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Investigation of mechanical properties of stress-relieved and electron-irradiated tungsten after hydrogen charging

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ABSTRACT

The effect of hydrogen on the hardness and tensile properties of pure tungsten was examined using Vickers hardness and tensile tests. Samples were exposed to high-pressure hydrogen gas (5.8 MPa). The tensile behavior, tensile fracture surface, and hardness of as-received and stress-relieved tungsten did not change after hydrogen charging, owing to the low solubility of hydrogen. Therefore, to understand the effect of hydrogen on these materials, experiments must be performed to trap more hydrogen atoms at dislocations. In contrast, the hardness of electron-irradiated tungsten increased after hydrogen charging. Additionally, after a heat treatment at 473 K, hydrogen atoms dissociated from single vacancies, and the hardness decreased to the pre-charged value. Thus, single vacancies decorated with hydrogen atoms are expected to obstruct dislocation motion.

1. Introduction

In a fusion reactor, plasma-facing materials (PFMs) must withstand the damage caused by plasma-borne neutrons [1], hydrogen atoms [2], helium atoms [3] and heat loads [4]. Therefore, a high melting point, high thermal conductivity, and low sputtering erosion are required for PFMs. High-Z materials such as tungsten have been employed as PFMs owing to their thermal properties and resistance to erosion [5,6]. Additionally, hydrogen isotopes penetrate PFMs upon exposure to a fusion plasma [7]. Irradiation-induced defects capture the hydrogen isotopes, which are retained by the materials [8–10]. In tungsten, hydrogen solubility is very low, and defects bind to hydrogen atoms very strongly [11]. For example, Ogorodnikova et al. reported that the binding energies of deuterium to dislocations, vacancies, vacancy clusters, and voids were 0.46 eV [12], 1.06 eV [13], 1.06 eV [14], and 1.4 − 1.9 eV [13,15,16], respectively. Therefore, it is especially important to investigate the interaction between hydrogen and tungsten defects.

The retention of hydrogen isotopes typically degrades the mechanical properties of materials, causing effects such as hydrogen embrittlement [17,18]. If tungsten is widely used for fusion reactor components, hydrogen embrittlement of tungsten as structural materials may become critical. Several mechanisms of hydrogen embrittlement, for example, hydrogen-enhanced localized plasticity [19], hydrogen-enhanced decohesion [20], and the hydrogen-enhanced, strain-induced vacancy model [21] have been suggested, but a detailed description of the mechanism has not yet been identified. By molecular dynamics simulations, Yu et al. showed that hydrogen atoms promote the motion of dislocations in tungsten [22]. Terentyev et al. investigated the surface hardness of tungsten exposed to high-flux deuterium plasma by nanoindentation [23]. Plasma-induced defects were demonstrated to obstruct the motion of dislocations. In this study, hydrogen atoms were charged on electron-irradiated and stress-relieved tungsten, and their effect on the hardness and tensile behavior of tungsten was studied to clarify the mechanism of hydrogen embrittlement.

2. Experimental procedure

High-purity tungsten samples (99.95%, A.L.M.T. Corp.) were used in this study, and Fig. 1 shows the shape of the samples used in the tensile tests. Samples for tensile tests were cut from a 0.1-mm-thick sheet using a wire electric discharge machine. To release stress, the tungsten was annealed under two different conditions (at 1173 K for 1 h and at 1273 K for 5 h under vacuum ($<10^{-4}$ Pa)). For the electron irradiation tests, 5-mm-diameter samples were cut from a 0.2-mm-thick

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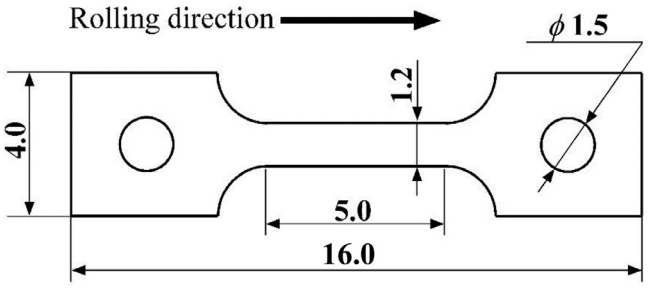

Fig. 1. Shape of samples for tensile tests. Thickness was 0.1 mm.

sheet using a wire electric discharge machine. Then, they were annealed at 1773 K for 1 h in vacuum ($<10^{-4}$ Pa) to allow recrystallization. Electron irradiation was performed using the electron linear accelerator of the Research Reactor Institute at Kyoto University. The electron acceleration voltage was 8 MV. The irradiation doses were 1.4×10^{21}, 4.2×10^{21}, 1.4×10^{22}, 3.0×10^{22}, and 6.5×10^{22}/m^2 (1.4×10^{-5}, 3.8×10^{-5}, 1.4×10^{-4}, 2.9×10^{-4}, 6.4×10^{-4} dpa). To calculate doses, an atomic displacement cross-section of 70.4 barns and a displacement threshold energy of 84 eV were used [24]. The irradiation temperature was 363 ± 10 K, which was maintained with water cooling. All samples were electropolished after electron irradiation to remove the oxidized layers formed during water cooling.

The hydrogen atoms were charged to the samples by exposing them to hydrogen gas at a pressure of 5.8 MPa and a temperature of 573 K for 240 h. Under these conditions, the hydrogen concentration of tungsten according to Sieverts' law is 1.12×10^{-11}, which is so low that hydrogen bubbles or platelets are not formed. Before hydrogen charging, the electron-irradiated samples were annealed at 573 K for 240 h in a vacuum to prevent the formation of vacancy clusters during hydrogen charging. The microscopic Vickers hardness $Hv_{0.1}$ was measured at 298 K with a load of 0.1 kg•f using a HMV-2T (Shimadzu Corp.), applied for 10 s. The average values and standard deviations of the hardness in 10 tests are plotted below. Different machines with the same model number were used for the hardness tests discussed in Sections 3.1 and 3.2. Tensile tests were performed at 298 K with a strain rate of 2.9×10^{-4}/s and a 500 N load cell. The tensile test machine was manufactured by INTESCO Co., Ltd. Results of the tensile tests of recrystallized and electron-irradiated tungsten were not obtained because the samples were broken while affixing them to jigs. Hydrogen-charged samples were cooled with liquid nitrogen unless otherwise specified in the description of the experimental tests above.

3. Results and discussion

3.1. As-received and stress-relieved tungsten

Fig. 2 shows the stress–strain curves in as-received and stress-relieved tungsten. Table 1 gives the 0.2% proof stress and tensile strength of as-received and stress-relieved tungsten with and without hydrogen charging. The 0.2% proof stress and tensile strength decreased with increasing annealing temperature during stress release. Both also slightly decreased after hydrogen charging in all samples, but the change was negligible. Therefore, it was unlikely that the observed change was due to hydrogen charging.

Fig. 3 shows the change in Vickers hardness in as-received, stress-relieved, and recrystallized tungsten before and after hydrogen charging. The Vickers hardness of the recrystallized tungsten was higher than that obtained in a previous study [25]. The tensile strength and Vickers hardness of the recrystallized tungsten have been reported to be 1070 MPa and 380 kgf/mm^2, respectively [25]. The application of a heat treatment at 1773 K for 1 h here was not sufficient to obtain perfect recrystallization. The hardness decreased as the annealing temperature increased during stress release. Fig. 4 shows SEM images of the

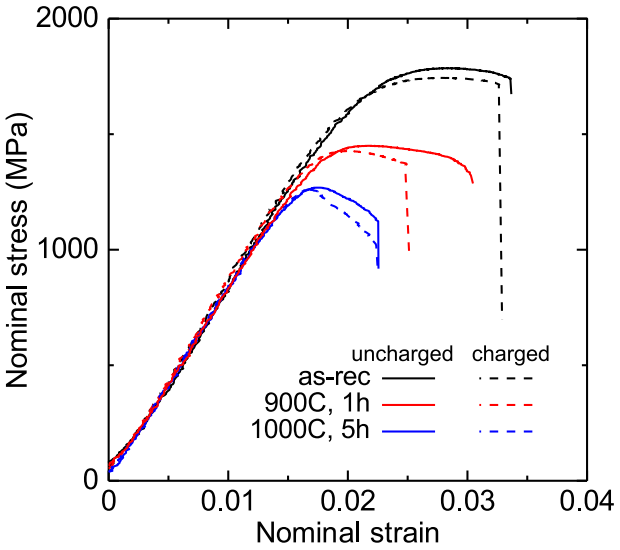

Fig. 2. Stress–strain curves of as-received and stress-relieved tungsten. Stress release was performed at 1173 K for 1 h and at 1273 K for 5 h.

Table 1
0.2% proof stress ($\sigma_{0.2}$), tensile strength (σ_{TS}) of as-received, and stress-relieved tungsten with and without hydrogen charging. Stress release was carried out at 1173 K for 1 h and at 1273 K for 5 h.

		Non-charged		Hydrogen charged	
		$\sigma_{0.2}$ (MPa)	σ_{TS} (MPa)	$\sigma_{0.2}$ (MPa)	σ_{TS} (MPa)
As-received		1677	1786	1659	1744
Stress relieved	1173 K, 1 h	1418	1450	1407	1427
	1273 K, 5 h	1264	1269	1258	1261

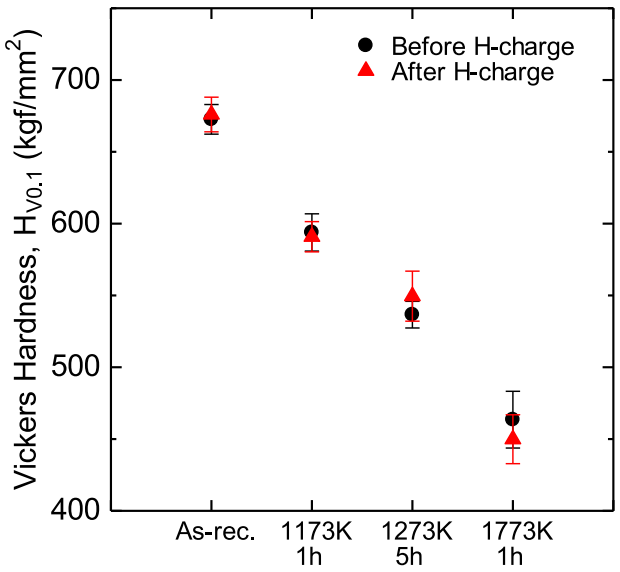

Fig. 3. Change in Vickers hardness in as-received, stress-relieved, and recrystallized tungsten after hydrogen charging.

surface of as-received tungsten, tungsten annealed at 1173 K for 1 h, tungsten annealed at 1273 K for 5 h, and tungsten annealed at 1773 K for 1 h after hydrogen charging. SEM observations were not performed before hydrogen charging; however, the same sample was used for hardness tests before and after hydrogen charging. The grain size did not change with increasing annealing temperature up to 1273 K, and substantially increased after the annealing at 1773 K. These results