Journal of Nuclear Materials 507 (2018) 78–86

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

Effect of neutron irradiation on rhenium cluster formation in tungsten and tungsten-rhenium alloys

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ARTICLE INFO

Article history:
Received 15 December 2017
Received in revised form
30 March 2018
Accepted 18 April 2018
Available online 20 April 2018

Keywords:
Tungsten
Tungsten-rhenium alloys
Re clusters
Atom probe tomography

ABSTRACT

To clarify the irradiation hardening behavior of neutron-irradiated tungsten(W), the formation behavior of rhenium(Re)-enriched cluster and W-Re precipitation with displacement damage in fission reactors environment was investigated by the atom probe tomography (APT) technique, and the results obtained by APT and microstructural observation by transmission electron microscopy (TEM) were compared. Neutron-irradiated pure W and W-10Re alloys were used for the analysis. These samples were irradiated to 0.96 dpa at 538 °C in a fast experimental reactor Joyo, and irradiated to 0.90 dpa at 500 °C in a mixed spectrum reactor HFIR. The chemical compositions of the matrix, Re clusters, and precipitates were determined by the AP analysis. The Joyo-irradiated samples contained lower levels of transmuted Re and Os, while the HFIR-irradiated samples contained higher levels of Re and Os. The Re-enriched clusters demonstrated characteristic spatial distribution related to defect cluster distribution in each irradiated specimen.

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1. Introduction

Tungsten (W) has a high melting point, good thermal conductivity, sputtering resistance, and low hydrogen inventory. Thus, W is a promising candidate material for plasma-facing components (PFC) such as diverter plates and first wall of blankets in fusion reactors [1,2]. These plasma-facing materials (PFMs) are exposed to neutron irradiation (first wall: 30 dpa for 5 fpy with 10 MWa/m^2, divertor plate: 15 dpa for 5 fpy with 5 MWa/m^2), and it is well known that neutron irradiation causes changes in material properties by displacement damages such as vacancies, self-interstitial atoms (SIAs), and their clusters (e.g., voids and dislocation loops) [3–8]. As for the effects on mechanical properties, the irradiation-induced defects and clusters cause hardening and brittleness [3,7,9], which prevent dislocation sliding motion on its slip plane during the plastic deformation process. These property changes affect the lifetime and reliability of W as a PFM.

Rhenium (Re) and osmium (Os) are known elements that transmute from W by neutron capture reaction. For example, Re forms from W to 6% in the first wall and 3% in the divertor [10]. Meanwhile, neutron irradiation by fusion reactors such as ITER and DEMO could not be simulated because a neutron irradiation facility, which has enough flux of 14 MeV energy neutron, is not presently available; therefore, neutron irradiation researches on materials are conducted in fission reactors.

The amounts of transmuted elements from W are affected by the thermal neutron flux in the fission reactors because cross sections of the neutron capture reaction of W are significant in the lower neutron energy region. In our previous works, irradiation hardening behavior of W were studied using two different types of reactors; a mixed spectrum reactor such as High Flux Isotope Reactor (HFIR) in the USA, and a fast experimental reactor Joyo in Japan. HFIR has a higher thermal neutron flux, and the irradiation to high dpa level (above about 1 dpa) causes significant irradiation hardening compared to the irradiation by Joyo, which has a lower

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https://doi.org/10.1016/j.jnucmat.2018.04.031
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thermal neutron flux [11]. These results show that defect cluster formation and irradiation hardening behavior at higher irradiation levels depend on the irradiation reactors. The significant hardening of W is considered to be caused by W-Re precipitates such as σ(WRe) and χ(WRe_3)-phases formed during irradiation [12–16]. On the other hand, it is well known that the solubility limit of Re in W under unirradiated condition is 25% [17]; therefore, these precipitates are considered irradiation-induced precipitates. These precipitates have very high hardness in unirradiated conditions [18]; therefore, the formation of these precipitates may affect the irradiation hardening of irradiated W samples. Microstructural observation of pure W, W-5, -10, and-26%Re was conducted and irradiation hardening was measured after neutron irradiation to 0.17 dpa at 400 °C, 0.37 dpa at 500 °C, 0.40 dpa at 740 °C, 0.96 dpa at 538 °C, and 1.54 dpa at 750 °C [3,13–15,19]. In order to investigate the relationship between the defect microstructure and irradiation hardening, the irradiation hardening was evaluated TEM (Transmission Electron Microscopy) with the equation $\Delta H_v = 6\alpha\mu b(Nd)^{1/2}$ summarized by Moteff et al. [20], where N is the number density of defect cluster, d is the diameter of defect cluster such as voids, loops, and precipitates, α is the constant that depends on the type of defect cluster, b is the burgers vector, and μ is the modulus of rigidity [4,13,20] of the examined material. It has been reported that discrepancy exists between the calculated and measured irradiation hardening values by Vickers hardness measurement in medium level (0.5–1 dpa) irradiated pure W and W-Re alloys [13,14,21]. Possibility of invisible defect clusters by TEM was proposed [22]. It has also been considered that invisible Re clusters would accelerate the irradiation hardening of neutron-irradiated W by forming Re-enriched clusters in bcc-W. For these reasons, to understand the W irradiation behavior under fusion reactor environment using fission reactor irradiation, it is necessary to clarify the effect of transmuted Re aggregation behavior on the displacement damage structure development of W.

Atom probe tomography (APT) is one of the excellent research techniques to quantitatively study nanometer-sized three-dimensional spatial distribution of tracer level impurity elements. Small Cu-rich clusters formation in neutron-irradiated fission reactor pressure vessel steels and doped impurity element distribution in semiconductor devices have been reported [23–26]. The purpose of this work is to investigate invisible Re-enriched cluster formation in neutron-irradiated W. APT technique was applied to the neutron-irradiated W and W-Re samples in two different types of reactors, which had different neutron energy spectra, after the same level of irradiation at similar irradiation temperatures.

2. Experimental procedure

Pure W and W-10Re specimens, which were irradiated to 0.96 dpa in Joyo and irradiated to 0.90 dpa in HFIR, were used for this study to compare with the specimens irradiated in different transmutation rate levels. The reason was that transmutation calculation of our previous work showed 1dpa irradiation in HFIR induced approximately 10%Re in pure W [16].

The examined specimens were neutron-irradiated and the details of chemical compositions and fabrication processes of these specimens have already been published in previous works [14,19]. The neutron irradiation experiments were carried out in Joyo and HFIR approximately 10 years ago. Irradiation in Joyo was carried out in the places of row 6 in the reactor core, while irradiation in HFIR was carried out at RB* position of HFIR in Helium atmosphere. The irradiation conditions are shown in Table 1. The irradiation temperature, neutron fluence ($E_n > 0.1$ MeV), and displacement damage were 538 °C, 7.8×10^{25} n/m^2, and 0.96 dpa in Joyo and 500 °C, 6.0×10^{25} n/m^2, and 0.90 dpa in HFIR, respectively. The calculations of displacement damage were conducted using JENDL-3.2 and NPRIM-1.3 code with a displacement threshold energy of 90 eV [27]. Irradiation temperatures were measured by SiC T monitor in HFIR and TED (Thermal Expansion Difference Temperature Monitor) in Joyo.

Table 1
Neutron irradiation conditions.

Capsule	Temp.(°C)	Fluence (10^{25}n/m^2) ($E_n > 0.1$ MeV)	dpa[a]
JOYO			
JNC-63	538	7.8	0.96
HFIR			
RB*-14J	500	6.0	0.90

[a] 1 dpa = 8×10^{25} n/m^2 ($E_n > 0.1$ MeV), $E_d = 90$ eV.

Microstructural observations of the irradiated specimens were conducted by TEM in our previous works [15,16]. The specimens for APT were prepared from the previously observed TEM disks. The needle-like specimens were prepared by using focused-ion beam (FIB) systems (Helios NanoLab 600i or Quanta 200 3D, FEI) at IMR, Tohoku University, Oarai. APT was conducted with 3D-LEAP 4000X HR, CAMECA, which has a reflectron lens that prolongs the time of flight. Since the flight time can be matched with the kinetic energy of each ion, the total detection efficiency is 42%. The temperature of the sample stage was 55 K and analysis was conducted with the laser assist mode. The laser pulse energy and repetition rate were 100 pJ and 100 kHz, respectively. To confirm the segregation of Re, isoconcentration and segregation analyses were performed. The concentration (in mass percent) of Re and Os were obtained from the number of atoms based on the AP data.

To verify the concentration of W, Re, and Os in the specimens, the mass spectrum was obtained by counting the peak of each mass from 185 to 189. The natural abundances of isotopes of W, Re, and Os were evaluated by unirradiated pure W, W-10Re, and W-5Os alloys using the mass spectrum data. The results obtained by APT agreed with the well-known database [28]. The calculated neutron transmutation ratio and the obtained concentration by APT of the whole needle was compared to investigate the differences in concentration and isotopic ratio changes. The calculation of transmutation was performed using the EASY (European Active System) code developed by the Euratom UKEA Fusion Association. The code system EASY conducts analysis using EASI-2001 using an inventory code of FISPACT. Input to the FISPACT code is mainly used for calculating the irradiated material composition and neutron energy spectrum. The concentrations of transmutation elements were calculated including the cooling period after the irradiation experiments.

To confirm the segregation of Re, isoconcentration and segregation analyses with maximum separation algorithm were performed [29] using the analysis program IVAS. To investigate the existence of Re clusters, the maximum separation algorithm was used to identify the clusters. This method used D_{max}, which is the distance between two solute atoms. The clusters that did not contain specific number (N_{min}) of solute atoms (Re and/or Os) were not considered a cluster. The combination of D_{max} and N_{min} of each specimen was different from each other. Because the chemical composition of each specimen was different, the size and concentration of the clusters were obtained by the envelope and erosion method [29]. To confirm whether the solute atoms clustered or not, the results of envelope and erosion method and isoconcentration analysis were compared.

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