2
JUNE 1978

PPPL-1448

CONF-780767-2

UC-20f

LONG-TERM CHANGES IN THE SURFACE CONDITIONS OF PLT

BY

S. A. COHEN, H. F. DYLLA,
S. M. ROSSNAGEL, S. T. PICRAUX,
J. A. BORDERS AND C. W. MAGEE

PLASMA PHYSICS LABORATORY

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

PRINCETON UNIVERSITY
PRINCETON, NEW JERSEY

This work was supported by the U. S. Department of Energy Contract No. EY-76-C-02-3073. Reproduction, translation, publication, use and disposal, in whole or in part, by or for the United States Government is permitted.

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the United States Energy Research and Development Administration, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

Printed in the United States of America.

Available from
National Technical Information Service
U. S. Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22151
Price: Printed Copy \$ * ; Microfiche \$3.00

<u>*Pages</u>	NTIS
	<u>Selling Price</u>
1-50	\$ 4.00
51-150	5.45
151-325	7.60
326-500	10.60
501-1000	13.60

LONG-TERM CHANGES IN THE SURFACE CONDITIONS OF PLT

S. A. Cohen, H. F. Dylla, and S. M. Rossmagel
Princeton Plasma Physics Laboratory
Princeton, New Jersey 08540

and

S. T. Picraux and J. A. Borders
Sandia Laboratories
Albuquerque, New Mexico 87115

and

C. W. Magee
RCA Laboratories
Princeton, New Jersey 08540

ABSTRACT

Long-term changes in the surface conditions of the PLT vacuum vessel wall have been monitored by the periodic analysis of a variety of sample substrates (stainless steel, alumina, silicon), exposed to PLT discharges for periods of up to several months and subsequently removed for analysis by Auger electron spectroscopy (AES), photoelectron spectroscopy (ESCA), ion backscattering, nuclear reaction analysis, secondary ion mass spectrometry (SIMS), and scanning electron microscopy. Samples exposed for extended time periods (2-6 months) showed deposited films containing limiter (W) and liner constituent metals (Fe, Cr, and Ni) and C and O. The film thicknesses ranged between 100-200 Å with 2-15 atomic percent W and 5-40 percent Fe as determined by sputter-AES and ion backscattering measurements. Increased deposition of metallic impurities (W, Fe) was noted following the first extensive application of low power discharge cleaning. We discuss possible mechanisms responsible for the deposition of metals onto the sample surfaces. Deuterium retention was observed in all the exposed samples with the deuterium depth profiles restricted primarily to the deposited films on the stainless steel substrates and extending deeper for Si. The deuterium retained in the exposed samples shows a saturation at $(1-11) \times 10^{15}$ D atoms/cm² for an estimated variation in the deuterium fluence of $10^{17}-10^{19}$ D atoms/cm².

1. Introduction

The surface composition and gas sorption properties of the inner vacuum vessel wall of a tokamak are dynamic characteristics of the device. The surface properties at the start-up of a tokamak are the end result of some type of vacuum preparation or chemical cleaning of the vessel material. However, with exposure of the vacuum vessel to discharge cleaning cycles and high power discharges, the wall will evolve in composition and sorption properties in a manner that is both difficult to predict a priori and to simulate in a laboratory device. The wall may be eroded due to a variety of possible mechanisms (desorption, sputtering, arcing, etc.) which generate plasma impurities, and subsequent plasma transport of impurities may deposit material onto the wall. This modified surface of the wall is the wall component subjected to subsequent plasma-wall interactions; it is both a source and sink of plasma impurities and the working gas. Thus, a study of wall modification in tokamaks will contribute to the understanding of the problems of impurity generation and hydrogen recycling.

As a means of monitoring the evolution of the surface conditions of the PLT tokamak, we have exposed for periods up to six months a variety of sample substrates (stainless steel, alumina, silicon) at a position recessed approximately 4 cm from the vacuum vessel wall. The samples were subsequently removed and analyzed using several analytical techniques to assess changes in surface composition, topography, and hydrogen retention. All of the exposed samples show extensive modification in surface composition and hydrogen content. This study of the compositional changes and hydrogen retention was initiated to quantify the integral surface changes in the actual vacuum vessel wall and the effects of these changes on discharge behavior.

In Sect. 2 of this paper we describe the experimental techniques used for analysis and in Sect. 3 present the resulting measurements. In Sect. 4 we discuss impurity generation mechanisms and the transport of impurities to the sample surfaces. In Sect. 5 we discuss the hydrogen retention results. Finally, in Sect. 6 we discuss the implications of these studies for future work.

2. Experimental Procedure

The sample materials chosen for this study were: (1) stainless steel (304), the primary structural material in the PLT vacuum vessel [1]; (2) alumina, because of its use as a ceramic break at two positions in the torus and at numerous other locations for electrical insulation and standoffs; and (3) single crystal silicon, because of its trapping properties and suitability for accurate hydrogen (deuterium) profiling measurements. The samples were exposed at a diagnostic port at the midplane of the torus 160° from the toroidal position of the W limiter and 55 cm from the centerline of the plasma. The furthest extent of the outside W limiter was 46 cm and structural components of the vacuum vessel wall extend from 48 cm to 51 cm (see Fig. 1). Groups of samples were exposed to three extended periods of six months, two months, and two months, respectively, of ohmically heated operation of PLT and were subsequently removed for analysis following each interval. In addition, one group of samples was exposed to neutral beam heated discharges for a much shorter interval (< 1 day). With the transfer of the samples from PLT to the analytic equipment, precautions were taken to minimize airborne and handling contamination; however no attempt was made for vacuum to vacuum transfers of the samples. All the samples were partially shielded with thin (.01 cm) stainless steel or V foil to allow comparison between exposed and

shielded sample areas.

A variety of surface analytical techniques were used to characterize the samples following exposure in PLT. Auger electron spectroscopy, sputter-Auger profiling, scanning electron and scanning Auger microscopy were performed on the samples in a high resolution (0.2 μm) scanning Auger microprobe [2a]. Additional sputter profiling and photoelectron spectroscopy were performed in a similar UHV apparatus which included a higher resolution electron spectrometer [2b]. Ion backscattering measurements [3] using incident 2 MeV ^4He were used to cross check the Auger spectroscopy measurements. Two techniques were employed to quantify and profile the hydrogen retained in the exposed samples. (1) For profiling deuterium in the stainless steel, Si and Al_2O_3 samples, a nuclear reaction $\text{D} (^3\text{He}, \alpha) \text{H}$ was used with an incident 700 keV ^3He beam [4]. The D profiling was accomplished by energy analysis of the emitted α particles at a laboratory angle of 77° . (2) For profiling both D and H in the Si samples secondary ion mass spectrometry (SIMS) was used [5]. Both hydrogen profiling techniques were calibrated against standard implantation samples.

3. Results

The results of the various sample exposures and subsequent analyses are summarized in Table 1. All the samples which were exposed for an extended period of time, (i.e., all sample groups except group D), showed a deposited metallic film with a thickness ranging between 100-200 Å. The notable difference among the long-term exposures is the considerably larger quantity of metal (Fe, Cr and W) deposited on the group B samples, which were exposed during the first application of low temperature discharge cleaning [6]. Examples of sputter-Auger depth profiles of the films deposited on samples exposed in groups A, B, and C are shown in Fig. 2. In Fig. 3 the backscattering spectrum

is shown for the Si sample from group C. These data show that the elemental makeup of the films is a mixture of the wall constituent metals, primarily Fe and Cr (Ni was usually < 2 at. %), the limiter material W, and the primary low Z plasma impurities C and O. (Cl has also been identified as an impurity in PLT plasmas and it was seen in the Auger spectra as a surface contaminant on most films with a typical coverage $\leq 10\%$ of a monolayer.)

Photoelectron spectroscopic measurements of the Fe in the deposited films on the group B alumina sample show the Fe $2p_{3/2}$ transition to be characteristic of oxidized Fe rather than free metallic Fe. Additionally, as the sample was further sputtered, there was no evidence in the Auger or photoelectron transitions of Al for the reduction of Al_2O_3 at the deposited film- Al_2O_3 interface.

Scanning Auger micrographs of the samples from group B and C show that the W deposition was spatially uniform over distances less than $10\ \mu$. Higher resolution ($< .2\ \mu m$) electron micrographs of the same samples show no evidence of blistering or hydrogen-induced, stress-corrosion cracking.

Profiles of the retained deuterium in the stainless steel and Si samples of group A and C measured by nuclear reaction analysis are shown in Fig. 4. The absolute accuracies are estimated to be within $\pm 20\%$ and the relative accuracies $\pm 10\%$ for the deuterium concentration and for the depth scale. Deuterium profiles for the Si and stainless steel samples from group D, which were exposed to only 52 high power discharges, are shown in Fig. 5a. In Fig. 5b is shown the retained deuterium and hydrogen profiles in the group D Si sample as measured by SIMS.

4. Impurity Measurements

The results summarized in Table 1 indicate the build-up of metallic

layers on the sample surfaces exposed to PLT discharges for extended time periods (i.e., groups A, B, and C). We shall discuss possible mechanisms for the introduction of these impurities (particularly the metals) into the plasma and the subsequent deposition onto the sample surfaces. Secondly we discuss the effects of such films on hydrogen isotope retention and recycling.

The original source of stainless constituent metals is certainly some part of the stainless structure of the vessel. Possible mechanisms of Fe (Cr, Ni) introduction include (1) unipolar arcing [7,8], (2) metallic dust dislodged from the wall [9,10], (3) evaporation or ablation by runaway electron impact, (4) chemical transport by volatile compounds such as M_xO_y or M_xCO_y , and (5) desorption or sputtering by charge exchange neutrals or cross-field diffusing ions impacting on areas of the vacuum vessel wall. The original source of W in PLT was the W limiters, however due to the subsequent dispersal of W around the vacuum vessel the wall now represents a secondary source of W. All of the mechanisms indicated above for the introduction of Fe are also possible sources of W. In addition, since the limiter forms the plasma current aperture, sputtering by the impact of streaming ions is a possible impurity source.

To relate the average surface deposition $\bar{n}_s$ per discharge to the plasma impurity concentration, $\bar{n}_i$, (which is assumed to represent a spatial average over the plasma cross-section and a temporal average over the discharge duration), we use the expression,

$$\bar{n}_s = \bar{n}_i \left(\frac{t_d}{\tau_p} \right) \frac{V}{A} \quad (1)$$

where t_d is the discharge duration, τ_p is the particle confinement time, and V/A the vacuum vessel volume to surface area ratio (for PLT $V/A \approx 20$). Implicit

in Eq. 1 are the following assumptions: the deposition is uniform over the vacuum vessel surface area; the recycling of impurities is negligible; and all the impurities deposited on the wall exit the plasma core during the steady state. Except for localized perturbations at the limiter position and possible asymmetries due to particle drifts, the assumption of uniform deposition is reasonable during the steady state portion of the discharge. Negligible recycling has been established for the heavy impurities (metals) [11] but this is not the case for the light impurities (i.e., C and O) [12,13]. This is corroborated in the present experiment by comparing the observed average deposition rates with those predicted from Eq. 1 using values of $\bar{n}_i$ inferred from spectroscopic measurements. Typically, Fe impurity levels in PLT are $(1-3) \times 10^{10} \text{ cm}^{-3}$ [14]. Assuming that τ_p for Fe is 100 ms ($t_d = .5\text{s}$ typically), Eq. 1 yields $\bar{n}_s = (1-3) \times 10^{12} \text{ cm}^{-2}$ for the average deposition per discharge. This agrees reasonably with the measured average deposition on sample groups A and C of $(3 \pm 1) \times 10^{12} \text{ cm}^{-2}$ and $(1 \pm .5) \times 10^{12} \text{ cm}^{-2}$ respectively. The observed Fe and W deposition on group B samples is an order of magnitude higher. (Possible reasons for the increased deposition during the group B exposure period are discussed below.) Reliable average deposition rates for the light impurities (C and O) are not derived from the sample measurements in Table 1 because a vacuum transfer was not used between exposure and analysis. However, in situ AES analysis of oxygen deposition rates at the PLT wall radius [15] have yielded values that are typically $<.01$ of the values obtained from vacuum ultra-violet spectroscopic measurements. A unity recycling coefficient for oxygen (from saturated surfaces) would be consistent with the observed discrepancy between oxygen surface outflux and VUV influx measurements.

An important impurity problem in PLT has been due to W [6]. Certain classes of PLT discharges have central radiation emissions (in a band 50-70 $\text{\AA}$)

exceeding $1\text{W}/\text{cm}^2$ which has been ascribed to reducing W ions [16] at an estimated concentration of $\bar{n}_W = (3-4) \times 10^{19} \text{cm}^{-3}$. We will discuss the possible impurity introduction mechanisms for W and present arguments to show that impurity ion sputtering and unipolar arcing of the limiter are the most likely sources of W.

4.1. Impurity Generation Mechanisms

4.1.1. Wall sputtering by charge exchange atoms (H,D,He).

Using the sputtering coefficients compiled of Behrisch, *et al.* [17], we estimate the upper limit for the W concentration in the plasma:

$$\bar{n}_W = \frac{A}{V} c \tau_W \int \Gamma_i(E) S_i(E) dE, \quad (2)$$

where A/V = PLT area to volume ratio

c = correction factor for non-normal H^0 impact ~ 1.8

$\Gamma_i(E)$ = charge exchange out-flux of H, D or He

$S_i(E)$ = sputtering yield of W

τ_W = W confinement time ~ 100 msec.

Assuming that the equivalent of 10% of a monolayer of W covers the PLT walls (q.v., see sputter-Auger profile in Fig. 2), we estimate that for $T_i(0) \sim 500$ eV, $\bar{n}_W \approx 2 \times 10^{19} \text{cm}^{-3}$ for pure W sputtering yield, or $\bar{n}_W \approx 4 \times 10^{19} \text{cm}^{-3}$ for W in a stainless steel alloy [18].

On the basis of the above crude calculation wall sputtering by H (D, or He) is a marginal source. Ruling against its importance we note that this process should strongly depend on ion temperature and working gas, and we have not observed 50% increases in $\bar{n}_W$ after switching to D from H, or when T_i has been high (1200 eV vs. 500 eV). Thus, we discount this mechanism as being responsible for most of the W influx.

4.1.2. Limiter sputtering by multiply ionized impurity ions.

The impact of multiply ionized impurity ions on the limiter is a much more effective sputtering mechanism than proton impact, because sputtering yields usually scale both with the mass and effective charge of the impactor particles: n -times ionized atoms impact with nV_1 more energy than protons (where V_1 is the potential difference between the limiter and the plasma). So O^{+6} impacting on a limiter with $V_1 = -100$ V would have 600 eV of energy and a sputtering yield 10^4 times higher than a 100 eV D^+ . The limiter potential is determined by the edge electron temperature and should approximately equal $4 T_e(a)$. $T_e(a)$ is decreased by impurity radiation from the edge, leading to the situation that both large and small impurity (oxygen) influxes will result in little W sputtering, while at modest O influxes (such that $n_O/n_e \sim 5-10$), the sputtering rate will be high (see Fig. 6). The maximum predicted W concentration is $\bar{n}_W \sim 5 \times 10^{10} \text{ cm}^{-3}$ and the corresponding average surface deposition per discharge is $\bar{n}_S \sim 5 \times 10^{12} \text{ cm}^{-2}$ (from Eq. 1 assuming $\tau_W = .1 \text{ s}$). This calculated limit for $\bar{n}_S$ is sufficient to account for the observed maximum deposition (group B, $\bar{n}_S \sim (4 \pm 1) \times 10^{12} \text{ cm}^{-2}$); moreover, the dependence of the mechanism on oxygen concentration qualitatively correlates with a drop in the oxygen impurity level in PLT from a 5-10% level during the group A period to the 1-2% level for the group B and C period when the metal deposition increased [6]. Systematic studies of the limiter potential versus $\bar{n}_W$ have not been made, but limiter potentials up to -200v (relative to the vacuum vessel) were measured during the group B period [19].

4.1.3. Unipolar arcs.

Another mechanism of W introduction which would show a similar qualitative dependence on edge temperature is the production of unipolar arcs on the limiter. As described by Robson [20], unipolar arcs of area 10^{-3} cm^2 with

current densities $\sim 10^6$ Amps cm^{-2} occur on any metal surface immersed in a plasma as long as the ion current to the total metal surface exceeds 10 Amps. Such is the case for limiters. Robinson also states that for copper, 10^{-4} g/coulomb is vaporized at the arc cathode spots. However, the erosion rate of a tungsten surface has not been measured. The W erosion rate need only be 10^{-3} of copper for the W density in PLT to be $\sim 4 \times 10^{10} \text{ cm}^{-3}$ for the expected ion saturation current to the limiter of 1000 A. Arcs, being initiated by field emission at microscopic protuberances and at the edge of cracks, should show a statistical variation in the WI - WII radiation from the edge plasma. The observation of radiation from the highly ionized species WXX - WXXI in the central plasma [16] may have this statistical variation smeared out, and thus is not suitable for diagnosing this mechanism.

Melt tracks have been observed on the smooth portion of the top and bottom PLT limiters (see Fig. 7). The tracks range in length from 0.1 to 20 mm, in width from 8 to 80 μm , and in depth from 1 to 10 μm . The tracks are at $4\text{-}12^\circ$ from the toroidal field normal, and in opposite directions on the top and bottom limiter, while the rotational transform angle has only been $\sim 2.5^\circ$. Also, by the shape of the track, we conclude that the arc motion is retrograde, i.e., in the $\bar{B} \times \bar{I}$ direction. The amount of W removed from each arc track is in the range 10^{16} to 10^{18} atoms, enough to account for the W observed in even the most contaminated discharge. The W from all the arc tracks is sufficient to account for all the W on the wall assuming the wall coverage is similar to that analyzed on the exposed sample surfaces. Similar observations have been reported by the DITE [7] and DIVA [8] research groups.

4.1.4. W dust dislodged from the PLT wall.

Ohkawa has described this effect [9]. A tungsten sphere $6 \times 10^{-3} \text{ cm}$ in radius contains sufficient atoms that only 30 particles would have to enter a

PLT discharge at the rate of 3 per 1 μ s need to account for $3 \times 10^{10} \text{ cm}^{-3}$ in the plasma. In 100 μ s, a droplet falls 5 cm, so it is possible to "feed" the discharge by gravity. Tungsten droplets with $r < 0.1 \text{ cm}$ dimension are abundant near the limiter because of runaway electron impact. Waelbroeck [10] has proposed hydrogen embrittlement of metals as another source for metal dust in tokamaks, but electron microscopy of the group A, B and C sample surfaces shows no features attributable to embrittlement. We estimate that the hydrogen flux (10^{15} - $10^{16} \text{ cm}^{-2} \text{ s}^{-1}$), total fluence (10^{18} - 10^{19} cm^{-2}), and incident energy range to which these samples have been subjected, would produce hydrogen concentrations below the levels necessary for the onset of these effects. Regardless of the source, this mechanism should reveal itself by the variation in the W influx, especially accompanying shocks to the vacuum vessel. As noted with the arcing mechanism, such variations would be most observable in studies of WI and WII radiation.

4.1.5. Limiter vaporization by runaway electron impact.

Localized heating of the limiter blades to $T \approx 4000^\circ\text{K}$ can occur at power depositions caused by MeV electron currents greater than 10 amp cm^{-2} for 1 μ s. If this mechanism were responsible for the W in PLT discharges, then the x-ray bursts from the limiter should precede the W influx. Though this occurs in some discharges, it is not the general rule [21]. (Runaways have caused the ejection of "meteorites" from the limiters and account for localized deposition of W onto the walls.)

4.1.6. Evaporation of volatile tungsten compounds from the limiter blades.

Three types of W compounds: oxides, halides, and carbonyls [22,23], readily evaporate at temperatures between 200 and 1500°C . In-situ surface analysis of exposed stainless steel have shown C, O and Cl to be present as surface

contaminant, with coverage of the order of 10^3 of a monolayer. Further, no correlation has been observed from the plasma, but does not increase with the W radiation. The large O background radiation from $\sim 5 \times 10^{11} \text{ cm}^{-3}$ O ions does not allow us to determine whether there is a 20% increase due to WO_2 influx. In fact, as noted in the discussion of oxygen ion sputtering, the W and O radiations have a negative correlation. Since the only portions of the tokamak that are heated above 200°C are the limiters, the evaporation area for these W compounds is only 500 cm^2 . One monolayer of W evaporated from this area would cause a W concentration of $(513) \times 10^9 \text{ cm}^{-3}$. Hence this mechanism is judged to be unimportant.

4.2. Impurity Transport to the Wall

Although there is good agreement, via Eq. 1, between the W and Fe observed in the plasma and the amount measured on the samples, the transport mechanism remains an open question. Radial impurity transport during the steady state part of the discharge is characterized by a diffusional velocity $V_D \sim (1-5) \times 10^3 \text{ cm s}^{-1}$ [14], not too different from the Bohm value. It is obvious from a schematic drawing of the region enclosing the samples (see Fig. 1b) that a small parallel velocity ($V_{\parallel}$ = thermal velocity, V_T) is required if this type of transport is responsible for the metallic deposition onto the samples. Assuming that the plasma ions have a 3eV temperature outside the limiter, then the fraction of W ions with a sufficiently low $V_{\parallel}$ to diffuse to the sample surface is given by

$$f = \left(\frac{m}{2\pi kT} \right)^{3/2} \int_0^{2V_D} \exp \left[- \frac{mV_{\parallel}}{2kT} \right] dV_{\parallel} \int_0^{\infty} \exp \left(- \frac{mV_{\perp}^2}{2kT} \right) 2\pi V_{\perp} dV_{\perp}$$

$$\approx .25 \left(\frac{V_D}{V_T} \right) \approx 5 \times 10^{-3}. \quad (3)$$

Thus, we can account for less than 1% of the observed deposition rate by a diffusive transport mechanism if "typical" values of V_D are assumed.

5. Hydrogen Isotope Retention

The recycle of hydrogen isotopes during tokamak operation is known to significantly influence the plasma discharge obtained. This has been demonstrated by the dramatic improvement in operating conditions obtained by titanium gettering experiments in several tokamaks. High density, low impurity discharges have been produced in ATC [24], DITE [25], PLT [26], ISX [27], and JFT-2 [28], when the hydrogen recycling was reduced by titanium evaporation of some fraction (.1-.4) of the vacuum vessel surface area. One way to better understand the hydrogen recycle problem is to directly measure the hydrogen retained in samples exposed at the first wall. In this paper we report, to our knowledge, the first direct measurements of total deuterium trapping and the D depth profile in the near surface region of samples exposed in a controlled fusion device [29].

As indicated in Table 1, all samples from groups A through D which represent the current operating life of PLT, were exposed to high power discharges with D_2 and were found to contain deuterium at concentrations ranging from $(1-11) \times 10^{15} \text{ D/cm}^2$ in the near surface region. When multiplied by the surface area of PLT ($\sim 2 \times 10^5 \text{ cm}^2$), this amount of retained D is comparable to the amount of D contained within the plasma during a given discharge ($10^{20} - 10^{21}$ atoms) and represents a potential source for cold hydrogen isotope recycle. The amount of D trapped varied by less than a factor of 10 even though the exposure times range from 6 months to one day and the number of high power discharges from 52 to $\sim 10^4$. This suggests that saturation of the trapping occurs in relatively short time (≤ 1 day) and that a hydrogen replacement mechanism becomes operative whereby newly trapped H or D replaces

previously trapped H or D which is re-emitted. Further evidence for this is given by the required time in the change-over from H to D plasmas in PRT after switching to D_2 fill gas (see Fig. 8).

The nature of the trapping is better understood by considering the D depth profiles for Si and SS in conjunction with the Auger and ion backscattering results. Considering the Si and SS sample results from groups A, C, and D, we note that group A has the most metallic deposited surface film and that the D profile is fairly similar in shape in the Si and SS samples (see Fig. 4a). In contrast, in group C and D samples the D profiles are resolution limited for the SS but not for the Si. In all cases, the Si is about three times as effective a trap for D as is the SS surface. We suggest that the D is readily trapped within the surface impurity film deposited on the samples, and in the bulk of Si, but not for the SS samples except perhaps in an intrinsic surface layer (based on group D trapping). There is evidence [30] that the trapping in Si results from displacements created by the energetic D ions. Appreciable damage induced by the exposure was directly demonstrated for the Si sample from group C by ion channeling measurements (see Fig. 3) which gave 1.6×10^{17} Si/cm² displaced relative to an unexposed region. The D depth profile extending into the Si sample several hundreds of Å beyond any surface film (see Fig. 5) may be a measure of the high energy tail of the energy distribution of charge exchange deuterium incident on the wall. At lower energies (≤ 1 keV) (shallower depths) the trapping efficiency would be expected to fall off rapidly as the threshold for Si lattice atom displacement is approached and also as the D concentration approaches saturation concentrations.

Studies of higher energy (13 keV) D implants in Si [30] indicate that ~ 100% of the incident D is trapped with fluences up to 10^{16} D cm⁻². Since the SIMS analysis of the group D Si sample (which was exposed during

40 keV D^0 injection showed less than 1×10^{-10} at depths greater than 2500 Å, we can set an upper limit for the fraction of D^0 beam atoms directly lost by charge exchange at 40 keV to be less than 10%.

The trapping of D in the (Fe, W, C, and O) surface film is believed to provide a major trapping mechanism on the SS samples from group A and C. A possible reason for such trapping may be the formation of strong chemical bonds, for example, with the C in the film. This would be analogous to D trapping in Si where the Si-D bonding is known to be strong and of a chemical nature [30]. Also, implanted D has been found to be strongly trapped and immobile at room temperature in graphite [31] and in W [32]. This is in contrast to implantation results for SS where only weak trapping is observed at room temperature, with the profiles extending far beyond the implanted range [33,34]. The SS sample from group D indicates, however, that even when the surface impurity layer is quite small, of the order of a monolayer of D can be trapped. This may be related to trapping in an intrinsic surface or surface oxide layer, and thus as long as SS304 is used for the first wall, the hydrogen isotope trapping problem will be present, even if the impurity problem can be controlled.

The D trapped in the SS samples was found to be released with continued bombardment by the 700 keV ^3He analyzing beam with about 50% loss after a fluence of ~ 750 μC . Such an effect was not observed to be appreciable in the Si samples. This further suggests a difference in the nature of the D trapping for the two types of surfaces and suggests the D is more weakly bound in the SS samples.

6. Implications and Future Plans

In the early operation of PLT the W limiters and the SS wall components provided the major sources of impurities. Relatively thick (> 500 Å) wall

coatings were deposited which now form the effective first wall.

During the most recent PLT operating period (July-Dec. 1977), preliminary experiments were performed with titanium gettering, a different limiter material (graphite), and neutral beam injection (with injected power up to 1 MW). All three experiments have important consequences relative to plasma wall interactions. During the next operating period, more extensive gettering will be tried, the original W limiters have been removed and replaced by three types (graphite, SS and Mo), and neutral beam injection power will be increased to 3 MW.

The analysis of samples exposed to the 1 MW power levels of neutral beam injection (group D) show a new metal impurity, Cu, which presumably was present in the discharge. The presence of Cu in the neutral beams has been confirmed directly by exposure of samples in the PPL Neutral Beam Test Stand and analysis by ion backscattering [35]. The most probable source of Cu in the beam is sputtering from the Cu accelerating grids in the ion sources. These results demonstrate the value of quantitative surface analyses of impurities in PLT, to lend better understanding of their origins and therefore to suggest possible methods of control.

Hydrogen isotope trapping and trapping profiles have established the level and nature of hydrogen retention at the first wall. Future studies will require exposure under a single type of discharge condition with possible vacuum transfers to the analysis systems. The group D samples with exposure to 52 high power discharges demonstrate that the techniques described here have sufficient sensitivity for such studies.

Furthermore, the present studies have led to the concepts of passive gauges for hydrogen fluxes and hydrogen recycle testing of coatings developed for interior components of tokamaks. For future devices it may be desirable

to develop first wall materials which will either trap all incident hydrogen isotopes and release the hydrogen in some subsequent cycle after completion of the discharge, or trap none of the hydrogen. This would be particularly important for D-T devices to minimize the tritium wall inventory [36]. In future studies amorphous Si layers will be introduced to monitor the total incident D, since very efficient trapping of energetically introduced D is expected for a-Si based on its known strong retention for hydrogen up to ~50 at. %. This in conjunction with SS, or test coatings, should provide a measure of the incident D flux and the fraction trapped in various materials. In addition, from the depth profile of D in a-Si it may be possible to infer the approximate incident D energy distribution since no D migration is expected after the D reaches thermal energies.

The ease of placing many small samples in remote torus locations gives this technique an advantage over standard charge exchange diagnostics for looking at (integrated) asymmetries in energetic hydrogen outfluxes.

Acknowledgements

The authors wish to acknowledge the assistance of R. Moore, G. Harper, J. McDonald, and the PLT experimental group. This work was supported by the United States Department of Energy Contracts EY-76-C-02-3073 and AT (29-1) 789.

REFERENCES

- [1] A description of the PLT vacuum vessel is given in S.A. Cohen,
J. Vac. Sci. Technol. 13 (1976) p. 449.
- [2] (a) Model 590 Scanning Auger Microprobe and (b) Model 555 Combined
Scanning Auger/ESCA analyzer manufactured by Physical Electronics
Industries, Inc. (Eden Prairie, MN, USA).
- [3] W.K. Chu, J.W. Mayer, M-A. Nicolet, T.M. Buck, G. Amsel, F.H. Eiden,
Thin Solid Films 17 (1973) p. 1.
- [4] J. Bøttiger, S.T. Picraux, N. Rud, in Ion Beam Surface Layer Analysis
vol. 2, ed. by O. Meyer, G. Linke, F. Küppler, Plenum, NY (1976) p. 811.
- [5] G.J. Clark, et al., Appl. Phys. Lett. 31 (1977) p. 582.
- [6] S. von Goeler, et al., in: Proc. Eighth European Conference on
Controlled Fusion & Plasma Physics (Prague, Czechoslovakia, 1977)
p. 17-28.
- [7] D.H.J. Goodall, et al., see paper this conference.
- [8] K. Ohasa, et al., see paper this conference.
- [9] T. Ohkawa, General Atomic Company Report No. GA-A14232 (1976).
- [10] I.A. Kahn, K.J. Dietz, F.G. Waelbroeck, and P. Wienhold (unpublished).
- [11] S.A. Cohen, J.L. Cecchi, and E.S. Marmor, Phys. Rev. Lett. 35 (1975)
p. 1507.

- [12] J.L. Cecchi, Princeton Plasma Physics Laboratory Report No. PPPL-1256 (1977).
- [13] DIVA group (to be published in Nucl. Fusion).
- [14] E. Hinnoy, D. Johnson, E. Meservey, and S. von Goeler, Bull. Am. Phys. Soc. 21 (1976) p. 1159.
- [15] H.F. Dylla and S.A. Cohen, Trans. Am. Nucl. Soc. 26 (1977) p. 149.
- [16] E. Hinnoy and M. Mattioli, Princeton Plasma Physics Laboratory Report No. PPPL-1375 (1977).
- [17] R. Behrisch, et al., Erice Summer School on Tokamak Reactors for Breakeven (1976).
- [18] The sputtering yield of W in a stainless steel matrix has been estimated using the theory of Sigmund [see P. Sigmund, Phys. Rev. 184 (1969) p. 383] and a sublimation energy of 5 eV.
- [19] K. Bol, private communication.
- [20] A.E. Robson, et al., Proc. Phys. Soc. 73 (1959) p. 50.
- [21] J. Strachan, private communication.
- [22] M.J. Cardillo and Y. Look, Surface Sci. 66 (1977) p. 272.
- [23] Handbook of Chemistry and Physics, R.C. Weast, ed., 56th edition, (CRC Publishing Co., Cleveland, OH 1975).
- [24] P.E. Stott, C.C. Daughney, R.A. Ellis, Nucl. Fusion 15 (1975) p. 431.

- [25] J.W.M. Paul, et al., in: Proc. Eighth European Conference on Controlled Fusion & Plasma Physics (Prague, Czechoslovakia, 1977) p. 49-62.
- [26] K. Bol, Bull. Am. Phys. Soc. 23 (1978) p. 187.
- [27] R. Colchin, et al., see paper this conference.
- [28] S. Konoshima et al., see paper this conference.
- [29] G.M. McCracken, et al., Nucl. Fus. 18 (1978) p. 1. This reference reports measurements of hydrogen retention in carbon samples exposed in DITE by thermal outgassing of the samples.
- [30] S.T. Picraux and F.L. Vook, submitted to Phys. Rev. B.
- [31] R.A. Langley, J. Roth, and R.S. Blewer, these proceedings.
- [32] S.T. Picraux and F.L. Vook, J. Nucl. Materials, 53 (1974) p. 246.
- [33] C.J. Altstetter, et al., J. Vac. Sci. Technol. (in press).
- [34] K.L. Wilson and M.I. Baskes, see paper this conference.
- [35] M. Ulrickson and J.A. Borders, private communication.
- [36] J.L. Cecchi, Princeton Plasma Physics Laboratory Report No. PPPL- (1978).
- [37] N. Laegreid and E.K. Wehner, J. Appl. Phys. 32 (1961) p. 365.

TABLE 1
LONG-TERM INTEGRATED SURFACE MEASUREMENTS IN P.T.

	Sample Group A	Sample Group B	Sample Group C	Sample Group D
Sample Materials	SS, Si, Al ₂ O ₃	SS, Si, Al ₂ O ₃ , V	SS, Si, Al ₂ O ₃ , e	SS, Si
Exposure Time	July - Dec. 1976	Jan. - Mar. 1977	Apr. - May 1977	Dec. 8, 1977
Discharge History ^a	H ₂ PDC ~10 ⁴ HPD (H ₂ , D ₂ , He)	H ₂ PDC H ₂ TDC ~(2-4)×10 ³ HPD (D ₂ , H ₂)	H ₂ TDC ~(2-4)×10 ³ HPD H ₂ , D ₂ ~5×10 ² HPD He ~10 ² HPD, Ne, C ₄	52 HPD in D ₂ with ~1 MW NBI (D)
Film Thickness ^b	~ 100 Å	~ 200 Å	~ 200 Å	< 20 Å
Film Composition ^c	Fe: 3.5 × 10 ¹⁶ cm ⁻² Cr: 9.0 × 10 ¹⁴ cm ⁻² W: 4.3 × 10 ¹⁵ cm ⁻²	Fe: 1.6 × 10 ¹⁷ cm ⁻² Cr: } W: 1.6 × 10 ¹⁶ cm ⁻²	Fe: 2.9×10 ¹⁵ cm ⁻² Cr: 5.0×10 ¹⁴ cm ⁻² W: 5.7×10 ¹⁴ cm ⁻² C: 6.2×10 ¹⁶ cm ⁻² O: 3.2×10 ¹⁶ cm ⁻²	Cu: 4.0×10 ¹⁴ cm ⁻² W: < 5×10 ¹² cm ⁻² Fe: < 1×10 ¹⁴ cm ⁻² Ti: 1×10 ¹⁴ cm ⁻² S: 8×10 ¹⁴ cm ⁻²
Total D Retention ^d	11.4×10 ¹⁵ cm ⁻² (Si) 3.8×10 ¹⁵ cm ⁻² (SS)	1.9×10 ¹⁶ cm ⁻² (Al ₂ O ₃)	9.7×10 ¹⁵ cm ⁻² (Si) 3.1×10 ¹⁵ cm ⁻² (SS)	3.8×10 ¹⁵ cm ⁻² (Si) 1.5×10 ¹⁵ cm ⁻² (SS)

^aPDC = high power discharge cleaning pulses, TDC = low power (Taylor-type) discharge cleaning pulses, HPD = high power discharges, and NBI = neutral beam injection, see reference [6].

^bFilm thickness measurements are derived from Ta₂O₅ equivalent sputtering rates with 5 keV A⁺.

^c2.0 MeV ⁴He ion backscattering measurements on the Al₂O₃, Si, or Be samples.

^dD(³He, α) H measurement on Si, Al₂O₃ or stainless steel (SS) samples.

FIGURE CAPTIONS

Fig. 1. (a) Schematic diagram showing the exposure position of the samples, and the surface analysis station (SAS) probe [15] relative to the structure of the PLT vacuum vessel. (b) Approximate geometry of the sample placement used for the calculation given in the text of the fraction of streaming tungsten ions which intercept the samples.

Fig. 2. Sputter-Auger depth profiles of the following samples: (a) Si exposed in group A; (b) alumina (Al_2O_3) exposed in group B; (c) Si exposed in group C; and (d) stainless steel exposed in group C. See Table 1 for exposure conditions and corresponding ion backscattering measurements of total elemental concentrations.

Fig. 3. 2 MeV ^4He ion backscatter spectra from the single crystal $\langle 111 \rangle$ Si substrate exposed in group C. Spectra are shown for both random and $\langle 111 \rangle$ channeling orientations.

Fig. 4. Profiles of the retained deuterium in the following samples: (a) Si and stainless steel exposed in group A; and (b) Si and stainless steel exposed in group C. The nuclear reaction $\text{D}(^3\text{He}, \alpha)\text{H}$ with an incident 700 keV ^3He beam was used to profile the deuterium. Depth scales are converted from atoms cm^{-2} to approximate depth (in $\text{\AA}$) using the following atomic densities: $5 \times 10^{22} \text{ atom}^2 \text{ cm}^{-3}$ for Si and $8.5 \times 10^{22} \text{ atoms cm}^{-3}$ for stainless steel. See Table 1 for exposure conditions.

Fig. 5. (a) Deuterium profiles in Si and stainless steel samples exposed in group D as measured with the $\text{D}(^3\text{He}, \alpha)\text{H}$ reaction. (b) Deuterium and hydrogen profiles in the same Si sample as in (a), but analyzed using secondary ion mass spectrometry (SIMS).

Fig. 6. Predicted steady state W concentration caused by multiply-ionized oxygen sputtering of the limiter as a function of oxygen concentration in the plasma. The impacted ions were assumed to have an incident energy of approximately four times the edge electron temperature after acceleration through the limiter sheath potential. Because of the slow recombination rates, even at low edge temperatures, all impacting ions were assumed to be in the O^{+6} charge state. O/W sputtering coefficients have not been measured so an extrapolation of data from Ne/W was used [37]. At high oxygen concentrations, the resulting edge cooling reduces the energy of the impacting ions, thus reducing the sputtering.

Fig. 7. Micrograph of the edge of the upper PLT tungsten limiter. Area A has been rolled during its fabrication, resulting in a cracked surface. Area B was ground before installation of the limiter in PLT. On Area B are numerous melt tracks at angles of $4-12^\circ$ from the limiter edge (which is $\perp$ to B_T). Most melt tracks originate (or terminate) at a large melt spot. Area C shows a magnified view of a melt track showing a crater-like trail characteristic of arcs.

Fig. 8. Ratio of the maximum partial pressures of H_2 relative to D_2 following the change-over of the fill gas from H_2 to D_2 . The measurement made on 11/1/76 followed four months of operation with H_2 , whereas the measurement made on 1/26/77 followed only two weeks of operation with H_2 .

Fig. 1. 783326

Fig. 2. 783366

Fig. 3. 783365

Fig. 4. 783363

Fig. 5. 783364

Fig. 6. 773792