



Contents lists available at ScienceDirect

Fusion Engineering and Design

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



Hydrogen isotopes transport in sputter-deposited tungsten coatings

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HIGHLIGHTS

- Hydrogen isotopes transport parameters for sputter-deposited tungsten (SP-W) have been evaluated.
- The obtained permeability for SP-W is comparable with the literature data for bulk polycrystalline tungsten (PCW).
- The apparent solubility and diffusivity are different from that of PCW by several orders of magnitude.
- Trapping effects on hydrogen migration have been discussed.

ARTICLE INFO

Article history:

Received 16 March 2017
Received in revised form 7 July 2017
Accepted 18 July 2017
Available online xxx

Keywords:

First wall
F82H
Tungsten coatings
Gas-driven permeation
Trapping effects

ABSTRACT

For a DEMO reactor, surface coatings made of tungsten are necessary to protect the plasma-facing wall made of reduced activation ferritic steels such as F82H. In this study, hydrogen and deuterium transport parameters for sputter-deposited tungsten coatings have been evaluated by the gas-driven permeation technique in the temperature range from 200 °C to 550 °C. The evaluated permeability for sputter-deposited tungsten is comparable with the literature data for bulk polycrystalline tungsten. However, the apparent diffusivity and solubility are different from that of bulk polycrystalline tungsten by several orders of magnitude, which is attributed to the presence of trapping sites resulting from the characteristic microstructure of sputter-deposited tungsten coatings. The trapping effects on hydrogen migration have been discussed.

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1. Introduction

Tungsten (W) has been proposed as a candidate plasma-facing material for the divertor of the International Thermonuclear Experimental Reactor (ITER) because of its beneficial properties such as high melting point, high thermal conductivity and low sputtering yield [1]. For a DEMO reactor, surface coatings made of W are necessary to protect the plasma-facing wall made of reduced activation ferritic steels such as F82H (Fe-8Cr-2W) [2]. The characterization of hydrogen transport in W coated F82H is of crucial importance to evaluate major reactor design issues including tritium retention, breeding feasibility and first wall particle recycling. There are a number of experimental data of hydrogen transport in bulk polycrystalline W (PCW) [3–8]. However, only limited experimental investigations aimed at understanding that of W coatings [9]. The purpose of this work is to evaluate the hydrogen and deuterium transport parameters for sputter-deposited W (SP-W) coatings

using the gas-driven permeation technique. The microstructure of SP-W has been characterized and the trapping effects on hydrogen migration have been discussed.

2. Experimental

The samples used in this work are bare F82H and SP-W coated F82H membranes which are prepared in the same dimensions as those commercially available conflat flanges with an outer diameter of 70 mm. The permeation area is 35 mm in diameter. Shown in Table 1 is the sample list. SP-W coatings are prepared by argon plasma, sputter-deposition technique at temperature of ~300 °C. The argon gas pressure is ~0.19 Pa. F82H substrate is mechanically polished before sputtering. Dense and pore-free W coatings can be produced by this technique. The density of W coatings is evaluated from the weight increase and film volume after W deposition, which is 19.2 g/cm³, ~99.5% of bulk W. Small samples with a size of 12 × 12 × 1 mm are also prepared for ex-situ analysis by scanning electron microscopy (SEM) combined with focused ion beam (FIB), energy dispersive X-ray spectroscopy (EDS) and X-ray diffraction (XRD).

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Table 1
Sample list.

Sample	Thickness of F82H (mm)	Thickness of W coatings (μm)
1	0.5	0.5
2	1.0	1.5
3	0.2	3.8
4	0.5	/
5	1.0	/

Hydrogen and deuterium gas-driven permeation (GDP) experiments are carried out using the VEHICLE-1 facility. Details of the experimental setup have already been presented elsewhere [10]. The background pressures of GDP upstream and downstream are $\sim 10^{-5}$ Pa and 10^{-6} Pa, respectively. Hydrogen GDP flux can be measured by a quadrupole mass spectrometer (QMS) on the downstream side. The gas pressure for GDP upstream is in the range of 10^3 – 10^5 Pa. A resistive heater is located beneath the membrane to control the sample temperature. All the samples are in-situ degassed at $\sim 500^\circ\text{C}$ for 8 h before experiments.

3. Theory

3.1. Diffusion limited GDP

According to the Fick's law, the one-dimensional hydrogen diffusion rate J is [11]:

$$J = -D \frac{\partial C}{\partial x}, \quad (1)$$

where D is the hydrogen diffusivity, $\partial C/\partial x$ is the hydrogen concentration gradient.

In the rest of the text, the hydrogen permeation through the membrane is assumed to be limited by hydrogen atom diffusion in the bulk while the adsorption, dissociation, recombination, and desorption processes at the upstream and downstream surfaces are rapid. In such case the hydrogen concentration at the upstream surface C is in equilibrium with hydrogen partial pressure P according to the Sieverts' law [12]:

$$C = S\sqrt{P}, \quad (2)$$

where S is the hydrogen solubility in the membrane material or the Sieverts' constant.

The upstream and downstream pressures are denoted by P_1 and P_2 . The downstream side is normally kept in high vacuum ($P_2 \approx 0$), hence the hydrogen concentration at the downstream surface is almost zero. The solution of Fick's law is [13]:

$$J(t) = \frac{DS\sqrt{P_1}}{L} \left[1 + 2 \sum_{n=1}^{\infty} (-1)^n \exp\left(\frac{-Dn^2\pi^2 t}{L^2}\right) \right], \quad (3)$$

where L is the membrane thickness. Steady-state permeation rate J_∞ is driven by the difference of the hydrogen concentration at the upstream and downstream surfaces. Hence, Eq. (3) becomes:

$$J_\infty = \frac{\Phi\sqrt{P_1}}{L}, \quad (4)$$

where Φ is the permeability and $\Phi = DS$. D and S are assumed to be independent of hydrogen concentration and time, being only a material property. The right term in Eq. (4) is valid also for multi-layer membranes if Φ represents the effective permeability coefficient Φ_{eff} .

Experimental evidence of diffusion limited permeation is $J_\infty \propto \sqrt{P}$ in contrast to $J_\infty \propto P$ which is characteristic for surface limited permeation regime. In general, the diffusion limited permeation regime is valid at high driving pressures P . More detailed conditions on validity of each permeation regime can be found in Ref.

[14,15] for single-layer membrane and in Ref. [16] for multi-layer membranes.

3.2. GDP through a two-layer membrane

Coating the membrane with an additional layer (barrier) always results in the reduced permeation if diffusion remains the rate limiting process. The experimental proof of the barrier efficiency is a relative reduction of the steady-state permeation flux measured at the identical conditions (P , T). Its definition is the ratio of the steady-state permeation flux through the uncoated membrane versus the one through the coated membrane, termed as the permeation reduction factor (PRF):

$$PRF = \frac{J_{\text{uncoated}}}{J_{\text{coated}}}. \quad (5)$$

The larger the PRF, the greater the efficiency of the coating material. Such membrane can be modeled as a membrane composed of two homogeneous layers. Its effective permeability Φ_{eff} is based on the sum of permeation resistances for each layer. For a two-layer membrane, the Φ_{eff} can be expressed as [17]:

$$\frac{L}{\Phi_{\text{eff}}} = \frac{L_1}{\Phi_1} + \frac{L_2}{\Phi_2}, \quad (6)$$

where indices denote layer 1 and 2, while the membrane thickness $L = L_1 + L_2$.

In contrast to the heat conduction where the temperature profile is continuous through the multi-layer membranes, the hydrogen concentration profile is not continuous through multi-layer membranes. Instead, the hydrogen chemical potential is continuous. Usually one suppose that the hydrogen concentration ratio at the interface is equal to the ratio of hydrogen solubilities in these materials [18]:

$$\frac{C_{1,i}}{C_{2,i}} = \frac{S_1}{S_2}, \quad (7)$$

where index i denotes the position at the interface.

By measuring the steady-state permeation fluxes of coated and uncoated membranes one can obtain the permeability for the coating material using Eqs. (4) and (6). Further determination of solubility and diffusivity requires measurements of transient permeation fluxes. The time-lag method can be used to evaluate the diffusivity. For single-layer permeation, the time-lag t_1 is directly related to its diffusivity as [19]:

$$t_1 = \frac{L_1^2}{6D_1}. \quad (8)$$

The time-lag for a two-layer membrane can be described as [20]:

$$t_2 = \left[\frac{L_1^2}{D_1} \left(\frac{L_1}{6D_1} + \frac{\alpha L_2}{2D_2} \right) + \frac{L_2^2}{D_2} \left(\frac{L_1}{2D_1} + \frac{\alpha L_2}{6D_2} \right) \right] \left(\frac{L_1}{D_1} + \frac{\alpha L_2}{D_2} \right)^{-1}, \quad (9)$$

where $\alpha = S_1/S_2$. The effective diffusivity D_{eff} can be obtained by measuring the time-lag t_2 of the two-layer membrane as $D_{\text{eff}} = (L_1 + L_2)^2/6t_2$. Then Eq. (9) can be reformed as [21]:

$$\frac{D_1}{D_2} = (1 + 3\beta\lambda) \left[(1 + \beta\lambda)(1 + \lambda)^2 \frac{D_2}{D_{\text{eff}}} - \lambda^2(3 + \beta\lambda) \right]^{-1}, \quad (10)$$

where $\beta = \Phi_1/\Phi_2$ and $\lambda = L_2/L_1$. Eqs. (9) and (10) are invariant to relative position of layers ($1 \rightleftharpoons 2$), hence the transient permeation as well as the steady-state permeation are not influenced by the order of the layers.

The solubility is usually obtained from the measured Φ and D using $\Phi = DS$.

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