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MATERIALS EXPOSED IN PLT

PLASMA PHYSICS LABORATORY

PRINCETON UNIVERSITY
PRINCETON, NEW JERSEY

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by

W. R. Wampler, S. T. Picraux, S. A. Cohen, H. F. Dylla,
G. M. McCracken, S. M. Rossnagel, C. W. Magee

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HYDROGEN ISOTOPE TRAPPING IN MATERIALS EXPOSED IN PLT*

W. R. WAMPLER and S. T. PICRAUX

Sandia Laboratories,† Albuquerque, New Mexico 87185, USA

S. A. COHEN, H. F. DYLLA, G. M. MC CRACKEN‡ and S. M. ROSSNAGEL

Plasma Physics Laboratory, Princeton University, Princeton, New Jersey 08544, USA

C. W. MAGEE

RCA Laboratories, Princeton, New Jersey 08540, USA

Samples exposed at various minor radii in PLT to small numbers of high power discharges have been analyzed for the amount and depth distribution of implanted hydrogen isotopes and higher Z impurities. Comparisons of the measured H/D concentrations and depth profiles with laboratory implantations and calculated depth profiles give the energy and fluence of the hydrogen implanted in the PLT samples. For a Maxwellian distribution, characteristic temperatures of 500 to 600 eV due to plasma charge exchange, and 50 to 350 eV due to ions at the plasma edge are obtained. Also a 5-40 keV component is observed due to neutral beam injection.

1. INTRODUCTION

The hydrogen isotope populations in the tokamak plasma boundary are comprised of two components: ions and neutrals. Their velocity and spatial distributions are important for considerations of energy and particle transport, and more general plasma diagnosis. In this paper we have extended the proposal of Erents et al. [1] and earlier work of Staudenmaier et al. [2] to allow interpretation of the hydrogen energy distributions and fluences from hydrogen trapping and profiling measurements. The experimental and theoretical work reported here emphasize the importance of the depth distribution as well as the total concentration of implanted hydrogen.

2. EXPERIMENT

Samples were exposed in the Princeton Large Torus (PLT) tokamak using retractable probe drives. The experimental configuration and results from earlier exposures are given in Refs. 3 and 4. Details of the present exposures, which have been made since the beginning of neutral beam injection (NBI), are summarized in Table I. These samples were exposed to a small number of high power discharges.

The position of the samples of the earlier sets [3,4] was a few centimeters recessed from the outer wall, which is 50 cm from the centerline of the

plasma. In these recessed positions most hydrogen which reaches the samples must be charge exchange neutrals. The samples of set E were located 56 cm from the plasma center in a port which also housed a neutral beam line. These samples were not in the direct path of the neutral beam.

The samples of set F were 2 silicon strips and 7 (papyex) carbon foil strips mounted circumferentially around a 12 cm long, 3 cm diameter cylindrical holder inside a Ta shield with 2 diametrically opposing slits. This permitted simultaneous sample exposure on both the side facing towards (ion side) and away from (electron side) the toroidal current. By rotating the sample holder different regions along the strips were exposed to individual shots; each strip was at a different minor radius. The position of the sample holder is illustrated schematically in Fig. 1a. The inner-most sample was at $r = 45$ cm, and hence experienced ion fluxes orders of magnitude greater than the samples at $r \geq 50$ cm. Graphite was chosen for its ability to trap large amounts of hydrogen before saturating and for the clean surface and low Z which make it well suited for analysis of deposited impurities. After exposure, the sample holder (Al) and sample shield were examined. Arc tracks were found on both. No other sign of melting was evident. Based on this, as well as thermocouple measurements of the probe and shield, we conclude that the sample temperature did not change sufficiently to desorb implanted H/D [5].

A variety of experimental techniques have been used to determine the composition of the near surface region of samples after exposure to plasma discharges. The techniques used are nuclear reaction analysis (NRA) for D, secondary ion mass spectroscopy (SIMS) and elastic recoil detection (ERD) for H and D, and ion backscattering spectroscopy (IBS) and sputter Auger electron spectroscopy (SAES) for heavier elements.

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† A U.S. Department of Energy Facility.

‡ Permanent address UKAEA Culham Laboratory, Abingdon, Ox14 3DB U.K.

TABLE I. Summary of Exposures in PLT

Sample Group	E-1, 2, 3 7/13/78	E-5 10/16/78	F 12/19/78
Plasma Parameters			
Plasma	D	H	D
Neutral Beams	H	D	None
Limiter	304SS, C	304SS	C
Limiter Radius	40	40	40
No. of Discharges	45, 39, 25	24	1, 1, 1, 2, 3, 6, 12
I_p (kA)	370, 300, 320	430	250
B_T (kG)	32	32	17
n_e ($10^{13}/\text{cm}^3$)	2-5	2.5	1
T_e (keV)	2.4, 2.3, 2.0	1.7	1.1
Sample Materials:			
D Retained	Si:3-9 (10^{15} cm^{-2})	Si:6.5	C:10-180 Si:0.4-2.5
Deposited Impurities	Be:4-5 (10^{15} cm^{-2})	Ti:110 O:70 Cu:37 Ta:6	Fe:2-18 Cu:3-28 Ta:2-13

3. DEPOSITED IMPURITIES

Impurity content (IBS) and depth profiles (SAES) were measured to ascertain their effect on the retained H. A summary of impurities collected on the samples is included in Table I. The lateral distribution of impurities over each sample surface was fairly uniform. The large amount of titanium ($\sim 0.02 \mu\text{m}$) on sample E-5 is due to its proximity to the evaporation source for the titanium gettering. Note that large amounts of O and H have been absorbed in this Ti layer.

Figure 1a shows the areal density of impurities versus the number of discharges for the sample from set F closest to the plasma on the ion side. For up to 12 discharges, the impurity level increases linearly with number of discharges. Samples 4 cm further out had 5 times less deposited impurities.

4. TRAPPING OF HYDROGEN ISOTOPES

4.1 Depth Profiles

The concentration of H and D in the samples has been measured as a function of depth using SIMS (Fig. 2). The depth scale for SIMS on Si is established by subsequent profilometry measurements. The accuracy is $\pm 10\%$. The depth scale for C is estimated using the ratio of C/Si sputtering yields. Its accuracy is approximately $\pm 20\%$, based on a calibrated marker implant at 40 keV. The absolute H, D concentration for Si was established against standard implanted samples to $\pm 10\%$ accuracy.

The depth distribution of H and D in Si samples exposed to PLT discharges of D plasmas with H NBI and H plasmas with D NBI are shown in Fig. 2b. The measurements show the profile of the injected isotope extending to a depth close to the normal incidence range [6] at the neutral beam injection energy of 40 keV. Since the concentration in this deep component is low, i.e., far below saturation,

Fig. 1. a) Impurity (ion side) and b) deuterium (ion and electron side) content of the samples from set F closest to the plasma as a function of number of discharges. Saturation trapping levels measured for laboratory monoenergetic implants in C are shown by the dashed lines.

one can assume that all of the incident energetic D or H has been retained, apart from the back-scattered fraction. The observed concentration of $2 \times 10^{19} \text{ D/cm}^3$ to a depth of $0.5 \mu\text{m}$ gives an estimate of the incident fluence of energetic D at the sample of the order 10^{15} D/cm^2 for the 25 shots seen by sample E-3. The shoulder at $0.2 \mu\text{m}$ is due to the half-energy component in the beam. A consequence of these deep components is that the amount of D, H in such materials as C would gradually build up over a large number of shots (10^6), reaching a higher saturation level ($\sim 10^{13} \text{ cm}^{-2}$) with NBI than found without NBI. This deep implantation of hydrogen may be due to charge exchange of circulating neutral beam particles or to charge exchange within the injector port.

The $1/e$ fall-off, λ , in the D profile in Fig. 2a is 10 nm for depths less than 80 nm, and 20 nm for depths from 80 to 160 nm. Table II gives the results of selected D profile measurements on Si and C samples of set F for single shot exposures. In all these tabulated cases, less than 5% of the total D was trapped in the deposited impurity layer.

4.2 Total Retained D, H

The retained deuterium in the "recessed" samples of set E was measured by NRA and is shown in Table I. This is only 2-3 times less than the amount found in the earlier (recessed) exposures of set A, B and C for exposures to ~ 100 times fewer discharges.

Figs. 2a and 2b. Depth distribution, determined by SIMS, of H and D in samples from sets E-2 and E-5. The calculated range for normally incident 40keV H (2a) and D (2b) are indicated. (The effect of the Ti layer (2b) was not included in the range calculation.)

Figs. 3a and 3b. Elastic recoil detection spectrum for Si samples exposed to the same PLT discharges as the samples used in the SIMS measurements of Fig. 2. Note the different vertical scales for 3a and 3b. The H and D concentrations in units of $10^{15}/\text{cm}^2$ are: 3a) 24 and 7.8 and 3b) 190 and 6.5.

The built-up impurity film is 10-100 times thinner here (Table I). The total retained D and H in the samples of set E was also measured by elastic recoil detection (ERD) [7]. Figures 3a and 3b show spectra obtained in this way for the same Si samples used for the SIMS measurements of Figs. 2a and 2b. In each case the incident beam was $10 \mu\text{C}$ of 2.3MeV ^4He , and the angles of the incident ^4He and detected H and D were 15° from the plane of the surface. The directly scattered ^4He was prevented from reaching the detector by a $10 \mu\text{m}$ Al foil. The ERD technique is useful since the area under the peaks allows rapid determination of both H and D content. Although the two samples contain similar amounts of D, this data shows that sample E-5 exposed to the H plasma with Ti gettering had an order of magnitude more H than sample E-2 of Fig. 3a. Combining the ERD data with the SIMS profiles shows that most of the retained H is locked in the Ti film.

Figure 1b shows NRA results for deuterium trapping measurements for the samples of set F as a function of number of discharges. Because these samples are actually immersed in the plasma, ion flow along the magnetic field will be a 10 to 10^3 times larger source of H,D than charge exchange, consistent with the observed higher concentrations in set F. The carbon strip was at $r = 45 \text{ cm}$ on the ion side and $r = 47 \text{ cm}$ on the electron side. We attribute the lower trapping on the electron side sample to its larger minor radius.

The fact that the amount of D retained does not increase linearly with the number of shots indicates that saturation effects are playing a role. This is consistent with the model described below. SIMS analysis of the carbon sample with ion side at $r = 47 \text{ cm}$ and electron side at $r = 49 \text{ cm}$ showed the same saturation behavior.

5. EVALUATION OF TEMPERATURES AND FLUENCES

5.1 Model

To relate the measured H/D depth profiles and concentrations to plasma parameters it is necessary to have data on the trapping of H/D ions in Si and C as a function of energy, fluence and angle of incidence. Normal incidence data on trapping vs fluence is available over most of the energy range required, 20eV to 40keV [3,4]. However, there is little data available for depth profiles and for bombardment at oblique angles of incidence. To remedy this, we formulate a model of H/D trapping in C or Si for arbitrary H/D energy distribution, angle of incidence distribution, and fluence. It is straightforward to extend this model to explain isotopic exchange without recourse to a direct ion-induced desorption mechanism.

The model uses projected mean range and straggling parameters from the TRIM code [9] for C and from the Brice code [10] for Si. The particle range is reduced by the cosine of the impact angle. Straggling is assumed to be a spherically symmetric Gaussian centered about the end of range. Loss of multiply scattered atoms from the front surface is taken into account by subtracting the image Gaussian [11]. This last approximation sets a lower limit on the backscattered fraction. In the code, after saturation levels are reached, additional implanted atoms are obliged to move to the next nearest trap towards the surface. This model was suggested to us by experimental D into C trapping results [8]. The only free parameter in the code is the H/D saturation concentrations which were obtained by fitting 50eV to 1keV trapping vs fluence data [2]. A saturation level of $2 \times 10^{22} \text{ D cm}^{-3}$ was found for D in papyx carbon, which is one-third of the value reported for 8keV D⁺ on pyrolytic carbon [8]. The saturation level depends on the range and straggling parameters used in the code. Some results from the code are shown in Fig. 4 where the total amount trapped (obtained by integrating the concentration distribution) is plotted as a function of fluence, Φ , for various temperature Maxwellian distributions. A cosine distribution of impact angles is used. The saturation value for Si required to fit the trapping data for Si [2] was $1.55 \times 10^{22} \text{ cm}^{-3}$ in agreement with other measurements [4].

Saturation occurs at a fluence, Φ , approximately equal to the maximum concentration times the ion range; and the ion range depends on energy. Thus true saturation is never reached with a continuous energy distribution. For a Maxwellian distribution the retained H/D increases as $\log \Phi$. The effect of the cosine distribution of impact angles is to increase reflection losses. At low fluences, $\Phi \leq 3 \times 10^{16} \text{ cm}^{-2}$, normally incident deuterons with energy less than 1keV have 3 times higher trapping probability than those with a cosine distribution. At $\Phi \geq 10^{19} \text{ cm}^{-2}$ this discrepancy is reduced to ~30%.

The depth profile has a nearly exponential tail. The 1/e fall-off, λ , is plotted in Fig. 5 for D bombardment of C and Si. Little difference is seen

between the normal incidence and the cosine distributions. This is because particles incident at oblique angles saturate the surface and only those with near normal incidence penetrate to the tail of the distribution.

5.2 Application to Data Analysis

If the incident fluence is known the ion temperature may be determined from measuring the trapping levels in the saturation region (Fig. 4). However by measuring depth profiles and utilizing Fig. 5 the temperature can be determined without fluence or absolute concentration measurements. If absolute concentrations are also measured then the fluences can be obtained from Fig. 4. The temperatures and fluences of the fluxes incident on various samples exposed in PLT and listed in Table II, were obtained in this way.

Fig. 4. Calculated D trapping in carbon as a function of D temperature and fluence. The impacting D was assumed to have a Maxwellian velocity distribution and a cosine distribution of impact angles.

Fig. 5. Calculated 1/e fall-off, λ , in depth distribution of a Maxwellian velocity distribution D implant in carbon and silicon. Results for both normal incidence and cosine impact distribution are shown.

TABLE II. Calculated D Temperatures and Fluences

Sample	r(cm)	Measured		Calculated	
		D/cm ² (10 ¹⁵)	A(nm)	T(eV)	Φ(10 ¹⁷ cm ⁻²)
C(F)	47	30±10	12.5±2.5	230±50	1.1 ^(+1.5) _(-.3)
C(F)	48.5	10±3	6.0±1.2	100±20	.45 ^(+0.4) _(.08)
Si(F)	47	2.5±.2	6.5±.6	155±20	.13±.02
Si(F)	52	.04±.03	3.5±.4	73±7	.022±.003
Si(E-2)	56	7±2	10.4±1.0	280±40	.3±.1
		2±.4	19.5±2.0	560±60	.07±.02

For concentrations near saturation, modest uncertainties in depth scale (and hence in temperature) or in H₂D concentration can lead to large uncertainties in fluence. To avoid this, two approaches are possible. Firstly, standard plasma diagnostics can be used to measure n_e at the probe position so that fluence and temperature can be estimated in a self-consistent way using H₂D concentrations alone. The second approach is to make trapping measurements over several decades of (unknown) fluence. The trapping vs fluence curve will then fit only for one temperature - provided that the plasma distribution function at the probe is given by a single temperature Maxwellian. Further complications which have still to be considered are deviations of the angular distribution from the cosine, the effect of surface topography on the trapping probability, and the effect of sheath potential on the angular and energy distributions. Over the range of energies and fluences (up to 10¹⁹/cm²) that we consider, erosion is unimportant.

The radial temperature profile measured in set F shows a slow fall from an average of ~180eV at r = 47 cm to 73eV at r = 52 cm. In contrast, the fluence falls from ~10¹⁷/cm² to 2 x 10¹⁵/cm². These point to the ions as the source of the implanted H₂D at r ≤ 47. The sample at r = 56 cm (set E-2) saw a fluence of 0.4 x 10¹⁵/cm²/discharge at temperatures of 280 and 560 eV. This is in line with the expected flux of charge exchange neutrals. Thus we infer that the r = 52 cm electron side set F sample (see Fig. 1a) that saw a temperature of 75eV was being bombarded by charge exchange neutrals reflected from the wall, or by the low density plasma. We stress that the temperatures inferred from the depth profiles depend on the energy dependence of the deuterium range which, at present, has not been measured at these low energies.

6. CONCLUSION

The model presented here appears promising for diagnosing the temperature and flux of particles escaping the plasma. However, the results obtained from the analysis of the trapped flux are as yet rather limited. Much more data are required to get a complete picture of the flux and temperature of particles arriving at the walls and limiters in tokamaks. The technique outlined shows promise of

being able to make measurements down to much lower energies than conventional charge exchange stripping cells. Another advantage of this technique is the relative ease of absolute calibration. The surface probe can also make direct measurements of the ion flux right up to the edge of the limiters provided the power flow is not so great as to thermally desorb the implanted ions. Data on the energy and fluxes to all surfaces will allow accurate quantitative estimates of the power loss through the ion channel, together with the impurity flux from the walls by such mechanisms as sputtering. Finally, the amount of gas trapped in these surfaces as a function of fluence is the direct information which we require in order to estimate tritium inventories in D-T breeding systems.

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