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CHANGES IN SURFACE CONDITIONS WITH FIRST PLASMA  
IN THE PRINCETON LARGE TORUS (PLT)

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ABSTRACT

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We report in-situ measurements of the changes in surface composition of 305 stainless steel exposed to the first plasma discharges in PLT. Auger electron spectroscopy of pre-exposure surfaces shows a multilayer coverage of carbon and a fractional monolayer coverage of oxygen. Surfaces subjected to increasing levels of plasma exposure show substantial removal of the carbon multilayer, increased fractional-monolayer coverages of oxygen and iron, and tenth-monolayer quantities of chlorine and sulfur.

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**MASTER**

## Introduction

The importance of the surface conditions of tokamak vacuum vessels is well established, but only recently have in-situ measurements of tokamak surface conditions been attempted [1-3]. Herein we describe the surface analysis station (SAS) built for the Princeton Large Torus (PLT), and present results of measurements performed since the first day PLT began operation. The character of the "first plasma" discharges in PLT is dominated by wall effects. Specifically, the time evolution and temperature of the discharge are strongly limited by a large influx of (primarily) C and O atoms which are desorbed from the wall during the discharges. We present evidence of changes that occur in the PLT vacuum vessel wall surface conditions with plasma exposure.

## Experimental Apparatus

A diagram of the SAS is shown in Figure 1, and the details of its construction and operation are described elsewhere [1]. Instrumentation for Auger electron spectroscopy (AES), secondary ion mass spectroscopy (SIMS), and electron stimulated desorption (ESD) are contained in an ion-pumped, ultrahigh vacuum chamber. Sample surfaces are provided by a movable, 1.2 cm wide, 0.008 cm thick 305 stainless steel tape loop. The tape loop rides on an insulated support rail, one end of which is centered in the analysis chamber. The other end protrudes into the PLT vacuum chamber through a long bellows section. After the initial surface conditions of a segment of tape have been determined, the sample surface is translated to a position inside the PLT vacuum chamber for exposure to plasma. After exposure the sample surface is translated back to the ultrahigh vacuum chamber for analysis.

For the initial PLT experiments the sample surface was located 2.5 cm inside the PLT vacuum vessel wall and 4 cm behind the limiter radial position. The exposed region of the tape was further defined by a 0.25 cm Ta slit located 0.1 cm from the tape surface. There is no line-of-sight between the SAS and the limiter, the latter being positioned on the torus 180° relative to the former.

The details of PLT construction and a discussion of the vacuum system are given elsewhere [4]. A quadrupole mass spectrometer is mounted directly on PLT for residual gas analysis. The resulting mass spectra (Fig. 2e) show CO, H<sub>2</sub>O, and H<sub>2</sub> to be the primary residual gases (partial pressures in the 10<sup>-8</sup> torr range) with significant amounts (10<sup>-9</sup> - 10<sup>-8</sup> torr) of both saturated and unsaturated hydrocarbons. Therefore, it is reasonable to expect the stainless steel vessel walls of PLT to be covered with multilayers of various hydrocarbons, CO and H<sub>2</sub>O.

### Results

SAS experiments on PLT to date have been concerned with the effect of the first series of discharges on the hydrocarbon contaminated stainless steel walls. These first discharges are characterized by comparatively low temperatures and short discharge durations. This is a result of the radiational and ionizational cooling of the plasma by the influx of impurities desorbed from the wall. The plasma current (2a), loop voltage (2b), and two vacuum ultraviolet signals, a CIII line (2c) and a OIII line (2d) are shown in Figure 2 for a discharge typical of the high power mode of operation (toroidal field = 30 kG, repetition period = 50 sec/pulse). These data show a high loop voltage, indicative of a large resistive component to the plasma impedance. This is likely due to the impurities. The

continued increase in intensity of the CIII and OIII signals is evidence for an increasing flux of carbon and oxygen into the plasma.

The surface of the stainless steel tape in the SAS was used as a sample surface for all the data presented below. The tape surface was degreased with perchloroethylene and acetone prior to installation and was then exposed to the PLT residual gas for several days preceding the measurements. The PLT vacuum vessel was not baked in-situ and received only a similar degreasing prior to first plasma. Therefore, the sample tape surface closely approximates the condition of the vacuum vessel surface. In these experiments we report data using only Auger spectroscopy for surface analysis of the tape before and after exposure to the plasma.

Figure 3a shows Auger spectra of a position of the sample tape before exposure to plasma. A strong multilayer carbon signal (272 eV) and a smaller oxygen signal (510 eV) are the only strong Auger transitions visible. With the sensitivity increased a factor of five small iron signals (598, 651, 703 eV) from the underlying stainless steel substrate are visible. In addition, there are trace amounts of chlorine (181 eV) and sulfur (152 eV). Figure 3b shows the same region of tape after exposure to the first discharge cleaning in PLT. The tape was exposed to 670 H<sub>2</sub> discharges in the low power mode (toroidal field = 10 kG, repetition rate = 14 sec/pulse, plasma current = 90-105 kA, and length of discharge = 10-15 msec). Here a decrease in the C Auger signal and increases in the O, Fe, S, and Cl signals are seen.

The surface changes due to plasma bombardment are more apparent when the peak-to-peak Auger signals are plotted as functions of tape position as shown in Figure 3c. This shows the decrease in C and corresponding increase in O and Fe signals on the exposed portion of the tape. It is

reasonable to interpret these data as evidence for the discharge removing a fraction of the C overlayer and exposing an oxidized underlying substrate. (The Auger data could be reproduced by the deposition of Fe by the discharge on top of the C overlayer, but such a large Fe signal is inconsistent with the vacuum ultraviolet measurement of the bulk plasma Fe impurity level.)

Figure 3d shows the change in C signal replotted along with the changes in Cl and S signals versus tape position. The Cl signal shows a curious asymmetry in position with respect to the exposure slit unlike the other Auger signals. This effect was reproduced in subsequent runs, always showing a higher Cl concentration on the higher index of the exposure slit. The effect was shown to be unrelated to the plasma exposure but due to enhanced surface diffusion of Cl by the Auger spectrometer electron beam. Subsequent to these surface measurements, Cl was identified in the discharge using the vacuum ultraviolet spectrometer. The original source of Cl in the wall could be a result of the pickling process of the steel (HCl or  $H_2SO_4$ -NaCl mixtures are often used), or a residue of an applied Cl-containing degreasing solvent. Possible damage to the wall could occur if Cl segregated at grain boundaries or weld fractures in the bellows section. The effectiveness of Cl in penetrating passivation films in stainless steel leading to intergranular or stress corrosion is well known [5].

Figure 3c shows the C(272 eV) signal plotted with the low energy C(20 eV) signal. The mean escape depth [6] of 272 eV electrons is approximately  $8 \text{ \AA}$  or the equivalent of several (3-4) atomic layers of C, whereas the escape depth of 20 eV electrons in carbon is essentially one atomic layer. Therefore, the constancy of the C(20 eV) signal relative to the

significant decrease of the C(272 eV) signal at the exposure position is evidence for a decrease in the multilayer carbon coverage but not below the level of monolayer coverage.

Figure 4 shows Auger spectra of exposed tape positions that have received increased plasma exposure relative to the data of Figure 3. Figure 4a shows a new area of the tape exposed to 1000 low power discharges. In this trace substantial removal of the carbon overlayer is noted. The stainless steel substrate is sufficiently cleaned that Cr (529 eV) and Ni (848, 783 eV) transitions are visible in addition to the enhanced Fe peaks. Consistent with the data of Figure 3, an enhancement of the O, Cl, and S signals is seen at the exposure region. Also visible for the first time is a significant amount of Ta (179, 171 eV) which obscures the Cl in the trace shown (4a). The high energy Auger spectrum, shown in Figure 4d, is necessary to distinguish Ta from W which has a similar low energy Auger spectrum. The Ta is no doubt ablated by the plasma from the Ta exposure slit onto the tape surface. W would be an expected impurity by plasma transport from the W limiters, but the absence of W (1571 eV) and W (1796 eV) signals above the noise level in Figure 4d, shows that no W has yet been detected above the sensitivity limit (= 1% W coverage).

Figure 4b shows a third tape position exposed to 1000 low power discharges followed by 180 high power discharges (with I, V parameters as shown in Fig. 2a,b). In this trace the base metal (F, Cr, Ni) and O peaks are further enhanced, and the appearance of Cu (920 eV) is noted. A stronger Cu signal along with appearance of Ag peaks (351, 356 eV) are evident in Figure 4c, which is a retrace of the tape position of 4a after an additional exposure to five high power discharges. A possible source

source of this impurity in PLT is the presence of Ag-Cu brazing alloys at several positions in the torus, particularly at the connections to the ceramic breaks.

The difficulties of quantitative analysis of Auger spectra are a point of debate in the literature. However, an empirical method of analysis [7] can correct the raw data for the differing ionization cross sections and give approximate relative surface concentrations. On a typical unexposed tape surface, the relative O coverage is 2-4% of the C coverage, Fe and Cl are typically 1% and sulfur less than 1%. For comparison, the Auger spectra of the exposed surface shown in Figure 4c, give oxygen and carbon coverages that are approximately equal, with coverages of 17% Fe, 11% S, 36% Cl, and 25% Ta relative to the coverage of C.

#### Summary

In summary, significant removal of the original carbonaceous or hydrocarbon multilayer is observed with subsequent exposure of an oxidized stainless steel substrate. This result is in marked contrast to a similar experiment on the ATC tokamak [1] where the carbon coverage is found to increase with plasma exposure. In both tokamaks, increased quantities of chlorine and sulfur are seen on surfaces exposed to plasma. Finally, it should be noted that better plasma parameters are being attained in PLT (i.e., longer discharge times, decreased loop voltages, etc.) as the wall is being cleaned.

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## Figure Captions

- Figure 1. Schematic drawing of the surface analysis station (SAS) on the Princeton Large Torus (PLT).
- Figure 2. Characteristics of a typical high power  $H_2$  discharge: (2a) plasma current, 175 kA/div vertical, 10 msec/div horizontal; (2b) loop voltage, 20 V/div vertical, 10 msec/div horizontal; (2c) intensity of CIII (977 Å) VUV line, 5 msec/div horizontal; (2d) intensity of OIII (600 Å) VUV line, 5 msec/div horizontal; (2e) quadrupole mass spectrometer scan of residual gas with a total pressure =  $1.0 \times 10^{-7}$  torr.  $H_2$  fill pressure was typically  $5.0 \cdot 10^{-5}$  torr.
- Figure 3a. Typical Auger electron spectrum  $N'(E)$  of the sample tape before exposure to plasma showing C(272 eV) and O(510 eV) peaks. A scan at increased sensitivity (x 5) shows small substrate Fe peaks (598, 651, 703 eV) and S(152 eV) and Cl(181 eV) surface impurities.
- 3b. Auger spectrum of the sample tape after exposure to 670 low power  $H_2$  discharges (discharge cleaning). Note decrease in C peak and increase in O, Fe, Cl, and S peaks.
- 3c. Plot of peak-to-peak,  $N'(E)$  Auger signals versus tape position for C(272 eV), O(510 eV), and Fe(703 eV). Position of exposure slit is noted. One unit of tape index = 0.57 cm.
- 3d. Plot of peak-to-peak,  $N'(E)$  Auger signals versus tape position for C(272 eV), Cl(181 eV), and S(152 eV).
- 3e. Plot of peak-to-peak,  $N'(E)$  Auger signals versus tape position for C(272 eV), C(20 eV), and the ratio C(20 eV)/C(272 eV).

Figure 4a. Auger spectrum of a new sample tape position after exposure to 1000 low power  $H_2$  discharges. Note appearance of Cr(529 eV), Ni(848, 783 eV), and Ta(179, 171 eV) peaks.

4b. Auger spectrum of a third tape position exposed to 1000 low power discharges and 180 high power discharges ( $i_{max} \approx 350$  kA). Note trend of C(272 eV), O(510 eV), and Fe(703 eV) peaks in series of spectra shown in 3b, 4a, 4b.

4c. Auger spectrum of tape position shown in 4a after an additional exposure to five high power discharges. Note appearance of Cu(920, 940 eV) and Ag(351, 356 eV) peaks.

4d. High energy (1400-1800 eV) Auger spectrum showing Ta but no W(1571, 1796 eV) peaks above the noise level.

# SURFACE ANALYSIS STATION









