

Journal of Nuclear Materials 241-243 (1997) 1022-1025



In-situ ion beam analysis and dynamic studies of deuterium retention in graphite exposed to a high flux magnetron plasma

B. Emmoth *, H. Bergsåker, L. Ilyinsky

Royal Institute of Technology, Physics Department Frescati, Association EURATOM-NFR, Frescativägen 24, S-10405 Stockholm, Sweden

Abstract

A new compact plasma simulator device has been mounted on line at the Van de Graaff accelerator and successfully used for deposition experiments of deuterium atoms with the mean ion energy of 120–140 eV. Deuterium retention in graphite exposed to a magnetron plasma at flux densities above 10^{17} D cm⁻² s⁻¹ has been studied by nuclear reaction analysis. A dynamic retention of about 1×10^{16} D/cm² has been identified in the discharge with a current density exceeding 16 mA/cm². This dynamic inventory is released within 60 ms after the discharge has been stopped. The interpretation is based on trapping, detrapping and diffusion of deuterium in graphite.

Keywords: Desorption; Low Z wall material; Wall particle retention

1. Introduction

Penetration and transport of hydrogen into carbonaceous materials is of special interest in fusion research, since these materials are widely used for surfaces in contact with the plasma. In particular, in non-stationary conditions of operation the density evolution in plasma discharges is largely dependent on transient pumping by the wall surfaces.

The trapping and release of hydrogen in carbon materials as a result of irradiation with energetic ions has been studied very intensely and for a long time [1], using mass analyzed and monoenergetic ion beams. The main drawback of these measurements is that they are limited to hydrogen flux densities of $\sim 10^{14}$ H cm⁻² s⁻¹ or less, whereas the flux density at limiter and divertor surfaces in the large fusion devices is in the range 10^{18} – 10^{19} cm⁻² s⁻¹. Since both models and experiments suggest a non-linear, flux dependent reemission of hydrogen from an irradiated surface, it is precarious to extrapolate only from ion beam results to reactor conditions. The same is true for carbon erosion due to energetic hydrogen erosion. Moreover, the result may not be relevant to the fusion machine

due to synergetic effects. Large simulation devices have been designed with plasma parameters close to those of tokamak edge plasmas, specifically to study plasma– surface interaction. Both carbon erosion and hydrogen retention in graphite have been studied [2].

We have developed a special compact magnetron plasma system which is suitable to study irradiation effects in an intermediate flux density range and to some extent to simulate the plasma surface interaction in a large fusion device. The plasma device is versatile and easy to operate and allows quick change of samples and a high flexibility in changing the plasma parameters. In this report we present measurements of transient hydrogen retention in graphite with flux densities up to 10^{17} cm⁻² s⁻¹.

2. Experimental

The experimental set-up is shown schematically in Fig. 1. The plasma generator consists of a gas discharge chamber with abnormal glow discharge in crossed electric and magnetic fields, combined with planar magnetron design [3]. The materials to be investigated are placed at the cathode position of a magnetron plasma discharge in deuterium or helium. The device is operated in the pressure range 1-5 mTorr and the magnetic field of 50-100 mT is

^{*} Corresponding author. Tel.: +46-8 161 000; fax: +46-8 158 674; e-mail: emmoth@msi.se.



Fig. 1. The schematic arrangement of the dynamic measurements of deuterium retention during magnetron plasma irradiation.

nearly parallel to the surface. The cathode current density can be chosen in a range up to 100 mA/cm², which corresponds to a flux density of up to 10^{18} D cm⁻² s⁻¹ in a deuterium discharge, considering that the ions which strike the surface are mostly D₂⁺. In-situ energy analysis of the ion flux by retarding potential shows (as in Fig. 2) that the average ion energy can be chosen in the range 100–300 eV by changing both the working pressure and the degree of discharge self-sustainment [3]. The cathode is provided with water cooling.

In the present series of measurements, 5 mm thick samples of Schunk FP15 graphite were mounted on the magnetron cathode. The target temperature was monitored



Nuclear reaction analysis with the 3 He(d, p)⁴He reaction was used to determine the areal density of deuterium at the sample surface. The beam current of the 1.8 MeV 3 He⁺ analyzing beam was monitored by elastic backscattering measurements at a beam chopping propeller situated



Fig. 2. Normalised integral energy distributions of hydrogen ions striking the sample surface: I_d is the discharge current, U_r is the retarding potential, $j_{ic}(U_r)$ is the current density of ions with the energy in the range from eU_r to eU_c and $j_{ic}(U_c)$ is the current density of ions with the energy eU_c , where U_c is the cathode drop voltage, which for the planar magnetron one may assume equal the discharge voltage $U_d = 380$ V.



Fig. 3. Sample equilibrium temperature as a function of flux density.

in the analytical chamber. The areal density of deuterium within a few microns depth below the surface of the exposed materials was measured during implantation as well as after the discharge had been switched off. The plasma 'on-off' cycles were repeated several times with each graphite sample.

3. Results

Fig. 4 shows an irradiation sequence with an initially fresh graphite sample. The irradiation starts at t = 50 min with a current density of 5 mA/ cm^2 , which corresponds to a flux density of 6×10^{16} D cm⁻² s⁻¹. The areal density of trapped deuterium quickly increases to a saturation level around 4×10^{16} D/cm². Subsequently there is a slight decrease in the areal density due to the temperature increase, while the sample reaches an equilibrium temperature at 80°C with a thermal response time of about 10 minutes. The areal density levels off towards a characteristic stationary level during implantation. After the discharge has been switched off at t = 160 min the measurement is repeated several times. The subsequent slow decrease has been shown to be due to detrapping by the analysis beam. By extrapolation back to t = 160 min the areal density shortly after the discharge was switched off, can be determined. Compared to the saturation level during implantation there is a decrement N_d as defined in the Fig. 4. In the 5 mA/cm² discharge the decrement $N_{\rm d}$ 2.5 × 10¹⁵ D/cm^2 . When the irradiation is repeated at 5 mA/cm² the areal density of deuterium reaches the same saturation level, and the subsequent decrement when the discharge is switched off is also repeated. The irradiation was finally performed with 10 mA/cm² current density, resulting in a saturation level at 2.8×10^{16} D/cm². Note that the target



Fig. 4. Deuterium irradiation of graphite in magnetron plasma.



Fig. 5. Flux density dependence of the dynamic inventory.

temperature according to Fig. 3 was 180°C in the case of 10 mA/cm², compared to 80°C in the low current case. The decrement at the end of the discharge was $N_d = 7 \times 10^{15} \text{ D/cm}^2$ at 10 mA/cm. Fig. 5 shows the decrement N_d as a function of incident flux.

4. Discussion

From Fig. 4 it is clear that the dynamically retained areal density N_d is released on a time scale shorter than 100 s, which is the time required to get sufficient statistical accuracy in the measurement. Furthermore it can be seen from Fig. 5 that N_d is approximately proportional to the incident flux density Φ . This is consistent with the assumption that

$$\frac{\mathrm{d}N_{\mathrm{d}}}{\mathrm{d}t} = \Phi - \frac{N_{\mathrm{d}}}{\tau}$$

which in steady state conditions gives $N_d = \Phi \tau$. From Fig. 5 the release time $\tau = 56$ ms can be identified, which should not be too much influenced by temperatures at a lower temperature range, below 300°C.

The transient retention may for instance be due to diffusion of deuterium molecules after recombination at the implantation range λ from the surface. TRIM calculations give an implantation range of the order $\lambda = 35$ Å at 130 eV [6], in which case a diffusivity of $D \approx \lambda^2 / \tau = 2 \times 10^{-16}$ m²/s can be derived. This is comparable to earlier measurement [7]. The comparatively short transient retention which is observed in this experiment is of consequence in particular for the interpretation of the recycling behavior in smaller plasma devices, such as EXTRAP T2 [8], which have pulse lengths of the order of 10 ms.

It should be pointed out that the dynamic retention according to Fig. 5 in this work would be below the detection limit in previous experiments [4,5], since they were performed with 1-3 orders of magnitude lower flux density.

According to our results, there is an increasing dynamic inventory with increasing fluence, and it should be possible to extrapolate our data to fluences which are striking divertors and limiters in modelling and calculations.

5. Conclusions

A new magnetron plasma simulator device has been set up with ion beam analysis on line at the Van de Graaff accelerator and has successfully been used for deuterium irradiation experiment with the average ion energy ranged 120-140 eV per atom to flux densities above $10^{17} \text{ D cm}^{-2} \text{ s}^{-1}$. Nuclear reaction analysis was used for dynamic studies of deuterium retention in graphite exposed to a high flux magnetron plasma. A transient retention of about $1 \times 10^{16} \text{ D/cm}^2$ has been identified in discharge with a current density exceeding 16 mA/cm². This dynamic inventory is released after the discharge has been stopped. The results are consistent with a release time of about 60 ms.

Acknowledgements

This work was supported by the Swedish Natural Science Research Council (NFR) and by a grant from the Royal Swedish Academy of Sciences.

References

- [1] W. Möller, J. Nucl. Mater. 162-164 (1989) 138.
- [2] Y. Hirooka, R. Conn, R. Causey et al., J. Nucl. Mater. 176–177 (1990) 473.
- [3] B. Emmoth, L. Ilyinsky, H. Hultberg et al., J. Nucl. Instrum. Methods B (1996), accepted.
- [4] C. Jandl, W. Möller and B. Scherzer, Proc. 18th Eur. Conf. Contrib. Fusion Plasma Phys., Berlin, 1991, Part III, p. 245.
- [5] K. Morita and Y. Hasebe, J. Nucl. Mater. 176–177 (1990) 213.
- [6] W.R. Wampler, D.K. Brice and C.W. Magee, J. Nucl. Mater. 102 (1981) 304.
- [7] J. Pillath, J. Winter and F. Waelbroeck, J. Nucl. Mater. 162-164 (1989) 1046.
- [8] H. Bergsåker, D. Larsson, P. Brunsell et al., these Proceedings, p. 993.