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In situ deuterium observation in deuterium-implanted tungsten

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ABSTRACT

In order to evaluate the tritium inventory in plasma-facing tungsten components of a fusion reactor, deuterium depth profiles in tungsten were observed in situ using nuclear reaction analysis (NRA) under continuous implantation of 3 keV D ions. Measurements were conducted at temperatures of 384, 473, 573 and 673 K. Recombination coefficients and rate constants for the surface recombination process were estimated from the observed deuterium concentration. It is indicated that the measured surface recombination rate constant is applied in a case wherein tungsten is exposed to hydrogen particles of various energies from a fusion plasma. The measured recombination coefficient was identical to that found by a different technique in a previous work. Deuterium in trap sites was found to contribute to deuterium retention in the samples as well as to deuterium in solution sites. The deuterium retention was low in the 384 K sample, in which trap sites had not appeared. Deuterium retention was very low in the 673 K sample, where most deuterium atoms were detrapped and desorbed. At an intermediate temperature of 473 K, the retention showed a maximum value due to a large occupancy of deuterium over many trap sites. The dependence of the retention on deuterium fluence was explained assuming that trap sites were produced by implantation.

1. Introduction

A divertor is one of the main plasma-facing components of a deuterium–tritium (DT) fusion reactor and is subjected to high heat and particle fluxes. Tungsten is a good candidate material for the divertor owing to its heat resistant properties and high threshold for physical sputtering. As the divertor is to be implanted with a radioisotope of tritium from the fusion plasma, the tritium inventory in the divertor is one of the primary considerations for the safe operation of a fusion reactor. For example, the accumulation amount of tritium in ITER is limited to 1 kg [1].

Many experiments have been performed in order to simulate tritium inventory by implanting keV deuterium ions into tungsten. Deuterium can be observed by thermal desorption spectroscopy (TDS) [2–4], nuclear reaction analysis (NRA) [2] and elastic recoil detection analysis (ERD) [5]. However, the experimental results can be rather complicated and difficult to interpret [3]. For a temperature of 300 K and a fluence of 1.0×10^{22} m^{-2}, saturation was observed by Haasz (1 keV-D_2^+) [2] but not by Ogorodnikova (200 eV-D^+) [4] and Nagata (10 keV-D_2^+) [5]. For 500 K and the same fluence, saturation was observed by Nagata but not by Haasz and Ogorodnikova. Deuterium retention at 500 K (1 keV-D_2^+) [2] has been reported to be much higher than that at 440 K (3 keV-D^+) [4]. These results indicate that deuterium retention depends not only on the deuterium fluence and the temperature but also on other conditions.

For the quantitative evaluation of retention, it is important to know the various interactions between tungsten and hydrogen, such as diffusion [6], recombination [7] and trapping [8]. In the present work, a similar experiment to that reported in Nagata's work [5] was conducted. A distinctive feature of our experiment is that deuterium depth profiles near the surface of tungsten have been directly observed by NRA under continuous implantation with deuterium ions. Effects of temperature and implantation fluence on the deuterium concentration and the surface recombination process are discussed in Section 3 and 4.

2. Experimental

The samples were tungsten disks with a diameter of 21.3 mm and a thickness of 1.0 mm which were obtained from A.L.M.T. Co with a purity is 99.97%. The main impurities in the samples were Mo-12, C-10, Fe-6, Al-2 and Cr-2 ppm by weight. The samples were stress-relief annealed at 1173 K for 0.5 h, mechanically polished, and re-crystallized at 1573 K for 1 h. One sample was set into a holder in a vacuum chamber, as shown in Fig. 1 and heated to 384 K. One side of the sample was implanted with 3 keV D ions from an ion gun (OMEGATRON, OMI−0045CKE) oriented at a 45°

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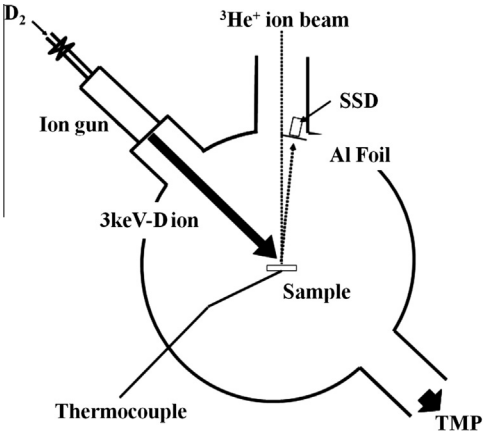

Fig. 1. Experimental setup for in situ deuterium observation.

angle to the surface of the sample. The diameter of the implanted area was 15 mm and the ion flux was 1.0×10^{18} $m^{-2}s^{-1}$.

During implantation, deuterium depth profiling by NRA was conducted with a 1.7 MeV $^3He^+$ beam obtained from the 4MV van de Graaff accelerator of Kyoto University. The analyzing beam was shaped into a circle with a 4 mm diameter by a round slit. The typical beam current and flux were 30 nA and 1.5×10^{16} $m^{-2}s^{-1}$, respectively. An incident beam angle was normal to the surface. The energy spectrum of protons emitted by the reaction D(^{3}He, p)^{4}He was converted into a deuterium depth profile near the surface [9]. The probe depth was 1.6 μm. Protons were detected by a solid state detector (SSD) located at a scattering angle of 174.3° and a solid angle of 1.08 msr. An aluminum foil with a small hole was set in front of the SSD to block backscattered ^{3}He ions. A small fraction of the ions backscattered through the hole was monitored in order to obtain the $^3He^+$ beam fluence. The beam fluence of each NRA run was limited to 2.7×10^{19} m^{-2} in order to minimize irradiation effects. Similar experiments were conducted with the three remaining samples at 473, 573 and 673 K.

3. Results

Depth profiles of deuterium in the sample maintained at 384 K are shown in Fig. 2. The D^+ ion is implanted in a very shallow region, at around 40 nm, as estimated by SRIM simulations [10]. Deuterium also diffuses from the implanted region into the bulk. As a result, the deuterium concentration in the bulk increases with deuterium fluence, as shown in Fig. 2. Implanted deuterium also diffuses to the surface and is absorbed there. Thus, there is a peak in the profile at 0 μm. A continuous increase in the concentration indicates that the system does not reach steady state conditions. However, at a fluence of 2.2×10^{23} m^{-2} the near surface concentration may be considered to be near steady state, since in this region, concentration profiles are flat.

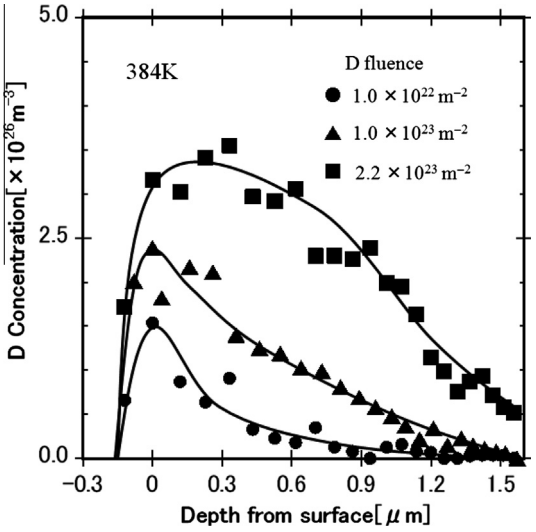

Fig. 2. Evolution of deuterium depth profiles in tungsten implanted with 3 keV D ions at 384 K.

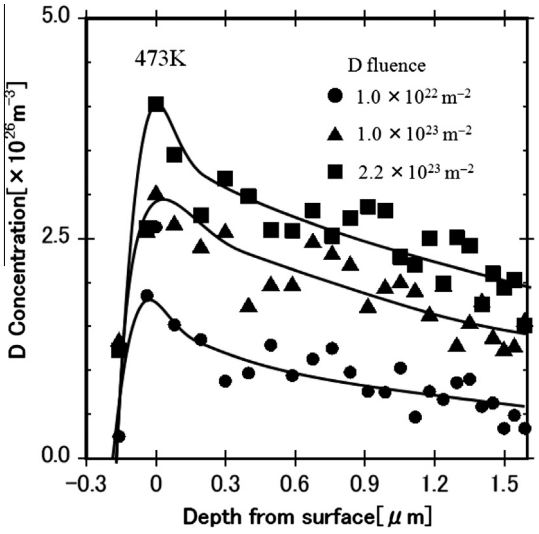

Fig. 3. Evolution of deuterium depth profiles in tungsten implanted with 3 keV D ions at 473 K.

Depth profiles of deuterium in the sample maintained at a temperature of 473 K are shown in Fig. 3. As in the case of Fig. 2, the deuterium concentration in this case increases with fluence. Compared to the profile at 384 K at a fluence of 1.0×10^{22} m^{-2}, the slope in the profile is smaller and the concentration is higher. The former observation is explained by the fact that the diffusion coefficient of deuterium at 473 K is nine times higher than that at 384 K [6]. In addition, deuterium can diffuse to greater depths. The deuterium concentration near surface mainly depends on the surface recombination and the incident fluxes since the diffusion flux toward the bulk region is very small. As the recombination coefficient at 473 K is higher than that at 384 K [11] and the incident flux does not depend on the sample temperature, the deuterium concentration in solution sites at 473 K should decrease. This point is discussed later.

The recombination of deuterium absorbed on the surface is now discussed. In the steady state, the incident flux of deuterium F is the sum of a permeation flux J through the sample and a reemission flux R from the ion implanted surface. F is considered to be much larger than J [11] and F can be regarded as R. The balance equation is expressed as,

$$F = R = k_2\theta^2, \tag{1}$$

where k_2 and θ are the rate constants for surface recombination process and deuterium surface coverage, respectively. The surface coverage is the ratio of deuterium occupancy on the surfaces and defined by

$$\theta = S/S_0, \tag{2}$$

where S and S_0 are the deuterium surface density and density of surface absorption sites, respectively. Considering a depth