

Hydrogen absorption capability of a niobium panel for pumping neutral atoms in divertor region

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Abstract

The capability of a niobium panel to absorb atomic hydrogen was investigated for particle control application in divertors. The absorption rate (1.3×10^{20} H/m²s) stayed almost constant up to a dose of 4×10^{23} H/m² with no hydride formation occurring. The absorption panel can be easily regenerated. The absorbed hydrogen can be retained in the panel even after 48 h at room temperature. The absorption rate was found to decrease with panel temperature for $T < 200$ °C, remarkably dropping in the vicinity of room temperature. This is connected most probably with the formation of a multilayer impurity coverage. The multilayer carbon coverage deposited during the panel exposure in the divertor region of LHD decreased the absorption rate, but the panel properties could be completely restored by heating at 850 °C in spite of panel carbidization.

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1. Introduction

A pumping panel made of a Group Va metal (V, Nb or Ta) is one of the promising means for particle flux control in the divertor region. In steady-state regimes, a superpermeable membrane should be used for particle control [1–6], while a periodically regenerated absorption panel (simpler and cheaper) can be acceptable for an intermit-

tent operation in non-tritium devices [7]. The Group Va metals have a combination of properties that make them most suitable for the absorption pumping of hydrogen. Those properties are: (1) a highest mobility of absorbed hydrogen atoms ($D \sim 10^{-5}$ cm²/s) at low temperatures (<300 °C), (2) a reasonably high solubility of hydrogen, (3) a low critical temperature for hydride phase formation (e.g. 160 °C for Nb). Due to these properties hydrogen entering the metal at a low temperature (e.g. 20–300 °C) can be uniformly distributed over a large material depth (e.g. in a few mm). That is expected to ensure a high absorption capability (e.g. $\sim 10^{24}$ H/m²) of the panel with no hydride phase formation as well as fast panel regeneration at modest temperature.

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In the present study, hydrogen absorption capability of a niobium panel was investigated with thermal atomic hydrogen produced by hydrogen dissociation at an incandescent Ta wire. The effect of panel temperature on the rate of atomic hydrogen absorption was investigated. The panel was also installed into the large helical device (LHD) [8], and the effect of carbon deposition on absorption capability is discussed with an account of the results of surface analysis after the experiment.

2. Absorption capability of panel made of Va-group metals

The maximum absorption capability of the panel can be found from a balance equation $\xi_{\text{H}} j_i = (k_{\text{r0}} + k_{\text{rL}}) c_{\text{H}}^2$ where c_{H} is the concentration of absorbed hydrogen atoms, j_i the density of incident flux of hydrogen atoms, ξ_{H} their absorption probability, k_{r0} and k_{rL} recombination rate constants for both sides of the panel. Since the net absorption rate is zero at the balance, the maximum concentration of absorbed hydrogen compatible with a high pumping speed can be estimated from

$$\delta \xi_{\text{H}} j_i = (k_{\text{r0}} + k_{\text{rL}}) c_{\text{H}}^2, \quad (1)$$

where the factor δ should be much less than 1 (e.g. $\delta = 0.1$). Assuming that $k_{\text{r0}} = k_{\text{rL}}$, one can get

$$c_{\text{H}} = \sqrt{\frac{\delta \xi_{\text{H}} j_i}{2k_{\text{r0}}}}. \quad (2)$$

The recombination rate constant k_{r0} is connected with the sticking coefficient of thermal molecules α_0 via the detailed balance principle [7]: $k_{\text{r0}} = z\alpha_0/S^2$, where z is the gas kinetic theory coefficient and S is the solubility constant [9]. Therefore, the maximum absorption dose per unit panel area at a panel thickness L can be expressed by

$$G_{\text{H}} = c_{\text{H}} L = SL \sqrt{\frac{\delta \xi_{\text{H}} j_i}{2z\alpha_0}}. \quad (3)$$

In the case of a Nb panel of 1 mm thickness at 200 °C (the hydride phase does not form at $T > 160$ °C), G_{H} can be estimated as $\sim 10^{24}$ H/m² taking $\delta = 0.1$, $\xi_{\text{H}} = 0.25$ [7], $\alpha_0 = 10^{-5}$ [6] and $j_i = 10^{20}$ H/m²s.

3. Experimental

Fig. 1 shows a schematic view of the experimental device. An absorption Nb panel of 1 mm thickness and 414 mm × 444 mm surface area, which has a strip-type structure with the strip width of 11 mm, was installed in an UHV chamber. The panel can be resistively heated up to about 850 °C. The temperature was measured by using a thermocouple and an IR thermometer. A hydro-

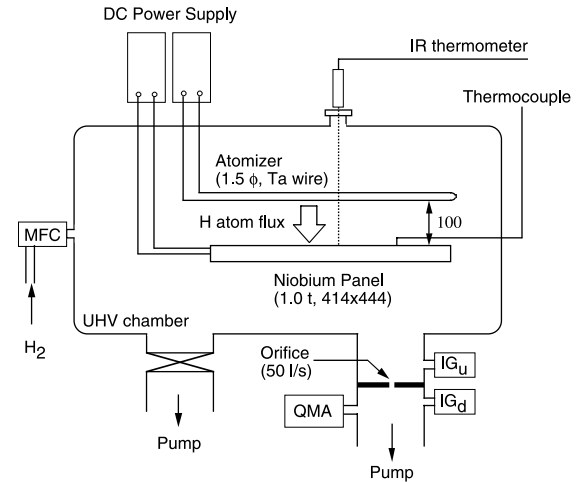


Fig. 1. Schematic view of experimental setup.

gen atom flux ($\sim 10^{20}$ H/m²s) onto the panel was produced by hydrogen dissociation at the surface of a Ta wire heated up to 1800 °C. The kinetic energy of hydrogen atoms is a few tenths of an eV, which is lower than that expected in divertor region in present fusion devices (a few tens of eV). The hydrogen gas is admitted into the chamber by a gas flow controller. The gas flow being absorbed/desorbed by the panel can be measured by using an orifice with a calibrated conductance (50 l/s for H₂).

4. Results and discussion

4.1. Absorption capability of Nb panel

Absorption of atomic hydrogen by the Nb panel causes a pressure drop as shown in Fig. 2. The absorption rate is increasing with the rise of atomizer (and panel) temperature and reaches 1.3×10^{20} H/m²s. Though the panel was heated by the radiation from the atomizer up to temperature as high as 450 °C, no noticeable decrease in the absorption rate due to hydrogen release from the panel was observed. The total amount of absorbed hydrogen gas was found by integrating the absorption rate with time (G_{abs}) and by measuring the amount of hydrogen gas desorbed by heating the panel (G_{des}). The hydrogen desorption was carried out by the panel heating at 680 °C just after hydrogen absorption (the total outgassing needed 15–20 min). Fig. 3 shows that both methods give similar amounts. That means that the pumping observed with atomizer heating is caused by absorption by the panel but not by the chamber wall nor other chamber components. Notice that the maximum absorption dose presented in Fig. 3 (4×10^{23} H/m²) was reached with no noticeable decrease in the absorption rate. The negligible reemission after

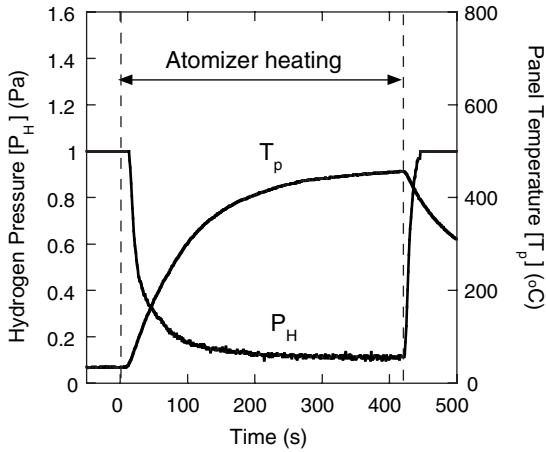


Fig. 2. Time history of hydrogen pressure and panel temperature. Before the atomizer turns on, hydrogen gas is admitted so as to keep the pressure 1 Pa H₂ at the constant pumping speed of 50 l/s (through the calibrated orifice). The panel temperature increases due to radiation from the atomizer.

such a significant dose at an elevated temperature (450 °C) is explained by a very low value of k_{r0} (and α_0) typical for the Nb surface covered by O segregating in the form of a monolayer from the metal bulk [6,7]. Hydrogen retention in the panel was also investigated by keeping the panel at room temperature and it was found that most of the absorbed hydrogen was retained in the panel even after 48 h as indicated by open squares in Fig. 3. One should also emphasize that the hydride phase is not formed at the maximum dose investigated (4×10^{23} H/m²) even after cooling the panel down to room temperature [9].

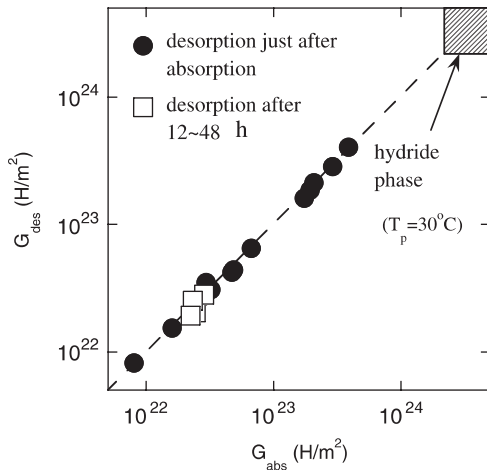


Fig. 3. Relationship between the total number of absorbed and desorbed hydrogen atoms per unit area of the panel.

4.2. Temperature dependence of absorption rate

We can see a big difference in the character of hydrogen pressure drop caused by the panel absorption at different initial panel temperatures (Fig. 4(a)). For the low temperature case (the initial panel temperature $T_{p0} = 34^\circ\text{C}$), the pressure gradually decreases with time (with increasing the panel temperature), while the pressure drops immediately for the high temperature case ($T_{p0} = 264^\circ\text{C}$). This suggests that the hydrogen absorption rate depends on the temperature of Nb panel. Fig. 4(b) shows the hydrogen absorption rate as a function of the panel temperature (T_p). The solid circles indicate the hydrogen absorption rate and the panel temperature 23 s after the atomizer was switched on – when the atomizer temperature comes to a constant level. In the high temperature region ($>200^\circ\text{C}$), we observed a high hydrogen absorption rate ($\sim 1.3 \times 10^{20}$ Hm²/s), which is the same order as the hydrogen atom flux density onto the panel. However, the absorption rate remarkably

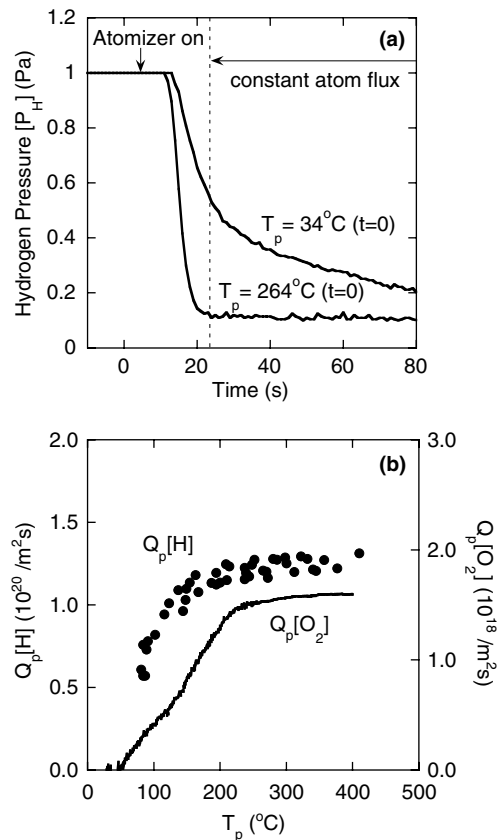


Fig. 4. (a) Absorption of atomic hydrogen at different initial temperatures of the panel, and (b) temperature dependence of absorption rate for hydrogen atoms and oxygen gas. The elevated panel temperature is obtained by resistive heating using electric current through the panel.

decreases in the low temperature range ($<200^{\circ}\text{C}$). We estimate that the absorption rate at room temperature is extremely low. This is caused most probably by the formation of a multilayer impurity coverage, which is not 'transparent' for thermal hydrogen atoms. At low temperature, the diffusion of impurities in the metal bulk is frozen, and all impurity atoms being deposited on the surface remain in the surface/subsurface zone. A radical decrease of absorption rate due to a polyatomic oxide film was observed in the case of interaction of atomic hydrogen with vanadium [10]. In order to observe the uptake of oxygen into the metal bulk, the absorption of oxygen gas was investigated by changing the panel temperature. As shown in Fig. 4(b), the temperature dependence of the absorption rate for oxygen is almost the same as that for hydrogen. This suggests that the dissolution of oxygen on the panel surface into the bulk is responsible for the recovery of hydrogen absorption rate at high temperatures. In order to understand this phenomenon in more detail, in situ observation of the panel surface for different panel temperatures will be required. In these experiments, we used thermal atomic hydrogen to estimate a lower limit of absorption capability. High-energy hydrogen atoms and ions are efficiently absorbed in the panel even at low temperatures as observed in glow discharge on LHD or in ion beam experiments. Therefore, a study of the dependence of absorption rate on energy of the incident hydrogen atoms will be required for applications in fusion devices.

4.3. Effect of carbon impurity on absorption rate

The panel tested in the above experiment was installed into the private region in LHD divertor and exposed to about 6500 plasma discharges. Then the absorption panel was extracted and tested again in the apparatus described above. The absorption rate after plasma exposure was found to have decreased to a value less than half of the previous one on account of carbonization of the panel surface [4]. However, the absorption rate was gradually restored by the panel heating at 680°C (the total time $\sim 5\text{h}$). It was completely recovered after heating at 850°C ($\sim 20\text{min}$). The surface analysis of the panel with Scanning Auger Electron Microscope (SAM) and X-ray diffractometer (XRD) were performed after the restoring. The layer-by-layer Auger analysis (Fig. 5(a)) indicates that the panel surface is covered with a thin carbon film, and Nb carbide (NbC) is formed beneath the carbon film with the thickness of several hundreds nanometers or more. On the other hand, the X-ray diffraction analysis shows that Nb_2C is the main carbide phase (Fig. 5(b)) and the thickness of Nb_2C layer is several micro-meters. From the above results, heating the panel to a temperature of 680°C for 10 min (used everyday for panel degassing in the LHD) is not enough for the conversion of carbon being deposited

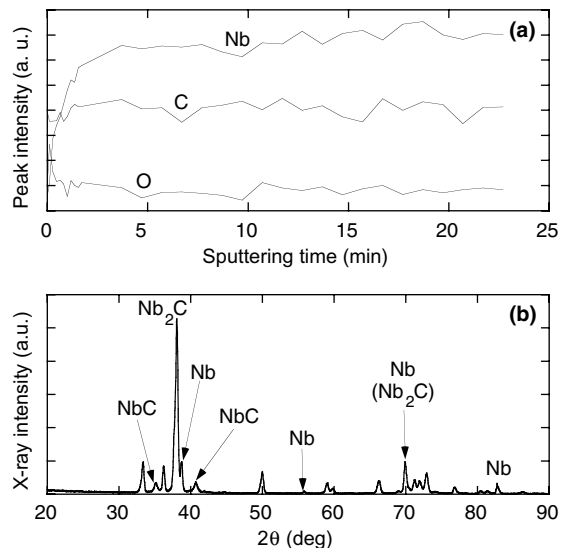


Fig. 5. (a) Depth profile of the panel surface obtained by SAM and (b) XRD ($\text{Cu-K}\alpha$ radiation) spectrum of the carbidized panel. In the XRD spectrum, other peaks are assigned to Nb_2C .

into carbide. Panel heating at a higher temperature (e.g. 850°C) is required for that. Taking into account that the absorption rate was restored by panel heating at 850°C , one can conclude that the multilayer carbide coverage has no influence on hydrogen atom absorption by Nb. This has also been observed in experiments on hydrogen superpermeation through a Nb membrane [11]. Thus the absorption panel is tolerable for carbon deposition and can be regenerated by heating to a high enough temperature.

5. Conclusions

Applicability of a panel made of a Group-Va metal for particle control in divertor region was investigated. The absorption of thermal atomic hydrogen with the rate of $1.3 \times 10^{20} \text{H/m}^2\text{s}$ remained virtually constant up to a dose of at least $4 \times 10^{23} \text{H/m}^2$ and most of absorbed hydrogen was retained in the panel even after 48 h at room temperature. The hydride phase was not formed even at 20°C . The absorbed hydrogen could be desorbed quickly at 680°C . A remarkable decrease of the absorption rate observed in the vicinity of room temperature is explained by the deposition of multilayer nonmetallic films at the panel surface. Carbidization (formation of NbC and Nb_2C layers) of the panel was found to have no influence on its absorption properties. From these results, Nb panel will be able to be used for pumping the neutrals in divertor region, if the panel is operated at a temperature between 200°C and 300°C so that a sufficient absorption rate is maintained.

Acknowledgments

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