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FIELD DESORPTION AND FIELD ION SURFACE STUDIES OF SAMPLES EXPOSED TO THE PLASMAS OF PLT AND ISX*

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Modifications to the surface of field-ion specimens exposed to plasma discharges in PLT and ISX determined by Imaging Atom-Probe, Field Ion Microscope, and Transmission Electron Microscope analyses have in the past shown several consistent features. Surface films consisting primarily of limiter material with trapped plasma and impurity species have been found to reside on samples with direct line of sight exposure to the plasma during the discharges. Control specimens placed in the tokamak, but shielded from the plasma, on the other hand, remained free of deposits. When exposed to only high power plasma discharges, samples placed at the wall position in PLT and ISX have survived the exposures with no evidence of damage or implantation. In this paper we describe the results of a recent exposure in PLT in which for the first time samples of stainless steel were included for High-Field Surface Analysis. Tokamak operating conditions, including stainless steel limiters, titanium gettering between discharges, and the occurrence of a disruption, also distinguished this exposure from those carried out previously. Even with stainless steel limiters, contaminant films were found to be deposited on the samples at a rate (50-60 Å in six shots) comparable to earlier exposures with carbon limiters (500 Å in 69 shots).

1. INTRODUCTION

Surface and near-surface analysis of samples exposed to plasma discharges in the Princeton Large Torus (PLT) and Impurities Studies Experiment (ISX) tokamaks has recently become an almost routine procedure for characterizing plasma-wall/limiter interactions occurring during machine operation [1,2]. Information relating to impurity transport, discharge cleaning effects, wall/limiter erosion and damage, and hydrogen retention and recycle can be obtained from the analysis of samples positioned at various locations in the tokamak. Measurements of the depth distribution of plasma species implanted into irradiated specimens can also be used to quantify the energy and flux of escaping plasma particles. Various surface and near-surface sensitive techniques have been used in this type of investigation including Auger Electron Spectroscopy (AES), Scanning Auger Microscopy (SAM), Scanning-Transmission Electron Microscopy (STEM), Rutherford Ion Backscattering (RIBS), Secondary Ion Mass Spectroscopy (SIMS), and Nuclear Reaction Techniques [1,2]. In recent publications [3,4] we have presented the results of the first application of High-Field Surface Analytical Techniques (Imaging Atom-Probe Mass Spectroscopy [5] and Field Ion Microscopy [6]), to the study of plasma-wall interactions in PLT and ISX. These techniques, which offer greater spatial and depth resolution than the surface analysis procedures listed above, were shown to be very well-suited to the study of interactions occurring at the tokamak walls, and the early data indicated that structural and compositional changes not observable with other techniques could be detected. We also introduced a novel method for measuring the thickness of contaminant films deposited on sample surfaces during plasma exposure. This procedure, in which a high resolution Transmission Electron Microscope is used to image the profile of a sharply pointed field-emitter specimen, was shown to be a very simple, accurate (± 10 Å resolution), and nondestructive method for monitoring wall contamination. In this paper, we report the results of a very recent study in which high-field techniques were used to analyze samples exposed in PLT under somewhat anomalous conditions. The results are compared and contrasted to those obtained from the earlier exposures.

2. EXPERIMENTAL PROCEDURES

The specimens used in this investigation, as in all Imaging Atom-Probe (IAP) and Field Ion Microscope (FIM) studies, are sharply-pointed needles (called tips), typically prepared by electro-chemical etching of wire segments. Prior to their in-situ tokamak exposure, the specimens are field evaporated [6] to produce an atomically smooth surface and field ion imaged to characterize their initial surface morphology on an atomic scale. The tips are also imaged in a high resolution Transmission Electron Microscope to determine their macroscopic contour and

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TABLE I

EXPOSURE CONDITIONS FOR FIM SAMPLE SETS

Set	#1	#2	#3	#4
Specimen Material	W	W,Rh	W	W,302SS
Total Number of Specimens	8	8	4	5
Number of Control Tips	4	2	2	2
Tokamak	PLT	PLT	ISX	PLT
Exposure Date(s)	3 to 4/77	11/29/77	3/2/78	12/19/78
Type of Discharges	D,H	D	H	D
Number of Discharges	$\sim 10^3$,$\sim 10^4$	69	39	6
Neutral Beam Injections	0	48	0	3
Limiter Material	W	C	Mo,C	SS
Titanium Gettering?	No	No	No	Yes

to ascertain that the surface is free of visible deposits.

Following these initial characterization procedures, the samples are transported to the tokamak where they are placed at the wall position and exposed to a given number of plasma discharges. In each set a certain fraction of the samples have direct line of sight to the plasma during the discharges, while the remainder are shielded, allowing effects of exposure to the plasma vs. effects of exposure to the laboratory atmosphere and/or tokamak ambient to be distinguished.

Upon their return to our laboratory, the specimens are reimaged in the TEM and the thicknesses of any deposited surface contaminant layers are measured. Any gross structural damage resulting from plasma exposure will also be detected in this step of the analysis. The remaining measurements are carried out in the Imaging Atom-Probe/Field Ion Microscope surface analysis apparatus. If contaminant layers have been observed in the TEM, their compositions are determined by IAP mass analysis. Once the surface film is removed, a field ion image of the substrate is obtained to identify the possible existence of minor structural damage resulting from tokamak exposure which would not be resolvable in the electron microscope images. Finally, an Imaging Atom-Probe depth profile [7] is carried out, in which the abundance of implanted plasma species is measured as a function of depth from the surface with a depth resolution of ± 2 Å.

3. RESULTS AND DISCUSSION

To date, a total of four sets of FIM samples (containing between four and eight specimens per set) have undergone tokamak exposure and analysis. Table I lists the specimen material, exposure dates, and relevant tokamak parameters for each set.

3.1 Sets #1-#3

A detailed description of the data obtained from the analysis of the first three sample sets can be found elsewhere [3,4]. In this section only the major results are summarized for later comparisons with set #4.

Evidence for specimen damage resulting from in-situ tokamak exposure at the wall position of PLT or ISX was seen only in the first sample set. Here, FIM images revealed that lattice damage, extending to a depth no greater than ~100 Å from the surface, had occurred to the four samples with direct line of sight to the plasma during exposure; but not to the four control samples which were shielded. The fact that all of the exposed and none of the control tips suffered some degree of lattice damage conclusively demonstrated that the damage resulted from direct plasma exposure. In all subsequent exposures, however, no specimen damage was detected.

Since routine analysis of the specimen surfaces with the TEM was not initiated until exposure of the second sample set, no quantitative data from our measurements exist for the thickness of any deposited contaminant films on the surfaces of samples of set #1. Macroscopic samples exposed simultaneously with the first set and analyzed with sputter-AES and RIBS were found to have a deposited surface layer of thickness ~200 Å containing primarily C, O, Fe, Cr, and W. In our second sample exposure in PLT, all eight specimens were imaged in the TEM both before and after tokamak exposure. Semi-transparent films, whose thicknesses varied from 50 Å-500 Å depending on the size of exposure aperture in front of the specimens, were observed on the surfaces of all of the exposed tips. IAP analysis identified these films to be composed primarily of carbon (limiter material) with trapped plasma (H,D) and impurity (O,Fe,Cr) species within. Samples in set #3 exposed in ISX were also found to have carbon surface films

present, their thicknesses were typically ∿ 60 Å. However, no conclusions regarding the cleanliness of one machine with respect to the other can be made at this point, since the location of the samples with respect to the limiters and other differing machine parameters may have resulted in different exposure conditions. One consistent observation for both exposures has been that no contamination was ever found on the control samples, implying that direct line of sight to the plasma was a prerequisite for the formation of the deposited surface films.

The only occurrence of implanted plasma or impurity species within the substrate was found in the damaged areas of specimens from set #1. Here, the implantation was found to exist only within the region where atomic rearrangement had occurred and not in the underlying, undisturbed substrate. In the other sample sets plasma and impurity elements were detected only in the deposited films.

3.2 Set #4

In sample set #4, three tungsten and two stainless steel samples were exposed in PLT for a very limited number of discharges. As seen in Table I, this exposure represented the first time specimens of an actual wall material (a 300 series stainless steel) were exposed in a tokamak for high-field surface analysis. The tungsten tips were included for overall consistency, since all previous exposures used tungsten samples. One specimen tip of each material was shielded and served as a control; the other three had direct line of sight to the plasma during the discharges. Figure 1 shows schematically the position of the samples with respect to the vacuum vessel of PLT.

The tokamak discharges during the exposure interval for set #4 were not typical of normal PLT operation. An air leak developed near one of the neutral beam injectors during the course of the exposure, causing the final shot to be a disruption. Hence, any significant increase in contamination observed on the sample surfaces of this set with respect to the earlier exposures could be attributable to this disruption. A second difference between this exposure and those carried out previously was the use of titanium gettering between discharges. In fact, one of the titanium sources was located in fairly close proximity to the samples (∿0.25 m in front of and ∿0.10 m below their position).

Prior to the analysis of the specimens, several interesting visual observations were made of the stainless steel sample holder. Photographs of this assembly, which is used for securing the tips during irradiation, are shown in Fig. 2. The wire specimens (spot-welded to 1.5 mm diameter stainless steel rods) are secured in holes forming a circular pattern at the top of a cylindrical block. A stainless steel cap is placed over the holder with 1.5 mm holes above those specimens to be exposed to the plasma. Fig 2(a) shows the holder with the cap in place; Fig 2(b) with the cap removed.

In photograph (a) of Fig. 2, one can see a slightly discolored region with a series of elongated tracks running along a portion of the outer perimeter of the cap. From the orientation of the holder with respect to the plasma, it is very probable that these marks are arc tracks; however, the occurrence of unipolar arcing at the position of the samples (0.59 m from plasma center) is very surprising.

Upon removal of the cap (Fig. 2b), four pronounced, colored spots of ∿1.6 mm diameter were observed. These spots were found by sputter-Auger analysis to be films (∿1000 Å thick) of titanium and/or oxides of titanium, obviously deposited during the titanium gettering operations in PLT. The absence of a corresponding discoloration of the sample holder cap was at first believed to be due to sputter-removal of

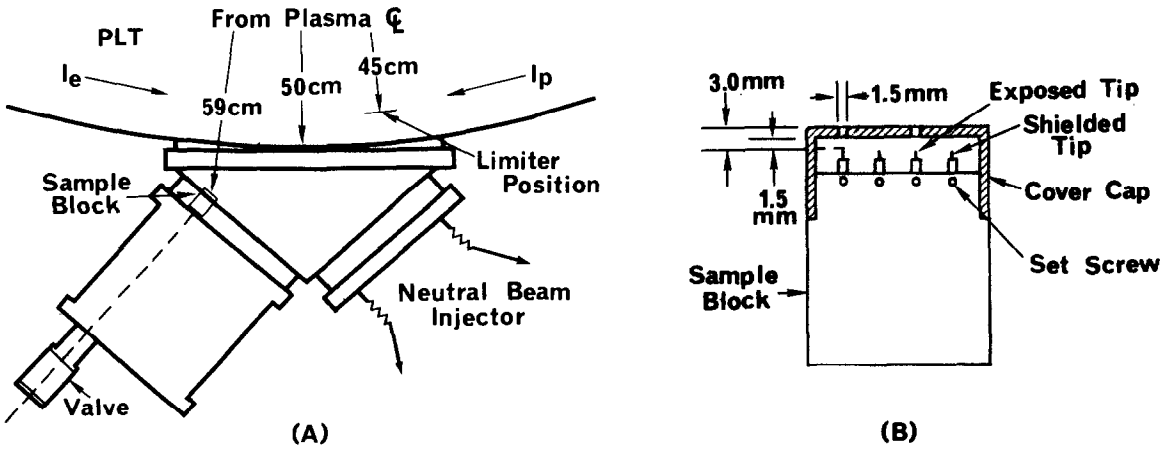

Fig. 1. Schematic drawing showing (a) the position of the sample holder with respect to the tokamak during exposure and (b) the position of the FIM "tips" with respect to the sample holder.

the titanium from the cap during the operational tokamak discharges. However, subsequent Auger analysis of the cap surface indicated that a layer of comparable thickness to that of the spots was also present on the cap. Significantly more carbon was found in the cap film than in the spots, possibly explaining the difference in observed coloration between the cap and the cylindrical block. The only other major species to be identified in the Ti films was oxygen, although traces of S, Cl, Ca, and Na were detected.

The positions of the deposit regions relative to the holes in the cap can be used to define the direction of the titanium particle flux within the holder. This direction along with the specimen tip height above the holder surface then determines whether the titanium should interact (be deposited on) with the specimens. In this instance the geometry indicates that the majority of titanium should just miss the ends of the specimen tips.

Following the observations and analysis of the sample holder, five field-ion specimens were analyzed according to the procedure listed in section 2. Transmission electron micrographs, taken to determine the extent of surface contamination, are displayed in Fig. 3. As in the previous exposures, surface layers were found to reside on the exposed tips (1, 3, and 5); however, for the first time contamination deposits were also observed on the surfaces of the control tips (2 and 4). Since the control specimens did not have line of sight to the plasma during the discharges and hence were not exposed to the impurity flux entering the holder, the presence of these films is quite surprising. This is particularly true in view of previous exposures, in which in one case (set #2) films 500 Å thick were observed on the exposed samples, and yet no deposits were detected on the control samples. Because of these past observations, one is led to conclude time of exposure were responsible for the control specimen contamination and not some consistent artifact of the experiment such as air exposure or TEM analysis.

The thicknesses of the surface films measured from the TEM micrographs of Fig. 3 were all found to be in the range from 50 Å to 60 Å. Dividing the average thickness by the total number of shots (six), one finds the approximately 9 Å/shot of contamination is deposited on the samples. This number is very close to that obtained from set #2, in which ~7 Å/shot was estimated. In light of the discussion in the previous paragraph, however, one must note that during normal PLT operation the contamination may be significantly reduced.

The compositions of the deposited films were determined qualitatively using the Imaging Atom-Probe. Several differences were observed between the composition of the contaminant films on the exposed tips and those on the shielded tips. Most prominent among these differences was the presence of large peaks at $m/n = 6(C^{2+})$, $m/n = 12(C^{+})$, and $m/n = 24(C_2^{+})$ in the mass spectra of the films on the exposed tips, compared to a small abundance of these species in the mass spectra of the control tips. This difference appears to eliminate the TEM as a possible source of the deposited films, since the primary contaminant associated with TEM analysis is carbon and would therefore be detected on all the sample surfaces. Since the resolution of the IAP is not sufficient to separate species with m/n values closer than 0.5 amu, the mass analysis does not allow one to identify the film compositions unambigiously. For example, large peaks at m/n = 16 and m/n = 32 were observed in the spectra of all five specimens. These peaks, which are normally associated with O^{+} and O_2^{+}, can also be associated with Ti^{3+} and TiO^{2+} to within the resolution of the instrument. As a result, the films observed in the TEM on all five tips may

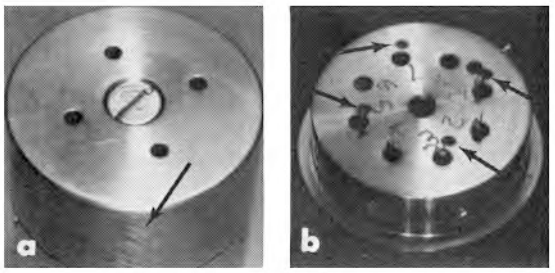

Fig. 2. Photographs of the stainless steel sample holder, (a) with cap in place and (b) with cap removed. Arc tracks are observed on the cap in (a). Small spots of Ti deposits are indicated by the arrows in (b).

most probably resulted from earlier, normal tokamak discharges. Clearly, data from future exposures in which all discharges are representative of normal operation will be required to eliminate such ambiguity.

It is difficult to make direct comparisons between the results of the FIM/IAP experiments and similar experiments using more conventional surface analysis techniques because of the widely varying plasma/sample conditions from exposure to exposure. This difficulty is further compounded in this particular study because of the anomalous plasma discharges which occurred during exposure. For example, carbon samples, which were exposed to discharges in PLT in a similar location and on the same day as the present exposures (but before the disruptions), were examined by RIBS, AES, and SIMS and were found to have quantities of Fe, Cu, and Ta deposited on the surface [8]. Such impurities were not present on the surface

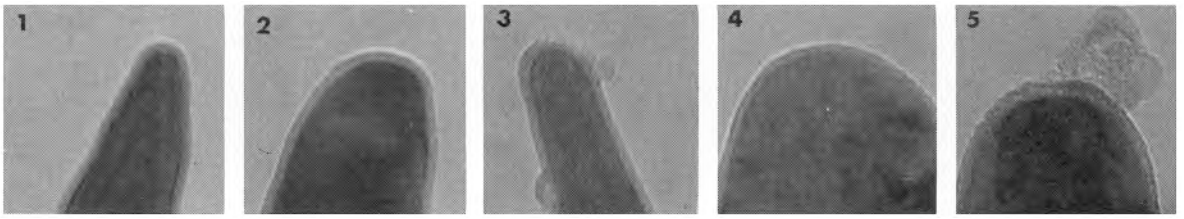

Fig. 3. TEM images of specimens of set #4 after plasma exposure. Contaminant layers of 50-60 Å are observed on all five tips.

of the FIM samples and are most likely associated with the particular local environment of the samples (e.g., sample holder, shield, etc.). Titanium gettering was not used between discharges during the carbon sample exposure and, therefore, no Ti was detected in the analysis. Obviously, carbon deposited on the carbon substrates could not be detected, and thus the macroscopic samples may actually have been covered with a carbon film.

Consistent with previous exposures was the absence of structural damage and implantation of plasma species. Because of space limitations, the field-ion images of the samples before and after exposure are not displayed; however, these images quite clearly indicate that no atomic rearrangement took place as a result of plasma exposure. Imaging Atom-Probe depth profiles into both the stainless steel and tungsten near surface regions, as in all earlier exposures, gave no indication of implanted plasma or impurity species.

4. SUMMARY

The results of the analysis of this most recent sample exposure allow us to make several qualitative statements regarding the plasma-wall interactions in PLT. First, it appears as though substantial amounts of low Z impurities were still being deposited on surfaces exposed to the plasma; even though the primary limiter was not carbon. Comparisons of film thicknesses with earlier exposures, in which carbon limiters were used, however, are difficult because of the occurrence of the disruption. As expected, we found no qualitative differnce between the interaction (deposition, damage, implantation, etc.) of the plasma with stainless steel samples as opposed to tungsten samples. Titanium gettering was found to leave thick coatings (∿1000 Å) on sample holder surfaces in direct line of sight with the Ti sources. Finally, as observed in all earlier exposures, samples exposed at the recessed wall position where these samples were located were not altered by structural damage or implantation effects, even on an atomic scale.

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