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Hydrogen gas driven permeation through tungsten deposition layer formed by hydrogen plasma sputtering

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HIGHLIGHTS

- H permeation tests for W layer formed by H plasma sputtering are performed.
- H permeation flux through W layer is larger than that through W bulk.
- H diffusivity in W layer is smaller than that in W bulk.
- The equilibrium H concentration in W layer is larger than that in W bulk.

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ABSTRACT

It is important to evaluate the influence of deposition layers formed on plasma facing wall on tritium permeation and tritium retention in the vessel of a fusion reactor from a viewpoint of safety. In this work, tungsten deposition layers having different thickness and porosity were formed on circular nickel plates by hydrogen RF plasma sputtering. Hydrogen permeation experiment was carried out at the temperature range from 250 °C to 500 °C and at hydrogen pressure range from 1013 Pa to 101,300 Pa. The hydrogen permeation flux through the nickel plate with tungsten deposition layer was significantly smaller than that through a bare nickel plate. This indicates that a rate-controlling step in hydrogen permeation was not permeation through the nickel plate but permeation though the deposition layer. The pressure dependence on the permeation flux differed by temperature. Hydrogen permeation flux through tungsten deposition layer is larger than that through tungsten bulk. From analysis of the permeation curves, it was indicated that hydrogen diffusivity in tungsten deposition layer is smaller than that in tungsten bulk and the equilibrium hydrogen concentration in tungsten deposition layer is enormously larger than that in tungsten bulk at same hydrogen pressure.

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1. Introduction

Evaluations of tritium permeation rate to a coolant and tritium inventory in the plasma confinement vessel are important issues from a viewpoint of fusion safety. Sputtering and deposition are fundamental mass transfer phenomena between plasma and interface materials. Continuous long-time operation of fusion reactors would generate unignorable amount of deposition layers and dust even if the sputtering yield of plasma-facing material is low. Tungsten (W) is a primary candidate material for plasma-facing components because of high melting point, low sputtering yield and low solubility for hydrogen isotopes. In order to estimate tritium inventory in the vessel, it is necessary to evaluate a generation rate of deposition layers and tritium concentration in the layer. The present authors have investigated hydrogen or deuterium retention in W deposition layers growing under hydrogen isotope plasma exposure [1] and reported that hydrogen isotope concentration in W deposition layer depends on temperature and a ratio of hydrogen flux and tungsten flux to the growing surface of the layer [2]. Since hydrogen retention capacity of W deposition layer is several orders of magnitude larger than that of W bulk, the presence of W deposition layer on plasma-facing wall may affect fuel recycling and inventory. Most hydrogen implanted in W deposition layer migrates in the layer and leaves to plasma or penetrates to the wall bulk. In order to evaluate tritium behavior in the plasma facing wall on which W deposition layer is formed, diffusivity and solubility of hydrogen isotopes in W deposition layer are required. In this work, gas driven permeation of hydrogen through W deposition layers, which was produced by hydrogen RF plasma sputtering, was observed and permeation behavior of hydrogen was discussed.

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2. Experimental

2.1. Preparation of W deposition layer

W deposition layers were produced in hydrogen plasma by RF sputtering device. Several circular nickel plates, 21.17 mm in diameter and 20 μm in thickness, and square quartz plates, 10 mm or 5 mm × 5 mm in size and 1 mm in thickness were mounted on the ground electrode as substrates. After mounting substrates, the plasma chamber was evacuated to about 10^{-3} Pa by a vacuum pump and hydrogen gas, 99.99% in purity, was introduced via a mass flow controller. Hydrogen gas pressure and RF power were set to be 10 Pa and 100 W (13.56 MHz), respectively. The sputtering–deposition process was continued for 527 h, 950 h and 307 h and these samples were named $W_{d1\text{-}1}$, $W_{d1\text{-}2}$ and W_{d2}. Two sputtering devices were used in this work although the device configuration is the same. The chamber size used to produce W_{d2} was slightly larger than that used to produce $W_{d1\text{-}1}$ and $W_{d1\text{-}2}$.

2.2. Hydrogen permeation experiment

The experimental apparatus is schematically shown in Fig. 1. The nickel plates on which W deposition layer was formed were clamped between a copper gasket and a stainless steel flange. The flange connected to stainless steel tubes was inserted in a quartz tube and set at a center position of an electric furnace. The effective permeation area, A [m^2] was obtained to be 2.62×10^{-4} m^2 from a circular notch formed on the Ni plate by the flange. Dry argon gas, which was passed through a Molecular Sieve 3A bed to exclude impurity water vapor, was continuously introduced into the quartz tube during the permeation experiment in order to suppress the oxidation of metal materials in the heated region. The deterioration of rubber stoppers by heat was suppressed by air-cooling and water-cooling.

Argon gas, 99.99% in purity, was introduced into the primary side of the permeation cell via a mass flow controller. The secondary side was evacuated below 10^{-5} Pa by a turbo molecular pump. Then the permeation cell was heated to the preset temperature by an electric furnace. The temperature of furnace was controlled by thermocouple 2 contacting to the outer surface of the quartz tube. The sample temperature was measured by thermocouple 1 contacting to the sample surface from the primary side. Just after the secondary side was closed under vacuum condition, hydrogen gas or mixed gas of hydrogen with argon was introduced into the primary side. 1, 20% H_2/Ar gas and hydrogen gas, 99.99% in purity, were used for $W_{d1\text{-}1}$ and $W_{d1\text{-}2}$ and the hydrogen gas was used for W_{d2}. A pressure rise in the secondary side was measured by a diaphragm pressure gauge (Baratron, MKS). The slow pressure rise from the background was preliminarily measured when dry argon gas was input to the primary side before each experiment. The volume of the secondary side, V_2 [m^3] was measured preliminarily by pressure–volume–temperature method. Hydrogen permeation flux, J [mol/m^2 s] was obtained from the pressure rise, the effective permeation area, A and the volume of the secondary side, V_2, and it is expressed under steady-state condition as follows:

$$J = \frac{K}{L}(P_p^n - P_s^n) = \frac{D \cdot S}{L}(P_p^n - P_s^n), \quad (1)$$

where L is the thickness of the sample [m], K is the permeability [mol m/m^2/s/Pa$^{1/2}$], D is the diffusivity [m^2/s], S is the solubility [mol/m^3/Pa^n], P_p is the pressure of hydrogen in the primary side [Pa] and P_s is the pressure of hydrogen in the secondary side [Pa]. The exponent n has a value from 0.5 to 1.0 depending on permeation phenomena. When n is 0.5, the permeation is dominated by diffusion of hydrogen atom dissolved in the layer. When n is 1.0, the permeation is dominated by surface reaction or by diffusion of hydrogen molecule in the layer. Numerical calculations of one dimensional diffusion were performed assuming D and SP^n to fit the calculated pressure rise to experimental one. SP_p^n indicates the equilibrium concentration in the primary side and SP_s^n is that in the secondary side. In order to confirm the validity of experimental and analytical procedure of this work, the hydrogen permeation experiments for Ni plates, for which many literature data have been reported, were preliminarily performed.

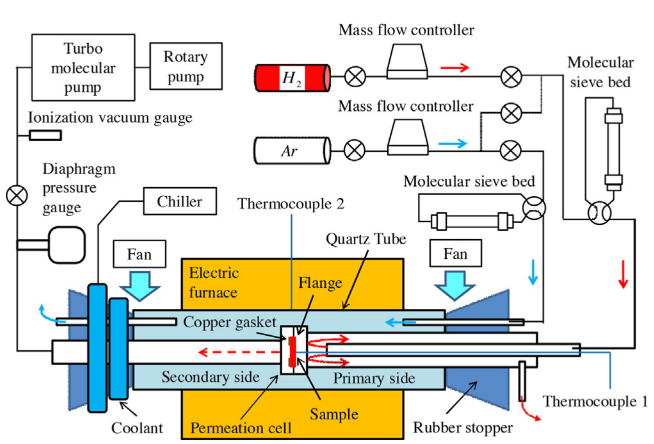

Fig. 1. Experimental apparatus for hydrogen permeation.

3. Results and discussion

The thickness of W deposition layer on Ni plates was estimated from that on quartz plates produced at the same time. The authors consider that the bulk structure of deposition layer is not influenced by the substrate structure because the surface diffusion of deposited atoms is insufficient under the deposition conditions. Fig. 2 shows SEM image of the cross section of W deposition layer, $W_{d1\text{-}2}$ formed on a quartz plate. The density was obtained from weight and volume of the deposition layer. The densities of $W_{d1\text{-}1}$, $W_{d1\text{-}2}$ and W_{d2} were obtained to be 11.4, 13.7 and 13.1 g/cm^3, respectively. The porosities were calculated to be 0.41, 0.29 and 0.32 from W density, 19.25 g/cm^3. It was found that three layers have different porosity. The pressure rise for the Ni plates with W deposition layer was considerably smaller than that for the bare Ni plate. This means that the rate-controlling step is the permeation through the W deposition layer.

In Fig. 3, the observed hydrogen permeation fluxes were compared with the fluxes through Ni plate [3,4], W bulk [5,6], W film [7,8] and Er_2O_3 coating [9], which were calculated by using literature data. Er_2O_3 coating is a candidate of tritium permeation barrier. The obtained permeability for Ni corresponds to literature values. This suggests that experimental and analytical procedures are valid. The hydrogen permeation flux through W deposition

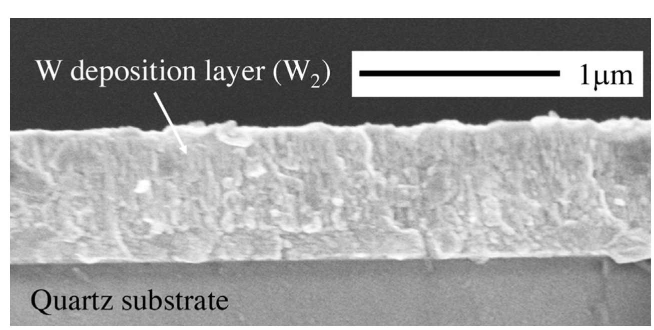

Fig. 2. SEM image of the cross section of $W_{d1\text{-}2}$.

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