§82. Microstructure and Thermal Desorption of Deuterium in Irradiated Pure Tungsten

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Introduction

Tungsten (W) and its alloys are primary candidates for plasma-facing materials in fusion reactors owing to properties such as low sputtering yield, low hydrogen permeability, and high melting temperature. In the present study, the interaction between D and radiation-induced defects in pure W was experimentally investigated by comparing the spectra of implanted D obtained using thermal desorption spectroscopy (TDS) and the microstructures of samples subjected to different heat treatments and levels of mechanical deformation.

Results[1)]

A pure W sheet obtained from Nilaco Co. Ltd. in Japan (0.1 mm-thick, 99.95% purity) was used in this study. The major impurities of the sheet are Mo(<300ppm), Fe(<50ppm), C, Si, O. Ca and Co(<30ppm). Samples for TEM observation and TDS analysis were punched from the sheet. To estimate the effects of the dislocation density on the desorption behavior, the as-received (AR-W) samples were annealed at 1173 K for 10 min, 1173 K for 30 min, 1223 K for 30 min, or 1273 K for 30 min. The samples were also stress-relieved at 1200 K for 3 h (SR-W) and re-crystallized at 2300 K for 20 min (RC-W). Heavy ion (Cu^{2+}) irradiation was performed at room temperature up to a dose of 2 displacements per atom (dpa). The peak damage region of 400 nm and the displacement damage distribution up to approximately 600 nm were calculated using the SRIM code with displacement energy of 55 eV.

Before and after ion irradiation, exposure of the samples to 2 keV-D_2^+ ions was performed at room temperature up to a dose of 1×10^{21} D_2^+/m^2 in an ultra-high vacuum evacuation apparatus equipped with a small duo-plasma-type ion gun. After exposure, the samples were transferred to the TDS apparatus, where the thermally desorbed D gas was measured with a quadruple mass spectrometer. Desorption rate for D was calculated from the data obtained for the D_2 and DH molecules. The temperature of the specimens was raised to 1000 K at a ramp rate of 1 K/s during this analysis. To understand the role of dislocations (pre-existing and introduced by cold working) in the desorption behavior of D, the AR-W samples were annealed in the temperature range of 1173–2300 K under vacuum conditions. Fig. 1 shows the microstructures of the samples used in the present study. The highly accumulated strain and dislocations in the grains decreased after annealing above 1273 K. Notably, the pre-existing dislocations observed in the AR-W sample annealed at 2300 K for 20 min disappeared, and the sample was fully re-crystallized. Fig 2 presents the (a) D_2 and (b) DH thermal desorption spectra obtained for these samples after exposure to 2.0 KeV- D_2^+ ions up to 10^{21} D_2^+/m^2 at room temperature. As can be seen in the figure, the peaks in the spectra can be classified into groups A, B, and C, each of which may have sub-peaks. Peak A was detected in the temperature range of 330–420 K. In this temperature range, most of the D was desorbed as D_2, and trapping of the D increased after heavy deformation but decreased following pre-annealing above 1173 K. In addition, the cold working (rolling) of a re-crystallized sample led to an increase in peak A. Single vacancies and dislocations are known to be induced by cold working. Therefore, peak A appears to be controlled by the density of dislocations and single vacancies induced by cold working. Peak B was detected in the temperature range of 420–560 K and appeared in both the D_2 and DH spectra and disappeared after annealing up to 1273 K. Peak C was detected only in the DH spectra in the temperature range of 660–900 K and disappeared after annealing above 1223 K. Furthermore, retention of D was significant for peaks A and B but not for peak C. Ion irradiation and exposure to D were performed at room temperature. As the irradiation dose of Cu ions increased, the trapping of peaks A, B, and C also increased. In particular, the trapping of peak A at 0.1 dpa and peak C at 1.0 dpa was remarkable.

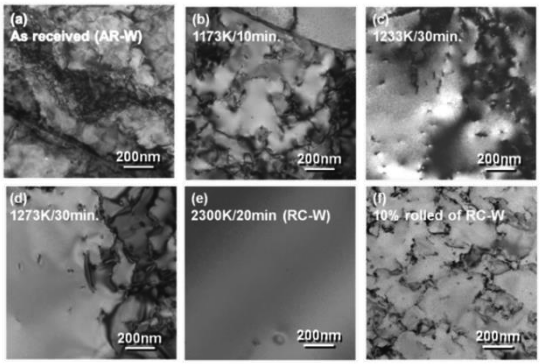

Fig.1 Microstructure of pure W used in the present study. (a) as-received sample (AR-W) (b) annealed at 1173 K for 10 min. (c) annealed at 1233 K for 30 min (d) annealed at 1273 K for 30 min. (e) annealed at 2300 K for 20 min. (RC-W) (f) recrystallized at 2300 K and 10% cold worked Dislocation loop formation of pure W irradiated in the temperature range of 300K to 1073K

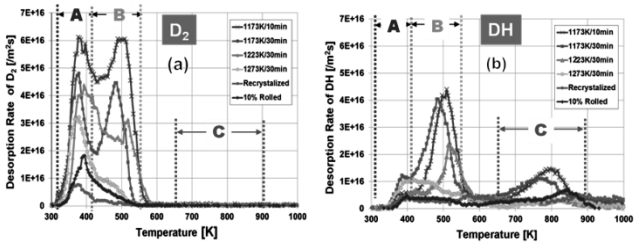

Fig. 2 Thermal desorption spectra for (a) D_2 and (b) DH from pure W exposed to 2 keV-D_2^+ ions up to a dose of 1×10^{21} D_2^+/m^2 at room temperature.

1) H. Watanabe, N. Futagami, S.Naitou, N.Yoshida, J. Nucl. Materials, 455 (2014) 51-55.