ELSEVIER Journal of Nuclear Materials 271&272 (1999) 330–332

journal of nuclear materials

Trapping of deuterium by niobium at eV ion bombardment energies

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Abstract

The results of the experimental investigation and computer simulation of the deuterium trapping by niobium at the normal angle of incidence are presented. The primary energy of D atoms ranged from 2.5 to 30 eV. Depending on surface conditions, the trapping coefficient at the very low energies may vary several-fold in the experiment. Computer simulation based on the binary collision approximation model with taking into account the binding between the incoming ions and surface atoms gave a good agreement with the experiment. The effective binding energies of 0.3–3.0 eV were determined from comparison of the experiment and calculations. © 1999 Elsevier Science B.V. All rights reserved.

1. Introduction

Interaction of low energy hydrogen with fusion materials is of great interest, especially for eV energies which are relevant to the gas divertor regimes. But for this energy range, no experimental results are known and only computer simulation results exist. As it was shown earlier by the computer simulation [1], the surface binding energy plays a major role in hydrogen trapping at these energies. Although a large number of computer models exists, there is no an adequate description of the processes at the very low energies [2]. In this paper we represent the first experimental results of hydrogen trapping at energies comparable with the binding energy of the hydrogen on the surface.

Niobium was used for investigations. It is proposed as a material for superpermeation membranes [3] in the pumping area of the fusion reactor. Besides, niobium is a good material for modelling of hydrogen interaction with other hydrogen active materials like vanadium or its alloys widely discussed now for fusion applications.

2. Experiment

The experiments were carried out at the 'Medion-2' experimental set-up described elsewhere [4]. Briefly, the primary ion beam is separated in the electromagnet, decelerated by the retarding system and comes to the target. The screen, made from Ni, is mounted around the target and can be heated up to 350°C to prevent the trapping of deuterium on the chamber wall. A high-sensitivity mass-spectrometer is used to control the partial pressure of HD and D_2 molecules. To control the residual gas pressure, the monopole mass-spectrometer is used.

The trapping coefficient is determined by the thermal desorption technique (Fig. 1). The target is irradiated at room temperature where the re-emission of the retained particles is known to be negligible, and then it is heated up to 1300°C to release the captured deuterium. The number of the deuterium atoms released is supposed to be equal to the number of the deuterium ions trapped, and the trapping coefficient η is calculated as the ratio of the numbers of trapped and incident particles. The measurements were performed in the so called 'dynamic regime' where the release rate of the particles is proportional to the partial pressure of HD and D_2 molecules.

The sample used was made from high purity niobium manufactured by Daido Steel. Before the experiments, a

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PII: S0022-3115(98)00789-2

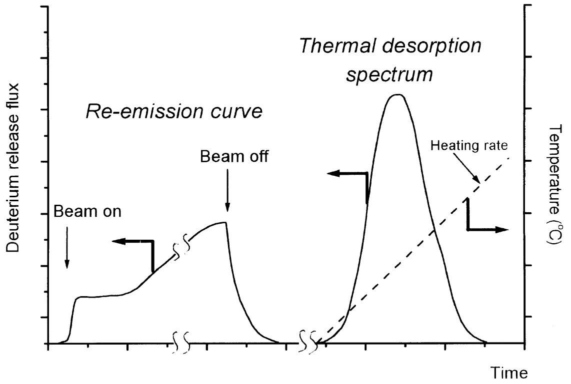

Fig. 1. The scheme of the experimental procedure of the trapping coefficient measurements.

surface impurity contamination analysis was performed using the secondary ion mass-spectroscopy, Auger spectroscopy, and low energy ion backscattering. It was found that the near-surface layer contains C (about 10%), O (5%), and Fe (2%) impurities and is covered by niobium oxide that can be removed by annealing up to 1400°C within few hours. Taking into account the residual gas pressure during the experiments (about 10^{-6} Pa), the oxide covers the surface within few minutes. Varying the time of irradiation and the time of annealing, we can vary the oxide coating on the surface.

We have done three series of the experiments. In the first one, the target was irradiated by D_3^+ ions with energies ranging from 15 to 90 eV. As the primary energy of ions is greater than energy of dissociation, we supposed that the energy of each deuteron is equal to one third the energy of the primary molecular ion. The fluence ranged from 10^{14} to 4×10^{14} D/cm^2. Because of the very low intensity of the ion beam in this energy range, the time of irradiation was rather high (tens of minutes) to accumulate the fluence necessary for measurements of the number of captured particles with reasonable accuracy. Surface was covered by NbO in this set of the experiments.

In the second and the third series, we bombarded the target by ArD$^+$ ions with the primary energies in the range of 50–400 eV. We supposed that ArD molecule dissociates as it strikes the surface, and the energy of D atoms is proportional to the ratio of D to ArD masses. So, using heteronuclear ions, it is possible to achieve extremely low energies for the light fragments of the molecule. It is necessary to note that the heavy component of the molecule has a much greater sputtering coefficient, and that can lead to cleaning of the surface during the experiment. Besides, the Ar atoms can, in principle, push deuterium into the bulk of the target due to collisions. The third series differed from the second by the long time annealing of the target before the measurements at temperature of 1400°C. The intensity of the ArD$^+$ at these energies was sufficient for TDS measurements of implanted deuterium (minimal necessary time of irradiation was about 2 min), and the fluence ranged from 2×10^{14} to 9×10^{14} D/cm^2.

3. Results and discussion

The experimental values of the trapping coefficient as a function of the primary energy of deuterons for the three series of measurements are shown in Fig. 2. In this figure we represented also the results of the computer simulations obtained by using the SCATTER program [5] based on the binary collision approximation (BCA). We calculated the particle trapping flux as the difference between the primary flux and the flux of backscattered particles. Calculations were performed both for NbO (solid lines) and Nb (dashed lines) to match the data obtained at different surface conditions. The surface binding energy E_S was considered to be an additional attractive potential in computations and was used as an adjustment parameter.

It is clearly seen that the experimental results in the three series differ significantly. For D_3^+ irradiation a weak minimum of the trapping coefficient was observed at $E_0 \approx 10$ eV. At lower energies η increases 1.8 times, and becomes equal approximately to unity at the minimum energy achieved. The computer simulations fit the experimental data at $E_S = 2.5 \pm 0.5$ for the NbO covered surface. For the purposes of practice it is important that for energies less than few eV, most of the particles are

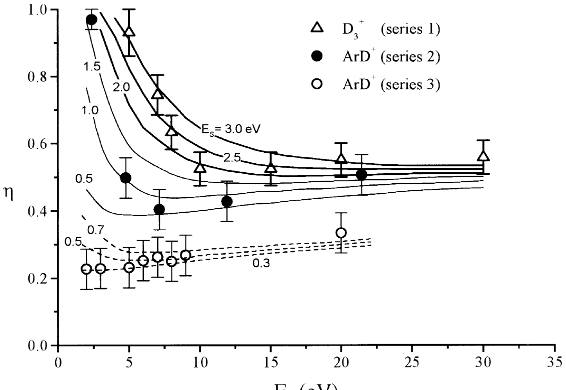

Fig. 2. The trapping coefficient of deuterium in Nb as a function of the primary energy of deuterium. Curves are computer calculations using a binary collision approximation: solid lines-calculations for NbO; dashed lines-calculations for Nb. Numbers at the curves are values of the 'effective' surface binding energy E_S, used as an adjusting parameter in computer calculations.

captured. So in this case, effective pumping of low energy hydrogen by Nb membranes is possible.

The experimental points for the second series (ArD^+ bombardment) lie lower than those for the first series and the minimum is shifted to the lower energies by about 3 eV. The maximum value $\eta = 1$ is achieved also at the energy of about 3 eV less than in the first series of the experiments made with D_3^+ ions. There are two reasons for the differences mentioned. First, the surface binding energy reduces under the heavy particle (Ar) bombardment due to partial sputtering of NbO. This results in decreasing the height of the potential barrier. The value of E_S in this case decreases to about 1 eV as estimated by comparing with computer simulations (see Fig. 2). The second possible reason for such $\eta(E_0)$ behaviour is sputtering of the trapped D atoms by Ar ions. As the sputtering yield for Ar in the range from 20 to 400 eV changes very strongly, the amount of deuterium retained in the target must decrease with energy in this energy range due to additional sputtering. But this effect was not observed. So, we suppose that the sputtering of captured deuterium was negligible. The ratio of the maximum trapping coefficient $\eta = 1$ obtained at the minimal energy of 3 eV to the minimum η at 7 eV is about 2.5.

In the third series, the values of η were even less than those in the second series. We compared these experimental data with calculations for a clean Nb surface. The reason is that Ar bombardment along with thermal annealing were supposed to produce a clean Nb surface. It is seen from Fig. 2 that the experimental and calculated data are in a good agreement. So, we can consider that the third series corresponds to a clean target surface. For this case the increase of η at low energies typical for the two previous series is not observed for energies down to $E_0 = 2.5$ eV. The calculated data correspond to the experiment at $E_S \approx 0.3$ eV. As for using Nb for hydrogen pumping, we can conclude that the most of incident particles are reflected form the clean surface.

So, for oxidised and clean surfaces of Nb, the number of captured particles at the minimal primary energies of 2–3 eV achieved in our experiments differ as much as 5 times. This must be taken into account in evaluation of hydrogen isotope capture and retention during cold plasma interaction with PFM.

From the calculations performed one must mention an additional finding. It is considered that the binary collision approximation is limited in description the low energy region in simulations of particle interactions with solids. Nevertheless, the data presented here show that the technique is able to describe the experiment even at energies as low as 2–3 eV if additionally to take into account the binding energy to the real surface.

4. Conclusion

Investigation of low energy deuterium trapping by niobium at the normal angle of incidence and ion energies of 2–30 eV was performed. It is shown that in the eV primary energy range, the number of captured particles strongly depends on the surface conditions. The 'clean' surface is supposed to be characterised by small value of the trapping coefficient $\eta \approx 0.2$–0.3 diminishing with decrease of ion energy. The contaminated or oxidised surface has higher trapping efficiency at any ion energies and is characterised by increase of trapping at the very low ion energies of 2–5 eV. The difference in experiments with the clean and contaminated surfaces can be as high as 5 times. Computer simulation based on the BCA model show a good agreement with the experimental results if the surface binding energy E_S is used as an additional adjust parameter. Values of E_S for different surface conditions were evaluated. For the clean and oxidised surfaces, E_S differs by a factor of 8 having the values $E_S = 0.3$ eV and 2.5 eV respectively. The difference at low energies can be as high as 5 times.The efficiency of deuterium pumping is expected to depend on the surface. It can be expected also that for hydrogen this effect is more prominent because of smaller trapping [6].

Acknowledgements

This work was supported in part by the Russian Foundation for Basic Research (Grant RFBR 96-02-18706).

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