

SURFACE CHARACTERIZATION OF TFTR FIRST WALL GRAPHITE TILES USED DURING DT OPERATIONS

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ABSTRACT

Surface characterization studies were performed on graphite tiles used as first wall materials during DT operation of the Tokamak Fusion Test Reactor (TFTR) at the Princeton Plasma Physics Laboratory. These *ex situ* analysis studies revealed a number of interesting and unexpected features. In this work we examined the spatial and (where possible) the depth distribution of impurity species deposited onto the plasma facing surfaces using X-ray Photo-electron Spectroscopy (XPS) and Secondary Ion Mass Spectrometry (SIMS).

This work determined that beyond the predominant species of carbon and oxygen, common impurities included silicon, boron, lithium and sulfur. Oxygen content in the plasma facing tile surfaces ranged from 20 to 50 atomic percent [excluding H-isotopes], clearly indicating an extensive zone of oxidized carbon. By contrast, carbon tile surfaces not exposed to the plasma have surface oxygen contents ranging from 2 to 6 atomic percent. Analytical measurements of the secondary impurities (B, Li, Si, S) levels were on the order of 1-4 atomic percent. (boron and lithium were injected for wall conditioning in TFTR.) The core level binding energies of these impurity species were consistent with the presence of common oxides or hydroxides (e.g., B_xO_y , Li_2O , $LiOH$, Silicates). XPS measurements performed in concert with depth profiling indicated that the tile oxidized zone was significantly deeper than 1 micrometer into the (averaged) surface. Surface analytical results clearly indicate that plasma operations clearly redeposit injected impurities (Li, B) and the depth profiles and distributions of the hydrogen isotopes may be impacted and/or influenced by this deposition process.

An attempt at determining hydrogen isotope concentration distributions was made using positive ion SIMS. Specific regions of some surfaces clearly indicated the presence of $m/z=3$ (HD, T) and $m/z=15$ (CH_3 , CHD, CT). Preliminary data examination using positive ion SIMS examination indicates that these mass markers are substantially higher in the near surface region when compared with spectra recorded deeper in the surface region. The deuterium and tritium concentrations were; however, sufficiently low or compromised by common isobaric interferences that accurate isotopic distributions using SIMS were not

possible. These findings are in agreement with results reported by others. [Morimoto et al, Sun et al, reference 3 Haasz et al]

I. INTRODUCTION

The distribution of deuterium and tritium in plasma facing carbon tiles in TFTR is of fundamental interest to plasma material interactions, tritium removal strategies and the decommissioning of these materials. Many strategies have been suggested and tested for detritiation including large scale laser annealing [Skinner this proceedings], UV laser irradiation [Shu, this proceedings], simple thermal heating, ozone treatments and exposure to other reactive entities designed to release tritium from the near surface region of these surfaces. [Federici et al., Counsel et al] In an effort to understand where tritium may be localized in these complex interfaces we have characterized the near surface region of these materials to address where and what are the likely impurities. Material properties and potential decontamination schemes may be impacted by these impurities and the role of re-deposition of materials and removal of entrained or embedded tritium may ultimately be a complex process in which these impurities may play a significant role. [reference 1 Haasz].

II. RESULTS AND DISCUSSION

Several tile pieces sectioned from graphite and carbon fiber composite (CFC) tiles from the TFTR inner limiter with section lines is shown in Fig. 1.

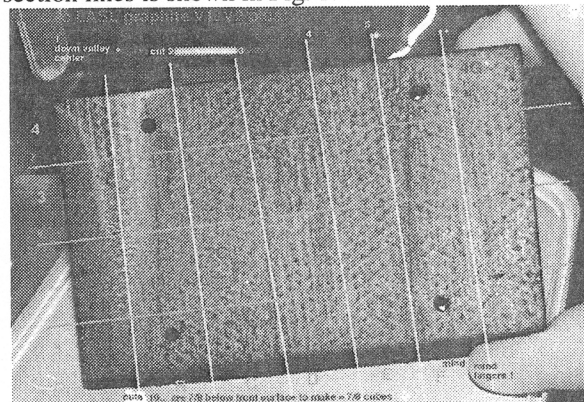


Fig. 1: TFTR carbon tile KC17 plasma facing surface with section lines indicated. Surface analysis results described

below originate from regions as denoted above. Sections labeled A thru G run left to right and sections labeled 1 to 4 from bottom to top in figure.

The sectioned CFC pieces were received with the plasma facing surfaces identified. The CFC carbon tile had approximate plasma facing dimensions of ca. 6 x 10 cm and is shown in Fig. 1. In addition, a variety of samples consisting of deposits (gathered from a poloidal limiter tile and inner limiter tile KB3) possessing irregular shapes were also received. These latter samples were of millimeter dimensions affixed to conductive carbon SEM tape. In most cases multiple spots (or flakes) were analyzed and in general, significant differences in atomic concentrations were not observed. Exceptions existed for samples where very noticeable physical differences were visually apparent on the surfaces. The list of sectioned carbon tile samples included those identified as 1G, 4G, 4F, B4, 2C, 4A, V1 and V2. Samples V1 and V2 came from a graphite tile KC22 not shown in Fig.1. The physical description and significance of examining the V 1 and V2 tiles is described separately later. Crud samples examined included those identified as Crud 1 and 2, (debris collected from a poloidal limiter tile) and tapes W, X, Y, and Z deposits from tile KB3. All samples were affixed to a bayonet style sample mount plate using conductive carbon tape (two examples of this mounting arrangement are shown in Figure 2).

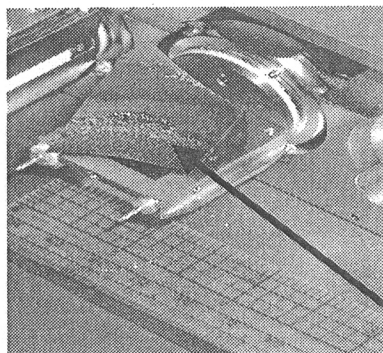


Figure 2: Mounted Specimen G4 (top) and V2 (bottom) on bayonet mount for translation in spectrometer. Note the

extreme concave nature of surface and rough residual deposits clearly identified by arrow on sample V2.

Occasionally the electrical conductivity between the carbon samples through the stage arrangement did not provide for unequivocal determination of ion beam fluences or reproducible SIMS data. In these cases minor surface charging was indicated in preliminary data collection and where possible was mitigated through use of a charge compensation electron beam flood gun

Surface characterization experiments were performed in an ultra-high vacuum (UHV) chamber with base pressures of less than 3×10^{-10} Torr that was obtained by a combination of turbo-molecular, vac-ion, and titanium sublimation pumps. The UHV chamber contains instrumentation for x-ray photoelectron spectroscopy (XPS), thermal desorption mass spectroscopy (TDMS), secondary-ion mass spectroscopy (SIMS), and sputtered neutrals mass spectroscopy (SNMS) measurements. The XPS data were taken using a non-monochromatic MgK α source ($h\nu = 1253.6$ eV) and the photo-emitted electrons were detected with a hemispherical analyzer. Analyzed areas were typically 0.8 mm square. Pass energies used were typically 29 eV and incident x-ray power was 200 W. All spectra reported herein are given in terms of binding energy (B.E.). The SIMS data were acquired using a differentially pumped 5 keV Xe $^+$ ion beam passed through a Wien filter rastered over a 2x2 mm area. For static SIMS (SSIMS) measurements, ion beam doses were kept below a level necessary for noticeable disruption of the surface concentrations. Depth profiling was performed using significantly higher ion beam fluences in separate experiments with a sputter removal rate estimated from the thin oxide layers on uranium to be ~ 30 Å/min. Positive ions were normally collected although provision for negative ions can also be followed using this quadrupole mass analyzer.

IIA. ANALYSIS OF CRUD SAMPLES

Crud samples were mounted on conductive carbon SEM tape and analyzed using both XPS and SIMS positive techniques. Representative XPS survey spectra over a small flake with a predominately yellow-white color is shown in Fig. 3. Transitions attributable to O1s, C1s, Si2s, Si2p are readily apparent. Although not as apparent, less intense transitions arising from Li1s and B1s transitions are also seen in multiplex spectra. More detailed spectra were recorded over specific transitions of interest and confirmed the existence of oxidized forms of Si, S, B, and Li. For this specific sample the elemental analysis routines indicate a nominal composition as shown in Table 1 below. Similar analysis of other flakes was also performed but the data are not shown for brevity.

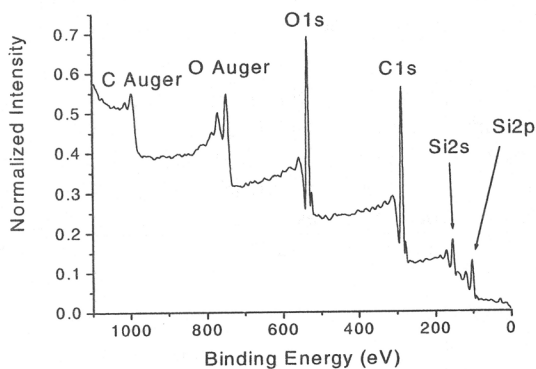


Fig. 3 Survey Spectrum from sample Crud 1.

Table 1 Atomic Concentrations of XPS Detected Species from Crud Flake

Sample	Li	B	C	O	Si	S
Crud 1	7.3	7.0	45.6	31.3	6.7	2.1
Crud W	-	2.9	70.1	26.4	0.6	-
Crud X	-	0.9	74.3	23.9	0.8	-
Crud Y	-	3.8	66.5	28.7	0.9	-
Crud Z	-	6.1	55.1	35.5	3.3	-

SSIMS spectra were also recorded from several flakes and a representative spectrum is shown in Fig. 4. Note the prominent lines arising in the mass spectrum arising from Li, B, C, Na, Si, K, Cr, Fe and many other elemental components present on the mounted tape.

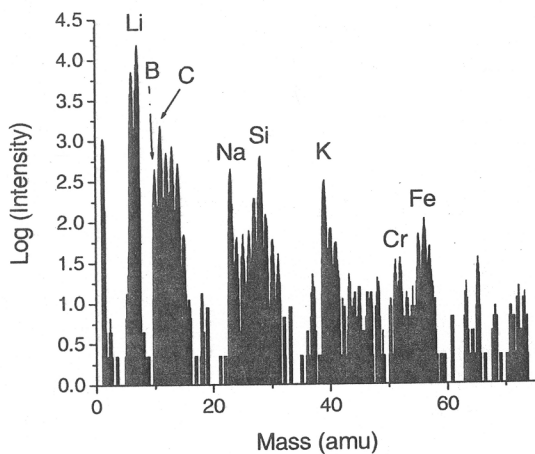


Fig. 4 SSIMS positive ion spectrum of TFTR sample Crud 1.

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The sectioned regular carbon tile samples examined using XPS surfaces included those identified as 1G, 4F, B4, G4, C2, 4A and V1, V2. Sample V1 and V2 originated from a graphite tile with a diagnostic penetration and were of interest because of unusual white deposits (in contrast to black carbon surface) and surface flaking. The extent of flaking and possible detrimental effects following scale up of operation is a serious concern in next step fusion machines. [reference 8 C.H. Skinner] Mixed material effects are a prime concern. XPS data provides quantification of elemental constituents and the chemical state of the species detected. Aside from H (not detected using XPS) the elements detected by this technique can be quantified by conventional methods following appropriate background subtraction and correction procedures. In Table 2 atomic concentrations are provided for specific samples. Elements detected above fractions of an atomic percent are listed. Note that for many samples several spots located on one cut sample have been analyzed. If surface morphology and/or physical features were apparent the table entry is denoted by the feature description (e.g., rough versus smooth). A critical comparison to the non-plasma facing concentrations can be obtained by comparison to data for the last two entries in the table (4F.cut edge1 and 4F.cut edge2).

Table 2 Atomic Concentrations of XPS Detected Species from Tile Samples.

Sample	B	C	O	Si	S	Au
G1	4.1	51.2	39.5	4.5	0.6	-
4F.1	5.2	49.7	40.4	4.1	0.5	-
4F.2	4.9	48.5	40.6	5.4	0.6	-
4B.1	3.7	51.6	38.8	5.9	-	-
4B.2	2.9	51.6	39.2	5.9	0.9	-
2C.1	3.1	50.5	39.1	7.2	-	-
2C.2	3.4	48.4	40.0	8.1	-	-
4A.1	4.8	31.0	52.6	11.6	-	-
4A.2	4.4	32.1	50.2	13.3	-	-
V1.1	-	76.5	21.1	2.4	-	-
V1.2	4.0	73.4	22.3	0.3	-	-
V1.3	-	94.7	3.8	0.2	1.3	-
cut edge						
V2 rough	3.4	30.6	50.4	13.9	1.7	-
V2 smooth	8.1	48.7	42.1	1.2	-	-

Non Plasma Facing Surface (Reference Concentrations)

4F.cut edge1	-	94.1	5.3	-	-	0.6
4F.cut edge2	-	91.1	6.8	-	-	1.4

The surfaces identified as 'cut edge' were not exposed to the plasma and clearly indicate that the extent of oxidation is significantly lower than that observed from the plasma facing surfaces. Also of note is the observation of Au in this surface region. This was observed in several

spots and was traced to physical abrasion of a sample mounting or cutting arrangement resulting in metallic Au spread across the edge of the carbon tile.

SSIMS data recorded from some of the selected tile samples indicated the presence of many of the same constituents as indicated in the TFTR Crud flake sample shown above. Not all samples have been examined, and preliminary depth profiling experiments clearly indicated that where examined the depth of the analysis zone (up to 0.5 micrometers) the constituents did not change significantly as judged from invariant ion intensity versus time profiles. Further work is continuing with these samples with the intent of hopefully being able to depth profile tritium and deuterium concentrations in the near surface region. [see also reference 9 R.-D. Penzhorn]

III. CONCLUSIONS

In examining data from both the SSIMS and XPS data taken from the large scale sectioned tiles shown in Fig. 1 and shown in Table 2 it is apparent that significant large-scale oxidation of the carbon face has occurred following the extensive operational history of the TFTR. We note that air ventilation at 150 C was used after the end of plasma operations to remove tritium. [reference 10 D. Mueller] The O content in the plasma facing portions of the carbon tiles ranges from 20-50% and compares with the references values of 2-8% on cut (non plasma facing surfaces). In addition to the high O content, many other secondary impurities (Si, S, Li, B) are observed in the plasma-facing surface and clearly indicate redeposition of either intentionally added plasma components (e.g., Li, B₂H₆) or spurious impurities that may arise from cleaning or other undefined operations occurring throughout the history of TFTR. Also of particular concern is the occasional observation of elements commonly found in steel (e.g., Cr, Fe, Ni) presumably redistributed during plasma disruptions. Operational experience has led to the observation of deposition layers in specific regions of the TFTR of approximately 100 micrometers thickness. However in other regions erosion of the carbon tiles is indicated from examination of the textured nature of the tiles. Adhesion of deposits from plasma deposition is of concern and these results indicate that the oxidized Si, B and Li components in the outward facing tiles may induce strain in the growing deposition film.

In comparing the dispersion of impurities as a function of position across the tile face it is evident that the occurrence of Si deposits (as silicates) is more pronounced on tiles in the lower left hand figure (Tile 4A). The source of the silicon is believed to be a residue from sanding of tile surfaces before plasma operations. The approximately 10% increase in oxygen and 4-5 % increase in Si atomic concentrations in this region over adjacent portions of the

tile is unexplained at this time. For samples V1 and V2 the rough deposits indicated in Figure 2 clearly have a significantly higher Si content and suggests that Si (as a silicate species) may serve as a chemical marker for scale nucleation and deposition of rough deposits. It is interesting to note that a fairly high surface concentration of B is observed in the smooth portion of this tile.

Atomic concentrations of elements observed in the Crud flakes clearly indicated again the presence of silicates along with oxidized lithium and boron. The white to yellow color frequently observed from this particulate matter is consistent with these observations. Furthermore, a significant variability is observed in these materials but this is consistent with the sample mounting methodology and analytical measurement for these loose materials mounted on conductive tape.

The SIMS data (both SSIMS and depth profiling data) clearly observe chemical species that could be attributed to tritiated surface components. However, the isobaric distinction between HD and T over either the mass region of 1-3 and the CH_x region makes the deconvolution of these entities is not straightforward. The T/D fueling ratio of TFTR was 0.03 so the tritium component is likely to be very small. Regardless of these limitations the depth distribution of the sum total of the hydrogen containing species as judged from the CH_x region was uniform to depths approaching micrometer dimensions. Given the intrinsic rough nature of the tile surfaces qualitative estimates of deep entrainment of tritium containing component in real parts is probably best accomplished using a combination of techniques in concert with severe surface removal methodologies such as high fluence ion milling or controlled physical abrasion as mentioned in reference 9.

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REFERENCES

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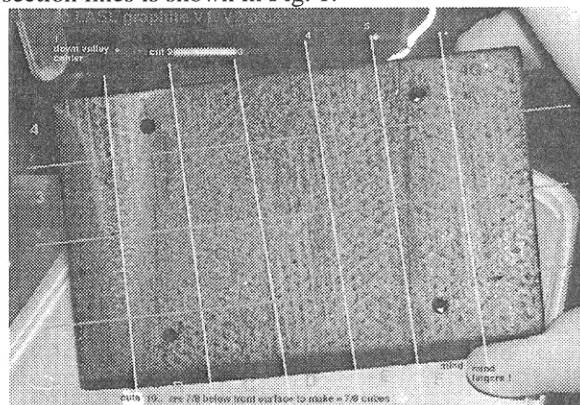


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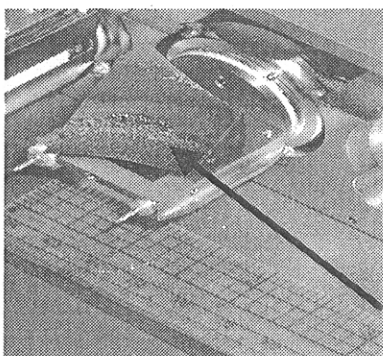
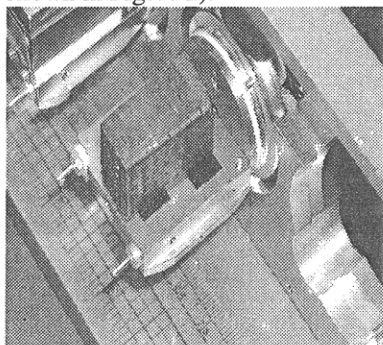


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extreme concave nature of surface and rough residual deposits clearly identified by arrow on sample V2.

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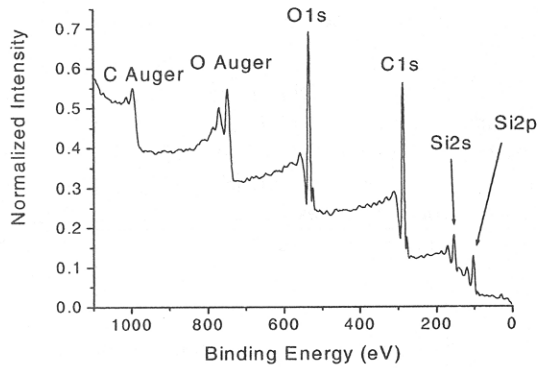


Fig. 3 Survey Spectrum from sample Crud 1.

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Sample	Li	B	C	O	Si	S
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Crud X	-	0.9	74.3	23.9	0.8	-
Crud Y	-	3.8	66.5	28.7	0.9	-
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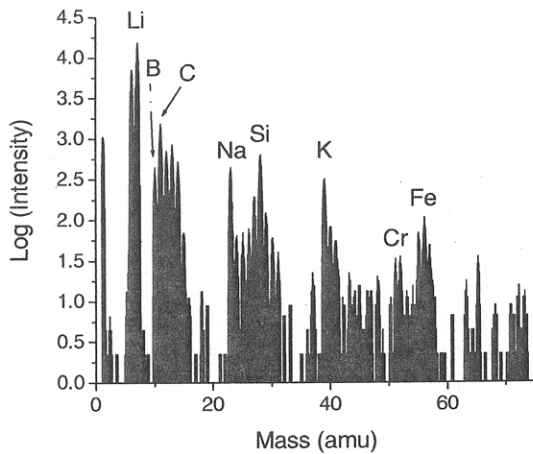


Fig. 4 SSIMS positive ion spectrum of TFTR sample Crud 1.

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Sample	B	C	O	Si	S	Au
G1	4.1	51.2	39.5	4.5	0.6	-
4F.1	5.2	49.7	40.4	4.1	0.5	-
4F.2	4.9	48.5	40.6	5.4	0.6	-
4B.1	3.7	51.6	38.8	5.9	-	-
4B.2	2.9	51.6	39.2	5.9	0.9	-
2C.1	3.1	50.5	39.1	7.2	-	-
2C.2	3.4	48.4	40.0	8.1	-	-
4A.1	4.8	31.0	52.6	11.6	-	-
4A.2	4.4	32.1	50.2	13.3	-	-
V1.1	-	76.5	21.1	2.4	-	-
V1.2	4.0	73.4	22.3	0.3	-	-
V1.3	-	94.7	3.8	0.2	1.3	-
cut edge						
V2 rough	3.4	30.6	50.4	13.9	1.7	-
V2 smooth	8.1	48.7	42.1	1.2	-	-

Non Plasma Facing Surface (Reference Concentrations)

4F.cut edge1	-	94.1	5.3	-	-	0.6
4F.cut edge2	-	91.1	6.8	-	-	1.4

The surfaces identified as 'cut edge' were not exposed to the plasma and clearly indicate that the extent of oxidation is significantly lower than that observed from the plasma facing surfaces. Also of note is the observation of Au in this surface region. This was observed in several

spots and was traced to physical abrasion of a sample mounting or cutting arrangement resulting in metallic Au spread across the edge of the carbon tile.

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In comparing the dispersion of impurities as a function of position across the tile face it is evident that the occurrence of Si deposits (as silicates) is more pronounced on tiles in the lower left hand figure (Tile 4A). The source of the silicon is believed to be a residue from sanding of tile surfaces before plasma operations. The approximately 10% increase in oxygen and 4-5 % increase in Si atomic concentrations in this region over adjacent portions of the

tile is unexplained at this time. For samples V1 and V2 the rough deposits indicated in Figure 2 clearly have a significantly higher Si content and suggests that Si (as a silicate species) may serve as a chemical marker for scale nucleation and deposition of rough deposits. It is interesting to note that a fairly high surface concentration of B is observed in the smooth portion of this tile.

Atomic concentrations of elements observed in the Crud flakes clearly indicated again the presence of silicates along with oxidized lithium and boron. The white to yellow color frequently observed from this particulate matter is consistent with these observations. Furthermore, a significant variability is observed in these materials but this is consistent with the sample mounting methodology and analytical measurement for these loose materials mounted on conductive tape.

The SIMS data (both SSIMS and depth profiling data) clearly observe chemical species that could be attributed to tritiated surface components. However, the isobaric distinction between HD and T over either the mass region of 1-3 and the CH_x region makes the deconvolution of these entities is not straightforward. The T/D fueling ratio of TFTR was 0.03 so the tritium component is likely to be very small. Regardless of these limitations the depth distribution of the sum total of the hydrogen containing species as judged from the CH_x region was uniform to depths approaching micrometer dimensions. Given the intrinsic rough nature of the tile surfaces qualitative estimates of deep entrainment of tritium containing component in real parts is probably best accomplished using a combination of techniques in concert with severe surface removal methodologies such as high fluence ion milling or controlled physical abrasion as mentioned in reference 9.

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REFERENCES