

# Hydrogen Permeation through Sputter-Deposited Tungsten Coated F82H in QUEST

Yue XU<sup>1)</sup>, Arseniy KUZMIN<sup>2)</sup>, Yoshi HIROOKA<sup>1,3)</sup>, Kazuaki HANADA<sup>2)</sup>,  
Naoko ASHIKAWA<sup>1,3)</sup> and the QUEST group<sup>2)</sup>

<sup>1)</sup>The Graduate University for Advanced Studies, 322-6 Oroshi, Toki, Gifu 509-5292, Japan

<sup>2)</sup>Research Institute for Applied Mechanics, Kyushu University, 6-1 Kasuga-Koen, Kasuga, Fukuoka 812-8580, Japan

<sup>3)</sup>National Institute for Fusion Science, 322-6 Oroshi, Toki, Gifu 509-5292, Japan

(Received 20 April 2017 / Accepted 14 August 2017)

Hydrogen plasma-driven permeation (PDP) experiments have been performed using a sputter-deposited tungsten (SP-W) coated F82H membrane in the spherical tokamak QUEST. It has been found that SP-W coatings tend to enhance hydrogen PDP compared with that of bare F82H membrane. Surface recombination is a key process determining the PDP flux, suggesting that surface effects on hydrogen PDP should be further investigated.

© 2017 The Japan Society of Plasma Science and Nuclear Fusion Research

Keywords: QUEST, first wall, F82H, tungsten coating, plasma-driven permeation

DOI: 10.1585/pfr.12.1305038

In magnetic fusion devices, confinement loss particles via cross-field diffusion will bombard the first wall to induce plasma-driven permeation (PDP) (atomic hydrogen permeation included). Hydrogen isotopes (deuterium and tritium) transported into the breeding blankets by PDP will hinder the recovery efficiency of tritium and necessitate isotopes separation. Tritium permeation through the first wall may raise reactor safety issues as well. In addition, particle recycling from the plasma-facing wall would directly affect the core plasma performance [1]. Thus, it is crucial to measure the particle flux to the first wall. In our previous work [2], a permeation probe made of a reduced activation ferritic steel (RAFS) F82H was used to measure the hydrogen permeation flux in QUEST at Kyushu University. Those measurements were conducted during wall conditioning steady-state discharges. For a DEMO reactor, surface coatings made of refractory metals such as tungsten (W) are necessary to protect the plasma-facing wall made of RAFS such as F82H [3]. The characterization of hydrogen transport through W coated F82H under plasma exposure is of crucial importance to evaluate major reactor design issues including tritium retention, breeding feasibility and first wall particle recycling. In this work, a sputter-deposited tungsten (SP-W) coated F82H permeation probe has been installed in QUEST. The data have been compared with experimental results obtained by a bare F82H permeation probe [2] and the effects of SP-W coatings on hydrogen PDP are discussed.

Experiments are performed in the medium sized spherical tokamak QUEST [4] with the major and minor radii of 0.68 m and 0.4 m, respectively. Schematic view of the tokamak cross-section is shown in Fig. 1 (a). The work-

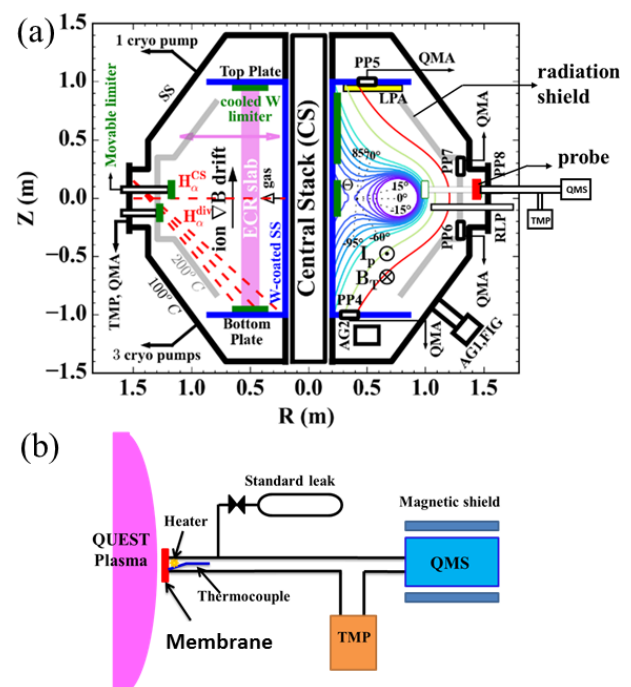


Fig. 1 (a) Schematic view of QUEST cross-section, and (b) schematic diagram of the permeation setup.

ing gas is hydrogen, supplied from a nozzle on the central stack. Gas puff is done in a pulse mode, the opening time of which varies in the range of 5 - 100 ms with the fueling rate of  $2.0 \times 10^{21} \text{ H s}^{-1}$ . The interval between puffs is in the range from 1 s to 50 s. The fueling gas pressure in the vessel is set in the range of  $1 - 10 \times 10^{-3} \text{ Pa}$ . Plasma is produced by electron cyclotron resonance (ECR) alone at 2.45 GHz (<10 kW). The annular slab plasma is used in

author's e-mail: xu.yue@nifs.ac.jp

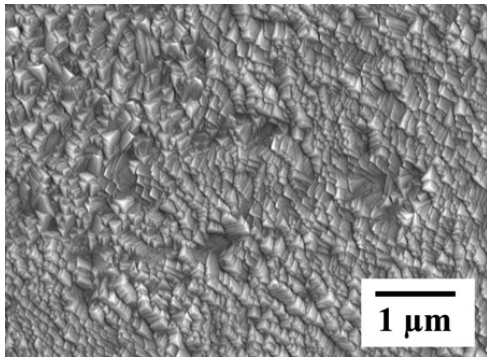


Fig. 2 SEM image of the SP-W coatings surface.

this study, indicated in pink in Fig. 1 (a). The plasma diameter can be adjusted by changing the toroidal magnetic field. The plasma parameters are measured by an array of Langmuir probes at the “top plate”. In addition, upper and lower oblique plates and vertical plates (radiation shield) are installed and denoted by grey lines. The vessel temperature is kept at  $\sim 100^\circ\text{C}$  during experiments.

Shown in Fig. 1 (b) is a schematic diagram of the permeation setup. In the present work, a SP-W coated F82H sample is used. The permeation area is 35 mm in diameter. The thicknesses of W coatings and F82H substrate are  $0.5\ \mu\text{m}$  and  $0.25\ \text{mm}$ , respectively. The permeation probe has been installed inside the closed port at the mid-plane and the membrane is  $\sim 15\ \text{cm}$  recessed from the radiation shield. A resistive heater is set behind the membrane to keep the sample temperature in the range of  $300\text{--}350^\circ\text{C}$ . The temperature is measured by a thermocouple attached to the downstream surface. The sample has been in-situ degassed at  $\sim 350^\circ\text{C}$  for 8 h before experiments. Hydrogen permeation flux is measured by a quadrupole mass spectrometer (QMS) which has been calibrated by a hydrogen standard leak. Shown in Fig. 2 is the surface morphology of SP-W coatings. The density of W coatings has been evaluated from the weight increase and film volume after W deposition, which is  $19.2\ \text{g/cm}^3$ ,  $\sim 99.5\%$  of bulk polycrystalline W. Characteristic microstructure of SP-W coatings has already been published elsewhere [5].

Hydrogen PDP flux through the SP-W coated F82H membrane has been measured under the wall conditioning 910 s discharge in QUEST. The hydrogen plasma is produced by the 2.45 GHz ECR system with an input power of 7.5 kW and the toroidal field coil current is kept at 17 kA. This condition is same with that of the previous work [2]. The steady-state permeation flux for the SP-W coated F82H membrane has been measured to be  $\sim 2.1 \times 10^{13}\ \text{H/cm}^2/\text{s}$  at  $300^\circ\text{C}$ .

In our previous work [2], the steady-state permeation flux was measured to be  $\sim 2.4 \times 10^{13}\ \text{H/cm}^2/\text{s}$  at  $270^\circ\text{C}$  using a 0.2 mm thick bare F82H membrane. Notice, however, that there was no radiation shield during that QUEST campaign. Therefore, to evaluate the effects of the radiation

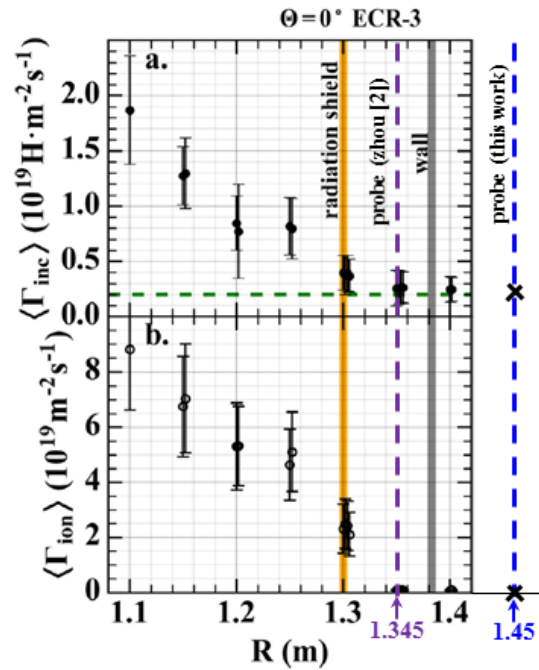


Fig. 3 Radial profiles of hydrogen incident flux ( $\Gamma_{\text{inc}}$ ) by a reciprocate PdCu permeation probe (indicated as PP8 in Fig. 1) and ion flux ( $\Gamma_{\text{ion}}$ ) by a reciprocate Langmuir probe (indicated as RLP in Fig. 1) in QUEST (reedit from Ref. [6]).

shield on hydrogen permeation, some calibration data are necessary. Shown in Fig. 3 are the radial profiles of hydrogen incident flux ( $\Gamma_{\text{inc}}$ ) measured by a reciprocate PdCu permeation probe (indicated as PP8 in Fig. 1 (a)) and ion flux ( $\Gamma_{\text{ion}}$ ) measured by a reciprocate Langmuir probe (indicated as RLP in Fig. 1 (a)) [6]. The PdCu membrane is selected for the probe calibration system from favorable viewpoints including the faster response time, wider permeation regime, and higher sensitivity independent of the incident hydrogen flux [6]. The characteristics of the PdCu permeation probe have already been experimentally investigated by Onaka *et al.* [7]. It has been found that hydrogen permeation through PdCu membrane is limited by the surface recombination process, in which case the steady-state permeation flux is proportional to the implantation (incident) flux.

In QUEST, two regions, i.e., the plasma region and inside the closed port section, are separated by the radiation shield at  $R = 1.3\ \text{m}$  (as shown in Fig. 3). For  $R < 1.3\ \text{m}$ , both  $\Gamma_{\text{inc}}$  and  $\Gamma_{\text{ion}}$  are smoothly increasing towards the slab plasma. The radial scale lengths and the relative radial profiles of  $\Gamma_{\text{inc}}$  and  $\Gamma_{\text{ion}}$  are consistent with each other, while a factor of 4-5 difference is shown. It should be noted that these radial profiles inside the plasma side reflect the radially diffusing plasma profile as well. There are steep gradients in both  $\Gamma_{\text{inc}}$  and  $\Gamma_{\text{ion}}$  near  $R = 1.3\ \text{m}$ , indicating that the diffusing plasma is terminated by the radiation shield around the torus. For  $R > 1.3\ \text{m}$ , the  $\Gamma_{\text{inc}}$  profile

is almost constant,  $\sim 0.2 \times 10^{19}$  H/m<sup>2</sup>/s, while  $\Gamma_{\text{ion}}$  immediately becomes zero. Thus, it can be concluded the ion flux is dominant in the plasma region ( $R < 1.3$  m) but the atomic hydrogen plays an essential role on the permeation behind the radiation shield or inside the closed port section ( $R > 1.3$  m).

In the present work, the permeation membrane is 15 cm recessed from the radiation shield. In the previous work, however, the membrane is 3.5 cm away from the out-board wall, the positions of the probes are shown in Fig. 3. It can be seen that without the radiation shield, the hydrogen incident flux  $\Gamma_{\text{inc}}$  measured by the PdCu probe is extrapolated to be  $\sim 0.8 \times 10^{19}$  H/m<sup>2</sup>/s at  $R = 1.345$  m, while with the radiation shield,  $\Gamma_{\text{inc}}$  at  $R = 1.345$  is reduced by a factor of  $\sim 4$  to be the value of  $\sim 0.2 \times 10^{19}$  H/m<sup>2</sup>/s. For hydrogen PDP taken in the RD regime (recombination-limited hydrogen release from the upstream surface and the diffusion-limited hydrogen release from the downstream surface) [2], the permeation flux  $J_+$  (atoms/cm<sup>2</sup>/s) is proportional to the square-root of implantation (incident) flux  $J_0$  (atoms/cm<sup>2</sup>/s) and inversely proportional to the membrane thickness  $L$  (cm):

$$J_+ = \frac{D}{L} \sqrt{\frac{J_0}{K_r}}, \quad (1)$$

where  $D$  (cm<sup>2</sup>/s) is the diffusion coefficient.  $K_r$  (cm<sup>4</sup>/s) is the hydrogen recombination coefficient on the plasma-facing surface.

To the first order of approximation, the membrane temperature (300°C in this work and 270°C in Ref. [2]) effects on  $D$  is negligible [8]. The membrane temperature effects on  $J_+$  for SP-W coated F82H has been shown in Ref. [5],  $J_+$  has been found to increase with decreasing the temperature, which means that  $J_+$  is supposed to be even higher than the present measured value for the membrane temperature at 270°C during experiments. Therefore, from Eq. (1) one obtains that the permeation flux will be reduced at least by a factor of  $\sim 2.5$  taking into account the effects of the incident flux and membrane thickness. The steady-state hydrogen permeation flux has been re-estimated to be  $\sim 5.3 \times 10^{13}$  H/cm<sup>2</sup>/s at 300°C in the present work.

Shown in Fig. 4 is a comparison of hydrogen PDP through the SP-W coated F82H and bare F82H membranes. The normalized PDP flux means that the different incident flux due to the radiation shield and the different membrane thickness have been considered so as to compare with the data in Ref. [2]. It can be seen that the permeation flux for bare F82H reaches steady-state within 100 s, while it takes  $\sim 700$  s for SP-W coated F82H. It is believed to be attributed to the trapping effects of W coatings on hydrogen migration which have already been under investigation and the details will be published elsewhere. The steady-state permeation flux through SP-W coated F82H in QUEST is about a factor of 2 higher than that through bare F82H, i.e., SP-W coatings enhance hydrogen PDP, which is in agreement with the data taken in the linear plasma fa-

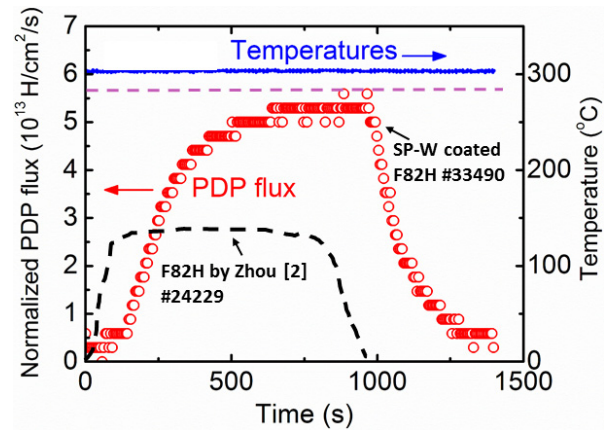


Fig. 4 Comparison of hydrogen PDP through the SP-W coated F82H and bare F82H membranes in a 910 s steady-state wall conditioning discharge. The thicknesses of SP-W coatings and F82H substrate are 0.5  $\mu\text{m}$  and 0.25 mm, respectively.

cility VEHICLE-1 except that the increase in permeation flux is relatively smaller. In VEHICLE-1, the permeation flux through SP-W coated F82H is about one order of magnitude higher than that through bare F82H in the temperature range of 300 - 550°C [5]. The difference could be due to the different particle bombarding energies in QUEST (no DC bias) and VEHICLE-1 (DC bias, -100 V). Besides, the permeation flux measured in QUEST is mainly a result of atomic hydrogen permeation, which is different from the plasma condition in VEHICLE-1. Nevertheless, further investigations are still needed to address this issue.

In this work, hydrogen permeation measurements have been conducted in the QUEST spherical tokamak for SP-W coated F82H membrane and the data have been compared with our previous results for bare F82H. A much longer breakthrough time and higher steady-state permeation flux have been observed for SP-W coated F82H. It is concluded that SP-W coatings enhance hydrogen PDP not only in the VEHICLE-1 linear plasma facility but also in the QUEST spherical tokamak.

This research is partly supported by the JSPS-NRF-NSFC A3 Foresight Program in the field of Plasma Physics (NSFC: No.11261140328, NRF: No. 2012K2A2A6000443).

- [1] J. Roth *et al.*, *J. Nucl. Mater.* **390**, 1 (2009).
- [2] H. Zhou *et al.*, *Plasma Fusion Res.* **9**, 340541 (2014).
- [3] T. Otsuka *et al.*, *J. Nucl. Mater.* **438**, S1048 (2013).
- [4] K. Hanada *et al.*, *Plasma Sci. Technol.* **13**, 307 (2011).
- [5] Y. Xu *et al.*, *Plasma Fusion Res.* **12**, 1205009 (2017).
- [6] A. Kuzmin *et al.*, *Nucl. Mater. Energy*, in press, DOI: 10.1016/j.nme.2017.03.027 (2017).
- [7] M. Onaka *et al.*, *Nucl. Mater. Energy* **9**, 104 (2016).
- [8] Y. Xu *et al.*, *Fusion Eng. Des.* in press, DOI: 10.1016/j.fusengdes.2017.04.022 (2017).