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RETENTION, ISOTOPE EXCHANGE, AND THERMAL
RELEASE OF HYDROGEN IN CANDIDATE MATERIALS FOR TFTR

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I. Introduction

This report summarizes the results of hydrogen retention, hydrogen isotope exchange, and thermal release measurements for low-z coatings and claddings being evaluated as candidate materials for use in TFTR. The materials used in TFTR will be exposed to high fluxes of hydrogen, deuterium, and tritium ions and neutrals with energies in the range of tens to thousands of electron volts. Retention and release of this hydrogen can grossly affect the plasma density, temperature, and isotopic composition during operation of TFTR. In addition, it is important to know how much tritium the materials will retain; and, therefore, processes which result in release of tritium from the materials, such as thermal detrapping and replacement by hydrogen or deuterium, must be clearly understood. For these reasons the hydrogen retention, isotope exchange, and thermal release characteristics of the various materials must be taken into consideration in selecting a material suitable for use in TFTR. These processes were studied in a variety of materials by implanting hydrogen and deuterium at energies (0.6 to 1.5 keV/atom) corresponding to typical particle energies in a tokamak environment.

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The materials studied included TiC, TiB₂, VB₂, B₄C, B, Si, graphite, and the metals Ti, V, and 304L stainless steel. The TiC and TiB₂ were formed by chemical vapor deposition on a graphite substrate[1]. The C/Ti ratio of the TiC was measured to be $1.0 \pm .05$ by ion backscattering analysis. The Ti and V were explosively bonded to copper substrates, and the VB₂ was made by borodizing vanadium. Carbon (compression annealed pyrolytic graphite from Union Carbide and Papyex graphite ribbon from Le Carbone) and single crystal silicon samples were included in the study as reference materials.

The hydrogen retention and isotope exchange behavior for these materials were studied by measuring the amount of H or D retained as a function of incident fluence using the D(³He,p)⁴He nuclear reaction analysis techniques for D and H(¹⁵N,αγ) profiling for H.[2]. In some cases the retained amounts of H and D were measured simultaneously using elastic recoil detection[3] with a 2.6 MeV ⁴He analysis beam. These techniques give the total amount of H or D within ~ 500 nm of the surface which is much thicker than the implant layer. The implants were done at room temperature. Incident fluxes in the range $1-3 \times 10^{14}$ atoms/cm²s were used which is low energy to avoid significant heating by the implant beam.

2. Hydrogen Retention

In the materials TiC, TiB₂, VB₂, B₄C, B, C, and Si, it was found that at low fluences ($< 3 \times 10^{16}$ /cm² for 1.5 keV/atom) all the incident H or D was retained in the sample, apart from a small fraction (typically 5-10%) which was kinematically reflected as shown in Fig. 1. As the incident fluence increased, the amount of D retained eventually approached a saturation value which was

in the range $1-3 \times 10^{16} \text{ D/cm}^2$ for an implant energy of 1.5 keV/ion. Depth profiling of D implanted in C[4], Si[5], B_4C , and TiB_2 (this report) show that at low fluences hydrogen is retained at the end of its implant trajectory in these materials. The profile of hydrogen deposition in the materials is therefore determined by the range and straggling of the hydrogen, which can be predicted theoretically[5,6,7]. As the incident fluence increases, a saturation hydrogen concentration is eventually reached in these materials. The retention behavior can be modeled[8] by assuming that hydrogen implanted into regions which have already reached saturation is not retained in the material. This type of behavior is illustrated in Fig. 2, which shows calculated retention profiles (D concentration versus depth) for various fluences of 1.5 keV D on graphite (solid lines). At high fluences where the material has reached saturation, the total amount of D retained depends on the saturation deuterium concentration for the material, and on the thickness of the saturated layer, and therefore on the energy of the incident deuterium. This can be seen in Fig. 3 which shows the areal density of D retained in a graphite sample as a function of D ion fluence for incident energies of 0.67, 1.0, and 1.5 keV. The solid lines are results of a calculation based on a model[8] which incorporates the deuterium deposition profile[7] and uses a saturation concentration of 0.53 H/C ratio. This figure illustrates the fact that a higher incident energy results in a higher areal density of retained hydrogen because of the greater thickness of the saturated layer.

The materials of Fig. 1 all behave similarly, but have different saturation concentrations, N_{sat} . Using N_{sat} as the only adjustable parameter, the model described above was used to obtain the value of

N_{sat} for each material from a fit to the data shown in Fig. 1. Table I summarizes the parameters used to model saturation in these materials. This table gives the saturation concentrations in units of D/cm^3 and $D/\text{host atom ratio}$, as well as the range and straggling parameters and the saturation areal densities for 1.5 keV D in the various materials. The estimated accuracy of the saturation concentrations and areal densities are $\pm 15\%$. Included in the table are the values for 316 stainless steel at -120°C where hydrogen diffusion is suppressed[9].

The D retention data of Fig.4 shows that Ti, V, and stainless steel (at room temperature) behave differently from the other materials studied. The reason for this different behavior is that the hydrogen is mobile in these materials at room temperature. The diffusion coefficient for hydrogen in stainless steel at room temperature is $\sim 10^{-12} \text{ cm}^2/\text{s}$ with an activation energy of $\sim 0.6 \text{ eV}$ [10]. This means that in the time required for a measurement of retention, the diffusion length for the hydrogen is large compared to the implant depth. In this case, the observed retention will sensitively depend on the sample temperature, on the permeability of the surface, and the implant flux. Due to the high mobility of D in stainless steel at room temperature, most of the D is lost from the near surface region within several hours of the implant [9,10].

The rate at which hydrogen implanted in stainless steel escapes was found to depend on the condition of the surface. The 304 SS sample of Fig. 4 was electropolished prior to implantation. After an incident fluence of $\sim 3 \times 10^{18} \text{ D/cm}^2$ at 1.5 keV, the analysis

gave $\sim 8 \times 10^{16}$ D/cm² in the near surface region. After 90 hours at room temperature, the analysis gave 1.5×10^{16} D/cm². These values are considerably higher than the retention previously observed for mechanically polished stainless steel samples[10]. This suggests the presence of a permeation barrier on the surface of the electropolished samples.

Hydrogen retention behavior in Ti and V is different from stainless steel because of the exothermic heat of solution of hydrogen in these materials and the tendency to form a hydride phase. Figure 4 shows that for 1.5 keV D implanted in Ti and V, most of the D is not retained in the first 500 nm probed by the nuclear reaction analysis. However, gas re-emission measurements [10] show that D implanted in Ti or V at room temperature is totally retained in the material (apart from the kinematically reflected fraction). Together these results show that most of the D is diffusing into the bulk of the material beyond the 500 nm range of the nuclear reaction analysis. This means that in a tokamak environment these materials would retain large amounts of hydrogen isotopes. This would be undesirable in a tritium fueled machine.

3. Isotope Exchange

From the tritium inventory standpoint, it is important to know if tritium retained in a material can be recovered. In the materials which exhibit saturation behavior (TiC, TiB₂, VB₂, B₄C, B, C, Si), this can be done by replacing the tritium with hydrogen or deuterium. To characterize the isotope exchange behavior, we have implanted samples of the candidate materials

with D (or H) to saturation and then switched the implant ion to H (or D) and monitored the D and H content of the samples as the second isotope displaced the first.

Figure 5 shows a composite of the isotope exchange curves for TiC, TiB₂, VB₂, B, C, and Si. The dots and circles show the measured amounts of D and H, respectively, retained in the samples versus H + D fluence. The break in the curves occurs where the implant was switched from D to H. The data for B, Ti, C, VB₂, and TiB₂ were taken using nuclear reaction analysis (NRA). The data shown for pyrolytic graphite and silicon were obtained using elastic recoil analysis which has the advantage of giving the amount of retained H as well as D. Similar measurements on C and Si using NRA gave the same results within experimental uncertainty. Also, no difference in the saturation or exchange behavior was found between pyrolytic graphite and Papyex. It can be seen that the exchange behavior is similar in all of these materials.

To describe the hydrogen isotope exchange behavior we have developed a new model which treats the exchange differentially in depth. The model uses the H and D deposition profiles which can be obtained from standard transport theory[6,7] and the saturation concentrations for the various materials determined from the retention measurements described in the previous section (see Table 1). These saturation concentrations can also be directly measured using high resolution depth profiling techniques. No additional free parameters are required by this model to describe the exchange behavior. This model assumes that for every H, D, or T atom which comes to the end of its trajectory in a region of the

material which is saturated, one H, D, or T is lost from the material so that the local $(H + D + T)/\text{host atom ratio}$ never exceeds the saturation concentration. Also the model assumes that local mixing of the hydrogen isotopes occurs. In other words that the probability of the displaced atom being H, D, or T is given by the ratio of the local H, D, or T concentrations to the saturation concentration respectively. Incident H, D, or T atoms stopping in regions which are below saturation are retained.

The calculated profiles for D retention in graphite shown in Fig. 2 illustrate how D initially implanted to saturation is replaced by subsequent implantation with H. The exchange proceeds most rapidly at a depth corresponding to the peak in the deposition of the H, but proceeds more slowly in the wings of the profile. Just after switching isotopes the exchange rate is close to 1:1 (one D lost for every incident H) because the D concentration is initially much larger than the H concentration at all depths. As the replacement proceeds, however, the exchange rate decreases because more and more of the displaced atoms are H rather than D. The total amount of D and H in the sample can be calculated by integrating over depth. Plotting this as a function of the total incident $H + D$ fluence gives theoretical isotope exchange curves which are compared to the data in Fig. 5. The agreement between the data and the model is within the experimental uncertainty in all cases.

In order to check that our isotope exchange model has the correct dependence on the incident particle energy, isotope exchange measurements were made in graphite using different incident energies for the H and D. This changes the overlap between the depth profiles of the two isotopes and thereby affects the exchange behavior. When the H and D are both implanted at 1.5 keV (Fig. 6a), the H and D profiles overlap very closely. The model shows that in this case the D on the deep side of the profile is hard to replace because fewer H atoms stop at this depth than at the peak. Decreasing the H energy to 1 keV (Fig. 6b) shifts the H profile toward the surface such that more of the D on the deep side of the profile remains in the sample. Decreasing the initially implanted D energy to 1 keV (Fig. 6c) shifts the D profile toward the surface such that the deeper D is more easily replaced by the H. The good agreement between the data (dots) and the model (lines) indicates that the model contains the correct dependence on the incident particle energy.

We have also measured the isotope exchange in graphite and Si both with H and with D as the initially implanted isotope. The exchange behavior in both cases was similar. This demonstrates that there are no unexpected isotope effects and supports the expectation that replacement of tritium would behave in a similar way.

4. Thermal Release

In addition to isotopic replacement by implantation, hydrogen isotopes can also be released by heating. The practicality of heating as a method to release tritium from tokamak components depends on the nature of the component and the temperature that

is required. On the other hand, some components such as limiters may reach high temperatures during routine operation of the machine. For these reasons, it is of interest to survey the thermal release properties of the candidate materials. Extensive studies of thermal release have been done by K. L. Wilson and A. E. Pontau at Sandia Laboratories in Livermore by measuring gas re-emission during ramp heating. These studies are described in a separate report[10].

To determine whether thermal release is preceded by diffusion into the bulk of the sample as was found to be the case for Ti and V at room temperature, nuclear reaction analysis can be used to determine the effect of heating on the near surface H or D content and depth profiles. Figure 7 shows the results of such measurements for B_4C and TiB_2 . These were implanted with $10^{17}H/cm^2$ at 60 and 40 keV respectively. The H profiles were measured using the $H(^{19}F, \alpha\gamma)$ reaction after annealing the samples for 20 minute intervals at successively higher temperatures. Most of the H was released after annealing at 400°C for TiB_2 and 700°C for B_4C . The fact that the depth profiles do not significantly broaden shows that the rate limiting process in the release is detrapping rather than diffusion through the bulk material. Previous studies[4] have shown a similar behavior for the release of D implanted in graphite. Table II summarizes the approximate temperature range over which thermal release of H or D has been observed in the various candidate materials.

5. Hydrogen Retention and Isotope Exchange in a Tokamak Environment

In order to estimate hydrogen retention and isotope exchange

in a tokamak environment, it is necessary to include the effects of a distribution of energies and angles of incidence for the hydrogen. Our model for saturation and isotope exchange can easily include these effects by using the hydrogen depth profile for the desired energy and angular distributions. In contrast to previous models for isotope exchange [9,11], the dependence on implant energy in our model is all contained in the deposition profiles. Experimental verification that our model contains the correct energy dependence is given in Figs. 6 and 8. Figure 8 shows data from measurements by Blewer et al [4] of saturation with D and replacement by H in 316 stainless steel at -120°C for incident energies between 1 and 14 keV. The solid line shows the behavior predicted by our model using a saturation concentration of 1.0 (H + D)/metal atom ratio. In all cases the agreement is excellent.

If we assume that a surface in a tokamak will be exposed to incident hydrogen with a Maxwellian velocity distribution the retention and exchange behavior can be calculated. Figure 9 shows the retention behavior expected under these conditions for TiB_2 , and TiC , for a range of Maxwellian temperatures. This is plotted in Fig. 10 as the areal density of D retained after an incident fluence of 10^{20} D/cm² as a function of the characteristic particle temperature. The right hand scale gives the equivalent activity in Ci/m² if the retained atoms were all tritium. From this, one can estimate an upper limit for the tritium inventory in these materials. For example, if one assumes a plasma edge

temperature of 1 keV for TFTR, then after an incident fluence of 10^{20}T/cm^2 , a 10% coverage of the internal surface of TFTR ($\sim 10 \text{m}^2$) by TiC could retain as much as $\sim 1000 \text{ Ci}$ if the retained atoms were all T. This is less than the 2300 Ci allowed T inventory in TFTR in readily releasable form.

Figure 11 shows the calculated isotope exchange behavior for T being replaced by D in TiC for the case of a Maxwellian velocity distribution. This illustrates the feature that the high energy tail of the Maxwellian distribution leads to a sustained gradual build-up of retained T to greater depths and that recovery of this deep component by isotope exchange will require high D fluences. If the initial T fluence is increased by an order of magnitude, then about an order of magnitude higher D fluence is also needed to reduce the T content to a given level. The efficiency of the T recovery can be greatly enhanced by using D which is more energetic than the T. This can be seen in Fig. 12 which shows a calculation to model the replacement of T from TiC which is initially exposed to a T plasma with $kT = 200 \text{ eV}$ to a fluence of 10^{18} T/cm^2 followed by exposure to; 1) a D plasma with $kT = 200 \text{ eV}$, or 2) a D plasma with $kT = 400 \text{ eV}$.

Finally, Table III gives the amount of retained D measured in several materials after exposure to high power discharges in PLT, including some of the coating materials and a piece cut from a graphite limiter. The incident D fluence to these samples was high enough to saturate the near surface. The amount of D retained is consistent with the retention characteristics of the materials, and the energy and flux of D at the plasma edge

determined from probe studies[12].

6. Conclusions

In summary, our results show that hydrogen retention behavior is qualitatively similar for the materials TiC, TiB₂, VB₂, B₄C, B, Si, and graphite. The amount of hydrogen retained depends on the implant depth, and hence on the energy of the incident hydrogen, and on the saturation concentration for the material. The saturation concentrations for these materials (at room temperature) was found to be in the range 0.16 to 0.57 hydrogen/host atom ratio. The areal density of hydrogen retained after saturation with a given incident energy was lower in TiC, TiB₂, and VB₂ by about a factor of 3 than in B, B₄C, and graphite. We have also found that hydrogen isotopes in these materials can be replaced by subsequent implantation with a different isotopic specie. This indicates that recovery of tritium from these materials by replacement with hydrogen or deuterium should be feasible. The model we have developed to describe this process agrees very well with the data and provides for the first time a way to predict retention and exchange behavior in a tokamak environment. This is important not only from the tritium inventory standpoint but also for modeling effects of recycling at the wall on the isotopic composition of the plasma during a discharge.

The retention and exchange behavior of hydrogen in Ti and V is different because of the mobility and exothermic heat of solution of hydrogen in these materials. In a tritium fueled tokamak, these materials could retain much larger amounts of tritium, and recovery by isotope exchange does not appear to be practical. Tritium release from these materials would require heating to ~300 to 500°C.

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Figure Captions

1. Retention curves measured by nuclear reaction analysis for 1.5 keV D incident on several low Z target materials.
2. Calculated D retention profiles for various incident D fluences showing saturation for the higher fluences. The dashed lines show the D profiles after partial replacement by H.
3. Calculated (solid lines) and experimental (symbols) retention of D implanted in carbon for different incident energies.
4. Retention curves measured by nuclear reaction analysis for 1.5 keV D incident on Ti, V, and 304 stainless steel.
5. Calculated (solid and dashed lines) and experimentally measured (dots and circles) saturation and isotope exchange behavior for various low Z materials implanted with 3keV D_2^+ followed by replacement with 3keV H_2^+ .
6. This figure shows the effect on isotope exchange of using different energies for the initial (D) and replacing (H) ion species.
7. Retention and depth profiles for H in B_4C and TiB_2 after annealing for 20 minute intervals at successively higher temperatures.
8. Saturation and isotope exchange in 316 stainless steel at 120°C, for different incident energies. The symbols show the data measured by Blewer et al[7], and the solid lines are the prediction of our model using $N_{sat} = 1.0$.
9. Calculated D retention versus incident fluence for isotropic and Maxwellian angular and energy distributions and different ion temperatures in TiC (a) and TiB_2 (b).
10. Calculated D retention in TiC, and TiB_2 versus plasma temperature for an incident fluence of $10^{20} D/cm^2$. The scale on the right shows the equivalent activity if the retained atoms were all tritium.
11. Calculated isotope exchange behavior for replacement of T by D in TiC for Maxwellian velocity distributions with $kT = 200$ eV.
12. Calculated retention and exchange for TiC exposed to T with $kT = 200$ eV followed by D with $kT = 200$ eV or D with $kT = 400$ eV.

Material	1.5 keV Deuterium			Saturation Concentration	
	Range ⁰ (Å)	Stragglng ⁰ (Å)	Saturation (10 ¹⁷ D/cm ²)	(10 ²² D/cm ³)	H/Host (%)
C	247	101	2.50	6.0	53
Si	335	161	1.91	2.5	50
B	216	95	3.40	5.9	45
B ₄ C	288	117	3.57	6.9	57
TiC	183	91	1.22	2.5	26
VB ₂	189	92	0.96	1.9	16
TiB ₂	208	102	1.00	1.8	16
316 SS [*]	100	117	3.00	8.5	100

* -120 °C

TABLE I

Material	Temperature Range for H Release ($^{\circ}$ C)
C	350 - 550
Si	300 - 600
B	100 - 525
B ₄ C	400 - 700
TiC	100 - 600
VB ₂	100 - 500
TiB ₂	100 - 500
316 SS	- 200
Ti	300 -
V	300 -

TABLE 2.

TABLE III

DEUTERIUM RETAINED IN VARIOUS MATERIALS EXPOSED TO DISCHARGES
IN PLT.

MATERIAL	# OF SHOTS (PLASMA)	10^{16} D/cm ² RETAINED
		10^{16} /cm ²
C (PROBE)	12 D	8 - 18
C LIMITER	HUNDREDS (H,D)	10 - 30
TiC	14 (D + He)	4.4
TiB ₂	14 (D + He)	3.9
V	14 (D + He)	1.5
C	14 (D + He)	9.4

Figure 1

Figure 2

Figure 3

Figure 4

LOW Z COATING MATERIALS

CARBON AND SILICON

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9

Figure 10

Figure 11

Figure 12

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