MASTER

HYDROGEN RETENTION AND RELEASE IN FIRST-WALL COATINGS FOR TOKAMAKS*

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Abstract

The hydrogen retention and release of two first-wall coating materials, TiB_2 and B_4C , were measured. Hydrogen is released from TiB_2 at substantially lower temperatures or laser energy fluxes (pulsed annealing) than for B_4C . Hydrogen also diffuses to a much smaller extent into TiB_2 than into B_4C . On the basis of tritium inventory control, TiB_2 appears to be superior to B_4C .

INTRODUCTION

The first requirement of Tokamak limiters, or other first-wall elements, is the ability to survive repeated high-energy deposition pulsing.¹ A primary problem is keeping the element's temperature below the melting temperature without inducing a destructive thermal shock which can be caused by intense beams of electrons, ions, and neutrals. It has also been well documented that the limiter constitutes a major source of impurities in the discharge plasma.² Because the potentially large radiation losses caused by this contamination have an exponential Z (atomic number) dependence, low-Z materials are desirable for limiters or other first-wall elements. Low-Z coatings offer the promise of separating the functions of impurity control and structural requirements.

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It has also been shown that the first-wall provides a good deal of fuel for the Tokamak resulting from the release of hydrogen which has accumulated on (or in) the wall from prior discharges.³ This release has been partially controlled by evaporation of thin layers of Ti. It is therefore important for plasma considerations to determine the hydrogen recycle characteristics of candidate coating materials.

In the future, although structural, plasma contamination, and hydrogen recycle requirements will remain imperative, emphasis will most certainly shift to include the question of tritium inventory control. Indeed, this is already of great concern in the design of proposed Tokamaks, e.g., TFTR. In this regard, the evaporation of Ti to control plasma refueling may lead to unacceptably large tritium inventories. Thus, with the approach of the era of D-T burning Tokamaks, comprehensive questions on the behavior of H (T in particular) in first-wall materials must be answered. In this paper three such issues are addressed: (1) the H release temperature, (2) H accumulation potential, and (3) the technique of pulsed annealing to purge H from first walls once it has accumulated.

EXPERIMENTAL

Two candidate first-wall materials, TiB_2 and B_4C , were examined. The TiB_2 samples were thin ($\sim 50 \mu\text{m}$) CVD coatings on POCO graphite substrates;⁴ whereas, the B_4C samples were hot pressed and in bulk form.⁵ B_4C has been used as a limiter material in TFR 400⁶ and behaved satisfactorily up to discharge currents of 80 kA, but failed at higher currents due to thermal shock. Although TiB_2 has yet to be tested on a limiter, simulation results on thin coatings are very promising.^{7,8} TiB_2 -coated limiters will be tested on ISX-B later this year. B_4C coatings are also under investigation with the hope that these coatings will not be as sensitive to thermal shock as is bulk B_4C .

In the present study, ^1H was injected into the samples by ion implantation. It is assumed that the behavior of ^1H simulates closely that of the other two hydrogen isotopes, deuterium and tritium. Hydrogen concentration profiles were measured by ^1H (^{19}F , $\alpha\gamma$) ^{16}O nuclear reaction analysis. The resonance at 6.4 MeV (^1H rest frame) was used. Thermal annealing was performed under argon to prevent surface oxidation.

RELEASE TEMPERATURES

Knowledge of the temperature at which H is released from a candidate material is necessary to permit prediction of recycling behavior and to anticipate problems relating to the control of tritium inventories. In order to measure the temperature at which hydrogen is released from TiB_2 and B_4C , ^1H was implanted (60 keV for TiB_2 , 30 keV for B_4C) to depths of 3000 - 4000 Å, and the samples were annealed isochronally for 20 minutes at increasing temperatures. ^1H profiles were measured between each anneal, and these profiles along with plots of H retention as a function of anneal temperature are shown in Figure 1. Clearly, H is retained in B_4C to much higher temperatures than in TiB_2 . One-half of the H in TiB_2 is released by 400°C, whereas, 700°C temperatures are required for the same fractional release in B_4C . The relative constancy of the width of the H peak as a function of temperature indicates that the release is trap-limited in both materials. This observation indicates that the implanted H is trapped in its own ion-induced damage.

In previous reports,⁹⁻¹¹ approximate H trap energies in TiB_2 were deduced from the isochronal anneal data by assuming no subsequent re-trapping of H once it was liberated. This assumption, however, may not be valid because re-trapping in He-induced damage has been directly observed.¹¹ Trap energies listed in our earlier works should therefore be considered as upper limits.

H ACCUMULATION POTENTIAL

The fusion facility TFTR at Princeton will be allowed only 50 kCi or 5 g of T on site.¹⁰ Of this $\approx 5\%$ can be in a releasable form in getters or limiters. If the entire 5 g of T were deposited uniformly over the inside of TFTR ($\sim 2 \times 10^6 \text{ cm}^2$), it would result in an areal T density of $5 \times 10^{17} \text{ T atoms/cm}^2$. At a concentration of 50 at.%, such contamination would be a few thousand angstroms deep. This level of contamination would be difficult to reach if the H deposited into the wall did not diffuse, but rather would be reemitted from the surface. Such behavior has been observed.¹³ The question of whether a first-wall material undergoes bulk-directed H diffusion, damaged assisted or otherwise, is therefore critical.

Bulk-directed H diffusion in TiB_2 and B_4C was searched for in two ways: (1) Samples were predamaged with He implantation on both sides, surface and bulk, of the H implant peak. They were annealed and then H profiled. The H decoration of the He-induced damage indicates the predominant direction of diffusion. (2) H was implanted at low-energy (250 eV) and very high fluences ($> 10^{19} \text{ H/cm}^2$). Subsequent H profiling indicates if bulk-directed diffusion occurs.

The predamage experiments showed decoration of surface-side He predamage for both TiB_2 and B_4C , but bulk-side predamage was decorated for B_4C only. The resulting H profile of predamaged B_4C is shown in Figure 2. H profiles of predamaged TiB_2 were reported elsewhere.^{9,11}

The low-energy, high-fluence, ¹H implantation results are summarized in Figure 3. The profiles plotted in this figure result from subtracting the pre-implantation profile from the post-implantation one. As can be seen from these data, bulk H diffusion is much more in evidence for B_4C than for TiB_2 .

PULSED ANNEALING

As previously suggested,⁹ pulsed annealing of first-wall materials, using either electron beams or lasers, may be an effective way of purging, and thereby controlling, hydrogen isotope inventories. Electron-beam annealing (2 kW/cm^2 , $\sim 1 \text{ sec}$) of H-implanted TiB_2 coatings removes all of the H from the coating.⁹ This release was attributed to the high temperature ($\sim 2400^\circ\text{C}$) which was attained, and, hence, similar results are to be anticipated for other materials (particularly B_4C), provided they can survive the thermal shock.

H-implanted samples of both TiB_2 and B_4C were annealed with a Q-switched Nd:glass laser. Annealing with 20 nsec laser pulses released H from both materials as shown by the data in Figure 4. A higher energy flux was required to release H from B_4C than from TiB_2 .

CONCLUSION

Our results indicate that, on the basis of T inventory control, TiB_2 is a better choice than B_4C as a first-wall material in a Tokamak. H is released at lower temperatures for TiB_2 than for B_4C , and lower laser energy fluxes are required using pulsed annealing to purge H from near surface regions of TiB_2 . H also undergoes less bulk-directed diffusion in TiB_2 than in B_4C , and hence the T accumulation potential for TiB_2 is smaller.

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References

1. Princeton University Plasma Physics Laboratory Publication PH-N-009, 1978.
2. P. Staib and G. Staudenmaier, J. Nucl. Mater. 76/77, 78 (1978).
3. G. M. McCracken, J. Nucl. Mater. (in press).
4. H. O. Pierson, E. Randich, and D. M. Mattox, J. of Less Common Metals (in press).
5. Norton Company, Industrial Ceramics Division, Worcester, MA 01606.
6. TFR Group, EUR-CEA-FC-852, October 1976.
7. D. M. Mattox, A. W. Mullendore, H. O. Pierson, J. Nucl. Mater. (in press).
8. D. M. Mattox, D. J. Sharp, SAND 78-1029, Sandia Laboratories, Albuquerque, New Mexico, 1978.
9. B. L. Doyle and F. L. Vook, J. Nucl. Mater. (in press).
10. B. L. Doyle and F. L. Vook, Nucl. Sci. Trans., NS-26, 1305 (1979).
11. F. L. Vook, B. L. Doyle, and S. T. Picraux, Nucl. Sci. Trans., NS-26, 1272 (1979).
12. J. L. Cecchi, Private Communication.
13. B. M. U. Scherzer, R. Behrisch, W. Eckstein, U. Littmark, J. Roth, and M. K. Sinha, J. Nucl. Mater. 63, 100 (1976).

Figure Captions

Figure 1 ^1H profiles and retention curves for B_4C and TiB_2 after annealing. Boxes b. and d. show the ^1H profiles as measured by nuclear reaction analysis for ^1H implanted into B_4C and TiB_2 , respectively. These samples were annealed isochronally at the indicated temperatures. The profiles have been displaced vertically for clarity. Boxes a. and c. show the respective retention curves for B_4C and TiB_2 .

Figure 2 ^1H profiles of ^1H implanted ($10^{17}/\text{cm}^2$, 30 keV) He predamaged ($10^{17}/\text{cm}^2$, 20 and 100 keV) B_4C a. as implanted and b. after a 700°C , 20-minute anneal.

Figure 3 H profiles of TiB_2 and B_4C after exposure to intense low energy (250 eV) H beams.

Figure 4 H retention in B_4C and TiB_2 after annealing with a Q-switched Nd:glass laser ($\lambda \sim 1 \mu\text{m}$, $\tau \sim 20 \text{ ns}$). In box a. the energy flux was $1.5 \text{ J}/\text{cm}^2$ while in box b. it was $3.0 \text{ J}/\text{cm}^2$.

Figure 1

Figure 2

Figure 3

Figure 4