increasing dose, and it reached $\Delta\text{HV} \sim 300\text{--}400$ after irradiation to $0.7\text{--}0.9 \text{ dpa}$. After $\sim 2.9 \text{ dpa}$ ($\sim 9 \times 10^{25} \text{ n/m}^2$), the magnitude of the irradiation hardening was $\Delta\text{HV} \sim 770$. As for the temperature dependence of the irradiation hardening in pure W (SX), the sample irradiated in the intermediate temperature range ($400\text{--}600 \text{ }^\circ\text{C}$) had higher irradiation hardening than material irradiated at higher ($650\text{--}850 \text{ }^\circ\text{C}$) and lower ($90 \text{ }^\circ\text{C}$) temperature ranges at below $\sim 1 \text{ dpa}$ ($3 \times 10^{25} \text{ n/m}^2$).

The trend of irradiation hardening of pure W irradiated in HFIR and JMTR was almost the same, although there is a lack of data between $0.2\text{--}0.6 \text{ dpa}$ in HFIR. The HFIR and JMTR are mixed-spectrum reactors, and the neutron energy spectra in these reactors are relatively similar [22,23]. As for pure W irradiated in Joyo, almost no difference in the irradiation hardening was observed below $\sim 1 \text{ dpa}$ compared to the samples irradiated in HFIR and JMTR.

The HR samples were heat treated at 1400 or $1600 \text{ }^\circ\text{C}$ for 1 h before the neutron irradiation, so these samples were in almost recrystallized state. The deformed texture such as high dislocation density and cell structures of the HR samples was almost recovered and the remaining dislocation density was quite small [18]. The AC samples were fabricated by arc-melting, thus, they did not have deformed texture. The average grain size of the HR and AC samples was $\sim 14 \text{ }\mu\text{m}$ and $>100 \text{ }\mu\text{m}$, respectively, and these materials showed almost the same hardness [18]. In contrast, the W material irradiated in HFIR was a single crystal, hence the grain boundaries were not present, and before irradiation defect density was very low. The chemical impurity level of HR and AC samples were almost the same with sample examined in this work [18]. It is known that dislocations and grain boundaries, which act as sinks for migrating defects, and chemical impurity levels can change the magnitude of irradiation hardening. However, the differences between the grain sizes and chemical impurities of the

SX, HR, and AC materials did not cause the typical change in the irradiation hardening behavior. The irradiation hardening behavior above ~1 dpa was clearly dominated by the different energy spectra of the reactors. The pure W irradiated in Joyo showed a lower magnitude of irradiation hardening than that irradiated in HFIR. The Joyo is a fast reactor, thus, the thermal neutron flux is much lower than HFIR and JMTR [22-24]. It is believed that the difference in thermal neutron flux affected irradiation hardening of pure W.

3.2. Microstructure development

Figure 3 shows the microstructure of pure W (SX) irradiated in HFIR. The numbers in these images correspond to those in Figures 2 (a) and (b). Dislocation loops and voids were observed in pure W (SX) after irradiation to 0.15 and 0.70 dpa at ~770 and ~710 °C. After irradiation to 0.90 dpa at ~430 °C, only dislocation loops were observed. In order to investigate the effect of difference in reactor type on microstructural development of pure W, the microstructure images of pure W irradiated in JMTR and Joyo are shown in Fig. 4. The types of irradiation defects appeared to be independent of the reactor type below ~1 dpa. The measured average size and number density of irradiation defects (i.e. dislocation loops and voids) in irradiated pure W are listed in Table 3. Differences in the average size and number density of dislocation loops and voids between samples irradiated in HFIR and JMTR were observed after irradiation to ~0.15 dpa at ~800 °C. At this irradiation condition, the pure W irradiated in HFIR had relatively higher number density and smaller size of dislocation loops than the pure W in JMTR. In addition, void formation was suppressed in pure W irradiated in HFIR. Although the quantitative evaluation of the effect of these factors will be completed as future work, one of the likely causes of this discrepancy is the difference in damage rates of the two reactors. The damage rates in HFIR and JMTR are $\sim 2.3 \times 10^{-7}$ and $\sim 3.3 \times 10^{-8}$ dpa/s, respectively [14,21,25-29]. One would expect that a higher damage rate would cause the

formation of relatively dense and small defect clusters. In addition to the different damage rates, there are other possible causes for the different defect densities such as chemical impurity level, grain size, and so on.

After irradiation to 0.70 dpa at ~710 °C in HFIR, voids were mainly observed, and small number of dislocation loops were also observed. In pure W irradiated to 0.42 and 0.96 dpa at 756 and 538 °C, respectively in Joyo, voids were also the predominant defect cluster. Although there is a difference in the number density of voids and dislocation loops between the HFIR and Joyo irradiation cases, this difference might be attributed to the difference in the dpa value and the amount of transmutation products but not the dpa rate. The dpa rate of Joyo was $\sim 1.5 \times 10^{-7}$ dpa/s, and this value is relatively similar to that of HFIR. The effect of the amount of transmutation products on microstructural development is discussed later in this section.

More significant differences in the observed microstructure were found above ~1 dpa irradiation, and these were irradiation induced precipitation and void formation. Irradiation induced precipitates were observed in pure W (SX) irradiated to 2.9 dpa in HFIR. In contrast, irradiation induced precipitates were not observed in pure W (AC) after irradiation to 1.54 dpa at 756 °C in Joyo [19,20]. The key difference between these reactors is the thermal neutron flux because the transmutation elements, namely Re and Os, are produced by transmutation reactions of thermal neutrons with W. The thermal neutron flux in HFIR is higher than that in Joyo, therefore, larger amounts of transmutation elements are created by neutron irradiation in HFIR than in Joyo at the same dpa level. The estimated compositions of pure W after the neutron irradiation at midplane in HFIR and at reflector region in Joyo, which is based on the calculation using FISPACT code, were W-9.22%Re-5.02%Os and W-1.47%Re-0.05%Os, respectively [9,30]. The formation of irradiation induced precipitates has previously been reported for pure W [31] and W-Re alloys [7,9]. The previous irradiation

induced precipitates were considered to be χ - and σ -phases [9,31], and these precipitates likely consist of not only W and Re but also Os.

The Vickers hardness of χ - and σ phases were measured by using as-cast W-Re alloys, and these phases hardness are $\sim$ HV500 and $\sim$ 1400, respectively [32]. These values are larger than that of matrix (HV376). Additionally, χ -phase, which forms on the (011) plane, might act as a strong obstacle for dislocation motion [31,32]. The effect of these irradiation induced precipitates on mechanical properties has been reported; bending test results showed an increase of fracture stress and decrease of fracture strain in W-26%Re alloys due to the irradiation induced precipitation [8]. Therefore, the irradiation hardening in pure W irradiated to $> \sim 1$ dpa in the present study was considered to be caused by the formation of irradiation induced precipitates.

In addition to the irradiation induced precipitation, a decrease in the number density of voids was observed after irradiation at all dpa levels. The effect of Re on void formation was previously investigated. After neutron irradiation to ~ 0.5 dpa, void formation in W-3, 5, and 10% Re was suppressed compared to pure W [18], which agrees with the result of this work. Suzudo et al. conducted a the first principle calculation based on density functional theory and the atomic kinetic Monte Carlo simulations to investigate the binding energies of the solute Re and Os atoms with vacancy and self-interstitial atom (SIA) and the migration of these solute atoms [33,34]. They found that the Re and Os atoms have positive binding energy to point defects and the rotation energy barrier of W-Re and W-Os mixed dumbbells was lower than that of SIAs. Because of the lower rotation energy barrier, Re and Os have 3D motion although SIAs prefer 1D motion. This means that the Re addition increases the probability of recombination in the cascade-displacement region, which suppresses void formation. The results of Suzudo et al. [33,34] showed good consistency with the results in this work.

4. Summary

The microstructure and irradiation hardening of single crystal pure W irradiated in HFIR was investigated and compared with previous work in Joyo and JMTR.

The irradiation hardening of single crystal pure W irradiated in HFIR observed in the early stage of neutron irradiation (< 0.16 dpa) was due to the formation of dislocation loops and voids. With increasing dose up to ~ 1 dpa, the microstructural development such as voids and dislocation loops formation was observed and the magnitude of irradiation hardening reached to $\Delta HV=400$. In the dose range up to ~ 1 dpa, the magnitude of irradiation hardening of pure W irradiated in HFIR, Joyo, and JMTR was approximately the same, and the neutron energy spectrum influence of irradiation hardening was not distinctive. The number density of voids in pure W irradiated to < 1 dpa in HFIR was significantly lower than that in Joyo. The higher amount of the solute transmutation products such as Re and Os might have suppressed the void formation in W irradiated in HFIR.

In the dose range above 1 dpa, the neutron energy influence on irradiation hardening and microstructural development was clearly observed. The magnitude of the irradiation hardening of pure W irradiated in HFIR was approximately twice as high as that in Joyo. The significant irradiation hardening of pure W irradiated in HFIR is likely caused by irradiation induced precipitates which consists of transmutation products such as Re and Os.

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Table 1 Chemical compositions of single crystal pure W examined in this work [14]

C	N	O	H	P	Cu
20	< 10	10	3	<5	140

Unit: ppm

Table 2 Neutron irradiation conditions

Capsule ID	Sample ID	Irrad. temp., °C	Fluence, 10^{25} n/m ² (E > 0.1 MeV)	dpa
PC1A	1W05	90	0.02	0.01
PC2A	1W25	90	0.10	0.03
PC4A	1W43	90	1.95	0.62
TB-300-2	1W47	460	0.10	0.03
T9G-11	1W07	430	2.82	0.90
TB-500-2	1W51	690	0.54	0.17
T9C-14	1W17	710	2.20	0.70
TB-500-3	1W19	770	9.00	2.88
TB-650-1	1W23A	830	0.13	0.04
TB-650-2	1W53	800	0.46	0.15

Table 3 Measured average size and number density of dislocation loops and voids in irradiated pure W

Reactor	Irrad. Temp., °C	Fluence, 10^{25} n/m ² (E > 0.1 MeV)	Dose, dpa	Dislocation loop		Void	
				Average diameter, nm	Number density, 10^{22} m ⁻³	Average diameter, nm	Number density, 10^{22} m ⁻³
HFIR	800	0.46	0.15	2.0	4.8	3.9	0.2
HFIR	710	2.20	0.70	2.2	2.4	4.1	0.3
HFIR	430	2.82	0.90	5.4	3.1	—	—
HFIR	770	9.00	2.88	3.6	3.2	6.3	0.3
JMTR	800	0.81	0.15	8.5	0.1	1.9	4.2
Joyo	756	3.30	0.42	5.6	0.1	2.5	12.1
Joyo	538	7.80	0.96	4.7	4.7	2.1	49.0

Figure captions

Fig.1 Irradiation conditions of this work and previous data

Fig.2 Fluence and dose dependence of irradiation hardening of pure W

Fig.3 Microstructure of single crystal pure W (SX) irradiated in HFIR

Fig.4 Microstructure of pure W irradiated in JMTR and Joyo

Fig.1 Irradiation conditions of this work and previous data

Fig.2 Fluence and dose dependence of irradiation hardening of pure W

① 800 °C, 0.15 dpa (HFIR)

② 710 °C, 0.70 dpa (HFIR)

③ 430°C, 0.90 dpa (HFIR)

④ 770°C, 2.88 dpa (HFIR)

Fig.3 Microstructure of single crystal pure W (SX) irradiated in HFIR. Dislocation loops and precipitates which identified by a weak-beam method and the under focused image of voids were shown on the left and right side, respectively.

Fig.4 Microstructure of pure W irradiated in JMTR and Joyo