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LIMITER IN ALCATOR C**

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**Abstract:** SiC-coated graphite limiter material was eroded by a plasma during the plasma edge experiments in Alcator C, and redeposited on the molybdenum surface of the Janus probe. Surface morphology, composition and structure of the deposit was examined after a total exposure time of  $\sim 1000$  seconds. The redeposited material typically showed a "layered" morphology. AES depth profiles revealed increased oxygen content in the redeposited material and an apparent diffusion of Si and C at the deposit-molybdenum interface. Two clearly distinguishable chemical states of silicon in the deposit, as observed by XPS, suggest that plasma ionized polycrystalline SiC is being redeposited as a mixture of SiC and SiO<sub>2</sub>. Preliminary examination of the near surface regions by SIMS indicated (a) the presence of deuterium in the deposit, (b) the presence of molybdenum in the first few layers of the deposit, (c) formation by the plasma of multiple states of Si<sub>x</sub>O<sub>x</sub>, Si<sub>x</sub>C<sub>x</sub>, and Si<sub>x</sub>C<sub>x</sub>O<sub>x</sub> which do not appear in the spectrum of the unexposed SiC.

## 1. Introduction

A study of redeposition of the SiC-coated graphite limiter is an extension of the edge plasma experiments which had been performed in Alcator C with a predecessor of the Janus probe. A detailed description of the Janus probe is given in reference [1]. Briefly, it is a multiple function edge probe used to diagnose the Alcator C limiter shadow plasma whose components include two sets of identical diagnostics, each set facing a different direction. As shown schematically in Fig. 1, included with each set of diagnostics are a retarding-field energy analyzer (RFEA), Langmuir probe, and calorimeter. Janus is constructed to make measurements both parallel and antiparallel to a magnetic field. It can withstand high heat fluxes and it can be scanned perpendicular to the magnetic field in the limiter shadow. The RFEA can alternatively sample both the ion and the electron parallel energy distribution functions during a tokamak discharge. The key element of the probe is the RFEA which consists of five components: a knife-edge slit, three double side mesh electrodes and a collector. The components are separated by the  $\text{Al}_2\text{O}_3$  insulators. The slit width is  $30\ \mu\text{m}$  which equals approximately three Debye lengths. The mesh grids are used to repel electrons or ions. The collector voltage is swept to measure the parallel energy of each species of the plasma from which their temperatures are deduced. The incident heat flux and metal surface temperature are measured by the calorimeter. The Langmuir probe serves as a calibration check.

The Janus probe was developed in order to make correlated studies of the material surface effects and plasma parameters. However, this probe has been operated in Alcator C for a long period of time, under wide range of plasma conditions. The probe has been scanned radially in the scrape-off layer. It has been as close as the plasma edge and it was exposed to high particle and heat fluxes. The first grid has been destroyed. The average temperature of the probe at the slit is about 1000 K. The temperature on the probe edge close to plasma was as high as 2000 K and on the far edge from the plasma is about 500 K. The average incident heat flux on the slit is about  $1000\ \text{W}/\text{cm}^2$ .

In this paper, we are reporting properties (i.e., morphology, composition and structure) of the redeposited SiC coating on the molybdenum surface of the Janus probe, from

the erosion of a SiC-coated graphite limiter. The experimental set-up is shown schematically in Fig. 2. The Janus probe is approximately 0.5 cm behind the limiter and its electron side flux tube has a limiter connection length that is twice as long as the ion side. SiC-coated graphite limiter material was eroded by plasma during the plasma edge experiments in Alcator C and then redeposited, in part, on the surface of the Janus probe. The SiC deposit was examined after a total exposure time of approximately 1000 seconds. Typical plasma conditions during redeposition are shown in Table 1.

**TABLE 1**  
Typical Alcator C Plasma Conditions

<b>Alcator C Parameters</b>	<b>Standard Values</b>	<b>Range of Values</b>
$R_{major}$	64 cm	57 – 71 cm
$r_{minor}$	16.5 cm	10 – 16.5 cm
$B_{toroidal}$	8 tesla	5.5 – 13 tesla
$I_p$	400 kA	100 – 700 kA
$n_e(central)$	$2 \times 10^{14} \text{ cm}^{-3}$	$0.1 - 20 \times 10^{14} \text{ cm}^{-3}$
$T_e$	1500 eV	1000 – 3000 eV
$T_i$	1100 eV	500 – 2000 eV

The results obtained in this study revealed new and interesting features of material behavior in the hot and dense plasma. A detailed correlation of the properties of the SiC deposit and the edge plasma parameters is not possible at this stage as the Janus probe has been operated under a wide range of plasma conditions. Available data will allow, however, a design of more selected experiments to study plasma-material interaction. The long term aim of this study is to provide new material and design options for fusion reactor components exposed to high heat and particle fluxes.

## 2. Experimental Procedure

A SiC coating redeposited on the molybdenum surface of the Janus probe during the plasma edge experiments was examined by scanning electron microscopy (SEM) in order to reveal its surface morphology. Surface and near-surface composition and structure of the redeposited SiC-coated graphite limiter was studied by using secondary ion mass spectrometry (SIMS) and x-ray photoelectron spectroscopy (XPS) techniques. Auger electron spectroscopy (AES) depth profiles through the redeposited material were also determined. Furthermore, an attempt has been made to examine the structure and composition of the SiC deposit-molybdenum interface, and the nature of irradiation damage in molybdenum, by transmission electron microscopy (TEM) and scanning transmission electron microscopy (STEM). Properties of the redeposited SiC coating (i.e., its surface morphology, composition and structure) were compared with those of the unexposed SiC-coated graphite limiter.

## 3. Results and Discussion

### a) SEM

Figure 3 is a SEM micrograph of the unexposed SiC-coated graphite limiter prepared by chemical vapor deposition (CVD) at the Sandia National Laboratory. Its microstructure consists of equiaxed grains, approximately  $100\mu\text{m}$  in diameter (Fig. 3a). Surface morphology of the unexposed SiC grains at a high magnification is shown in Fig. 3b. It changes significantly after exposure to the Alcator C plasma. It appears that erosion is enhanced along the SiC grain boundaries (Fig. 4a). Similar observations were made by other investigators [2]. A magnified view of the SiC surface after erosion is shown in Fig. 4b. It is important to note that the erosion of the SiC-coated graphite limiter by plasma was not as uniform as it may appear in Fig. 4b. Some isolated areas of the limiter showed an extreme erosion, with the SiC coating completely eroded away; in adjacent areas, SiC was typically covered with a thick layer of carbon.

Surface morphology of the SiC-coated graphite limiter redeposited on the molybdenum substrate is shown in Fig. 5. "Layered" morphology which is obvious in Fig. 5 when compared with the polycrystalline unexposed SiC may be due to the pulsed nature of the plasma and chipping during the redeposition process. Redeposited material on the ion side (Fig. 5a) and electron side (Fig. 5b) showed a similar surface morphology. Furthermore, there was no apparent difference in the surface morphology of the deposit as a function of distance from the plasma.

#### **b) AES depth profiles**

Auger analysis for this study was performed on a Perkin Elmer Model 590 digital scanning auger microprobe. The depth profiles represented in Fig. 6 were taken using a 4 keV Ar<sub>+</sub> ion beam at 400 μA/cm<sup>2</sup>. The depth profiles in Fig. 6 are displayed in atomic concentration. This mode of operation takes into account various instrument parameters and equalizes them among different graphs. Because the sensitivity factors used to convert data to atomic concentration are calculated for pure elements and not for compounds, the % concentration for some compounds such as SiC does not appear in the proper stoichiometry. The graph of the SiC-coated graphite limiter material is provided to indicate how the instrument "sees" pure SiC.

AES survey scans on the sputter-cleaned surface of the redeposited SiC coating revealed the presence of silicon, carbon, oxygen and molybdenum. The ion and the electron side, as well as the areas near to or far from the plasma, showed similar AES scans. Based on this information, AES depth profiles of Si, C, O, and Mo were determined and they were compared with those of the unexposed SiC-coated graphite limiter. As shown in Fig. 6, the Si/C ratio remains relatively constant with increasing sputtering time (i.e., depth below the surface), suggesting a uniform composition of the unexposed SiC (apart from the surface enrichment in oxygen). AES depth profile of the SiC-coated graphite limiter redeposited on the ion side, when compared with the unexposed SiC, suggests an increase of the oxygen content in the deposit. Si and C concentrations remain constant over a certain depth below the surface and then gradually decrease with increasing depth, and

the molybdenum concentration increases simultaneously. These concentration curves are indicative of the diffusional processes taking place during redeposition. STEM studies are in progress to confirm AES observations. AES depth profiles on the electron side appear to be similar to those on the ion side, apart from the extended depth below the surface where the Si and C concentrations remained constant due to somewhat thicker redeposited layer (thickness of the SiC deposit has been measured by SEM).

The AES depth profiles in Fig. 6 represent our preliminary data and more detailed work is needed to properly evaluate redeposition processes. These profiles are based on a single measurement, the location of the measurement being randomly selected. For the results to be truly representative, multiple measurements are needed. As pointed out in the introduction, there is also uncertainty in the temperature distribution as the Janus probe has been operated under a wide range of plasma conditions. To evaluate the extent of the diffusional processes taking place during redeposition, it would be useful to analyze the composition of the SiC deposit after a single plasma discharge and to compare it with that of the multilayered deposit in the present study. Based on the average deposit thickness of 2 to 3 microns and a given total exposure time, the average thickness of the deposit of a single plasma discharge is approximately one nanometer. The available high resolution AES and SIMS techniques should allow the analysis of the interface at this level. A good test control would be also to profile a thin film of SiC on molybdenum.

### c) XPS

XPS studies were performed on the unexposed and redeposited SiC-coated graphite limiter samples using a Perkin Elmer Model 548 digital XPS spectrometer with  $MgK\alpha$  x-rays at a pass energy of 25 eV. Spectral peaks were fitted and where necessary curve resolved in order to obtain chemical shift positions, as well as relative peak areas. Results are displayed histographically (Fig. 7) as peak area with respect to binding energy.

For comparison purposes, spectra of standard materials such as C (graphite), Si metal, single crystal SiC and SiO<sub>2</sub> were obtained under the same conditions. In order to obtain the listed value of SiO<sub>2</sub>, the process of Biased Referencing was used to compensate for

the charge buildup on insulating material [3]. No charge effects were exhibited on other samples. Energy positions of these standards are displayed in Fig. 7 as arrows under the graphs.

The two clearly distinguishable chemical states of silicon in the redeposited material suggests that plasma ionized polycrystalline SiC is being redeposited as a mixture of SiC and SiO<sub>x</sub>.

Variation in energy position between the RE and RI positions in these graphs is small and probably attributable to error in curve resolution. The difference between redeposited samples (RE, RI) and the unexposed sample (U) is also small but significant since no appreciable surface charge can be demonstrated on these surfaces and might be attributed to the change in surface morphology (see Figs. 3 and 5).

Further analysis of the deposition area with respect to the silicon chemical states suggests on first observation that under relatively the same analysis conditions there is a greater incidence of oxygen related compounds on the electron side of the probe (RE), whereas the ratio of oxides to carbon related compounds on the ion side is more nearly equal.

It is additionally of interest to note the exact correlation of the unexposed (U) SiC and standard SiC carbon peaks when compared with the silicon peaks of the same samples, suggesting an effect from nearest neighbor dispositions within the two structures, i.e., polycrystalline and single (standard SiC) crystal.

It should be emphasized that these data are preliminary results and clearly suggest the need for further observation of these materials under various redeposition conditions.

#### d) SIMS

SIMS spectra for the unexposed and redeposited SiC-coated graphite limiter samples are displayed in Fig. 8. 100 amu survey spectra of the near surface were obtained for the ion and electron sides and for the unexposed SiC using a Perkin Elmer Model 06-600 SIMS analyzer and sputtering the surface with Ar<sub>+</sub> ions. While these spectra represent



preliminary survey information of only the near surface regions, comparison of the re-deposited samples with the unexposed (starting) material which was analyzed under the same conditions, demonstrates several salient features.

The presence of molybdenum peaks (90+ series) in the first few layers of the re-deposited material suggests that there is a mixing of sputtered probe material with re-deposited limiter material in the plasma.

Since the redeposition also took place in a deuterium plasma, the strong peak at 2 amu which is not present in the unexposed SiC sample, suggests the presence of deuterium within the matrix of the redeposited SiC material. The retention and thermal release of deuterium in SiC has been studied previously [4].

The complex spectra of the redeposited samples in Fig. 8 suggest the formation by the plasma of multiple states of  $\text{Si}_x\text{O}_x$ ,  $\text{Si}_x\text{C}_x$ , and  $\text{Si}_x\text{C}_x\text{O}_x$ , which do not appear in the spectra of unexposed material and therefore cannot be considered simply an artifact of the analytical technique.

#### 4. Summary

SiC-coated graphite limiter was eroded by a plasma during the plasma edge experiments in Alcator C, and redeposited on the molybdenum surface of the Janus probe. The redeposited material typically showed a "layered" morphology. AES depth profiles revealed increased oxygen content in the redeposited material and an apparent diffusion of Si and C at the deposit-molybdenum interface. Two clearly distinguishable chemical states of silicon in the deposit, as observed by XPS, suggest that plasma ionized polycrystalline SiC is being redeposited as a mixture of SiC and  $\text{SiO}_x$ . Preliminary examination of the near surface regions by SIMS indicated (a) the presence of deuterium in the deposit, (b) the presence of molybdenum in the first few layers of the deposit, (c) formation by the plasma of multiple states of  $\text{Si}_x\text{O}_x$ ,  $\text{Si}_x\text{C}_x$ , and  $\text{Si}_x\text{C}_x\text{O}_x$  which do not appear in the spectrum of the unexposed SiC.

The results obtained in this study revealed new and interesting features of material behavior in the hot and dense plasma. A detailed correlation of the properties of the SiC deposit and the edge plasma parameters is not possible at this stage, however, as the Janus probe has been operated under a wide range of plasma conditions.

### **Acknowledgements**

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### **References**

- [1] A. S. Wan, T. F. Yang, B. Lipschultz, B. LaBombard, MIT Plasma Fusion Center Report No. PFC/JA-85-44, to be published in Review of Scientific Instruments.
- [2] Y. Gotoh, H. Hoven, K. Koizlik, J. Linke, E. Wallura, M. Kugel and M. Ulrickson, Proc. 13th Symp. Fusion Tech., Varese, Sept. 24-28, 1984, p. 1247.
- [3] W. L. Landis and J. R. Martin, J. Vac. Sci. Tech., A2 (2), (1984) p. 1108.
- [4] S. Nagata, S. Yamaguchi, Y. Fujino, M. Hirabayashi and K. Kamada, J. Nucl. Mater., 128 and 129 (1984) p. 760.

## Figure Captions

- Fig. 1: 3-D view of a set of diagnostics on a single side of the Janus probe.
- Fig. 2: Janus experimental set-up. The Janus probe is situated between two full poloidal ring limiters with shorter connection length on the ion side (as defined by  $I_p$ ).
- Fig. 3: SEM micrographs showing a polycrystalline structure (A) and surface morphology (B) of the unexposed SiC-coated graphite limiter.
- Fig. 4: SEM micrographs of the SiC-coated graphite limiter exposed to Alcator C plasma; a low magnification view of the SiC erosion (A); surface morphology of the eroded SiC (B).
- Fig. 5: SEM micrographs showing the SiC-coated graphite limiter redeposited on the molybdenum surface of the Janus probe; ion side (A); electron side (B).
- Fig. 6: AES depth profiles; unexposed SiC-coated graphite limiter (top); SiC-coated graphite limiter redeposited on the ion side (middle); SiC-coated graphite limiter redeposited on the electron side (bottom).
- Fig. 7: XPS of the unexposed and of the redeposited SiC-coated graphite limiter. Carbon chemical states (top); silicon chemical states (bottom).
- Fig. 8: Positive ion mass spectrum; unexposed SiC-coated graphite limiter (top); SiC-coated graphite limiter redeposited on the ion side (middle); SiC-coated graphite limiter redeposited on the electron side (bottom).

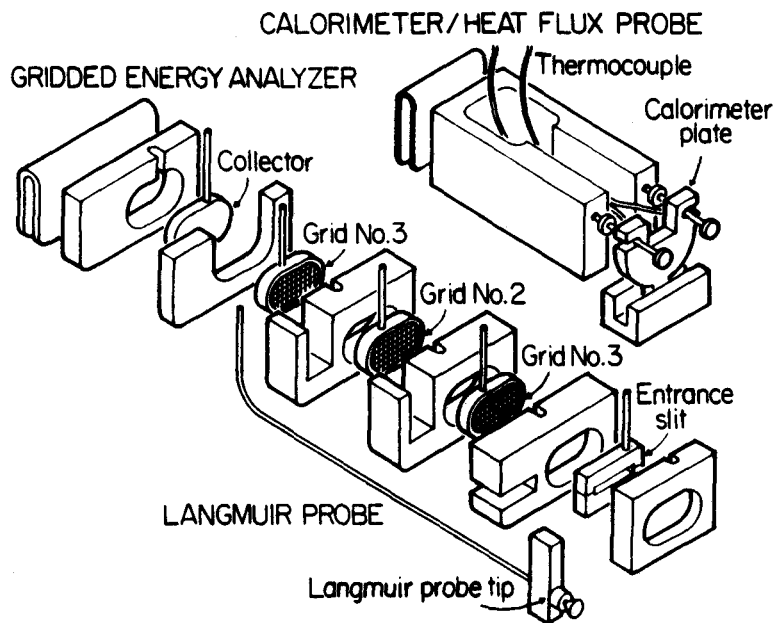


Fig. 1: 3-D view of a set of diagnostics on a single side of the Janus probe.

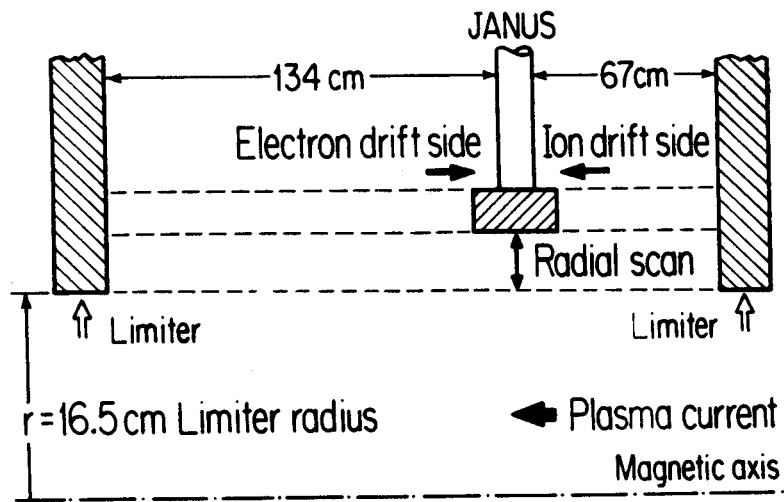
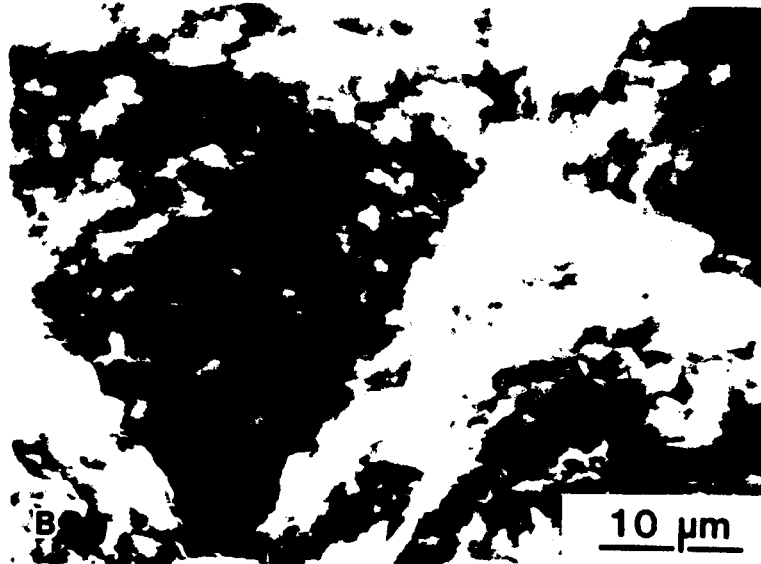


Fig. 2: Janus experimental set-up. The Janus probe is situated between two full poloidal ring limiters with shorter connection length on the ion side (as defined by  $I_p$ ).



**Fig. 3: SEM micrographs showing a polycrystalline structure (A) and surface morphology (B) of the unexposed SiC-coated graphite limiter.**

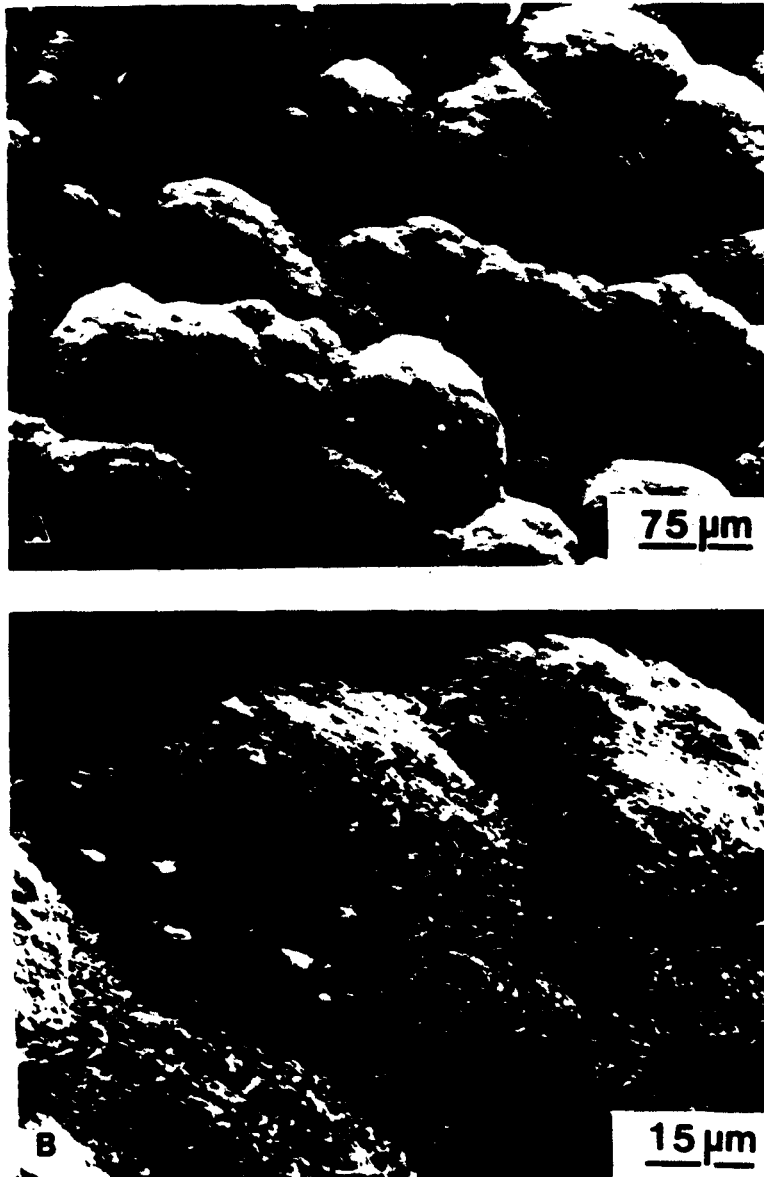


Fig. 4: SEM micrographs of the SiC-coated graphite limiter exposed to Alcator C plasma; a low magnification view of the SiC erosion (A); surface morphology of the eroded SiC (B).

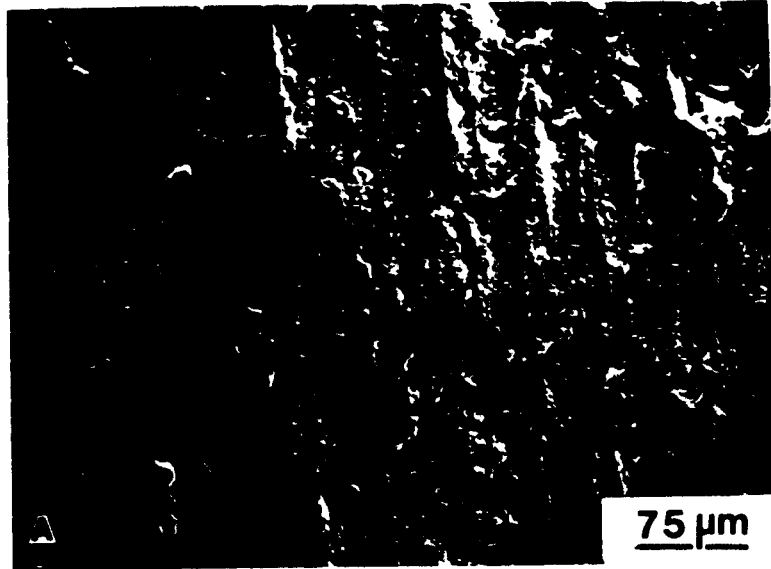


Fig. 5: SEM micrographs showing the SiC-coated graphite limiter redeposited on the molybdenum surface of Janus probe; ion side (A); electron side (B).



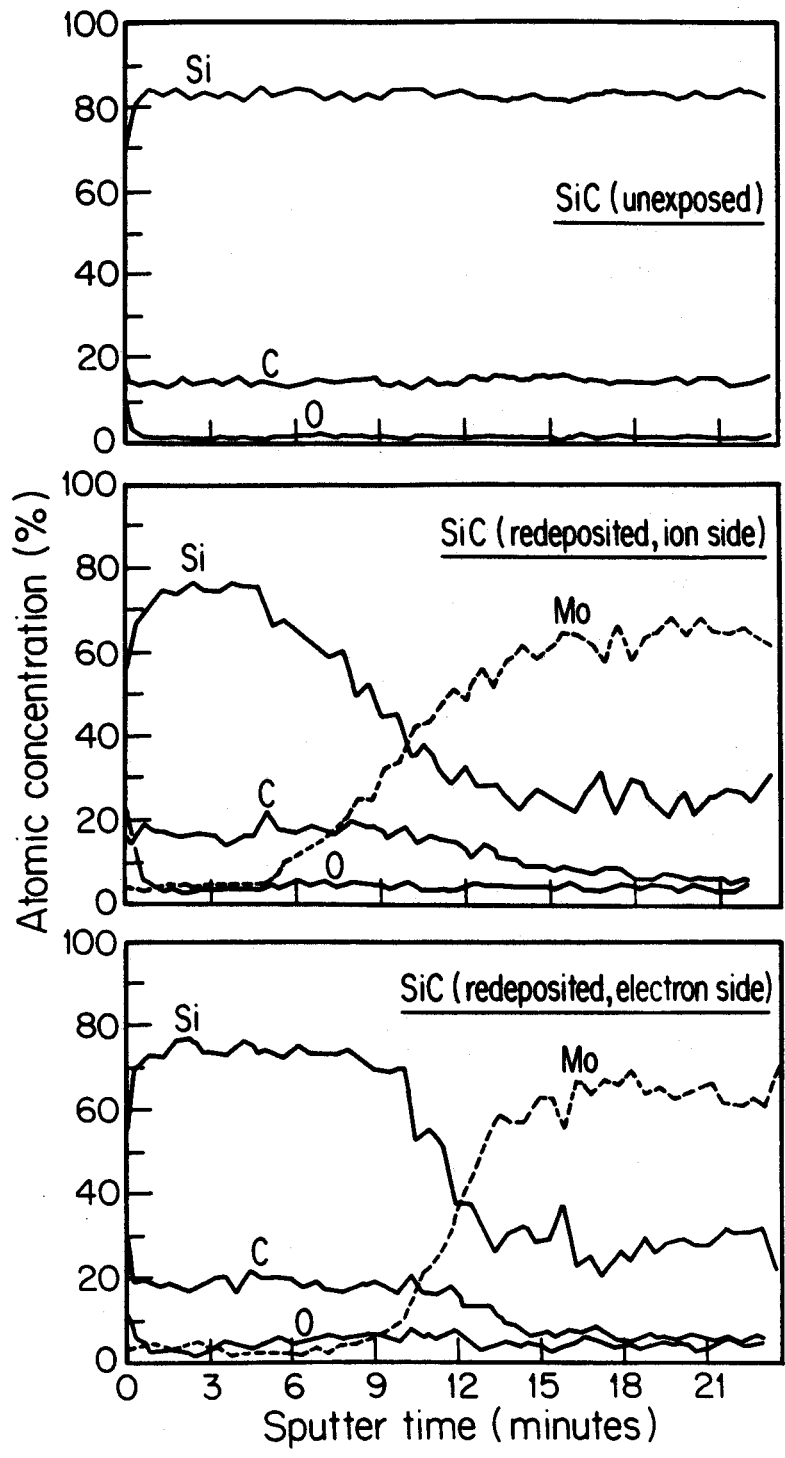


Fig. 6: AES depth profiles; unexposed SiC-coated graphite limiter (top); SiC-coated graphite limiter redeposited on the ion side (middle); SiC-coated graphite limiter redeposited on the electron side (bottom).

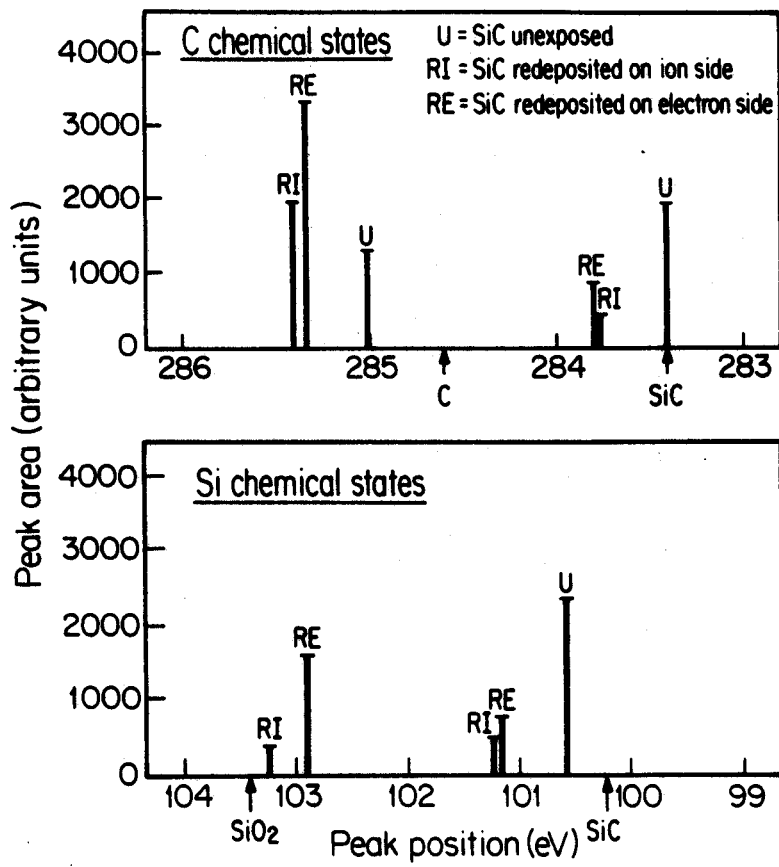


Fig. 7: XPS of the unexposed and of the redeposited SiC-coated graphite limiter. Carbon chemical states (top); silicon chemical states (bottom).

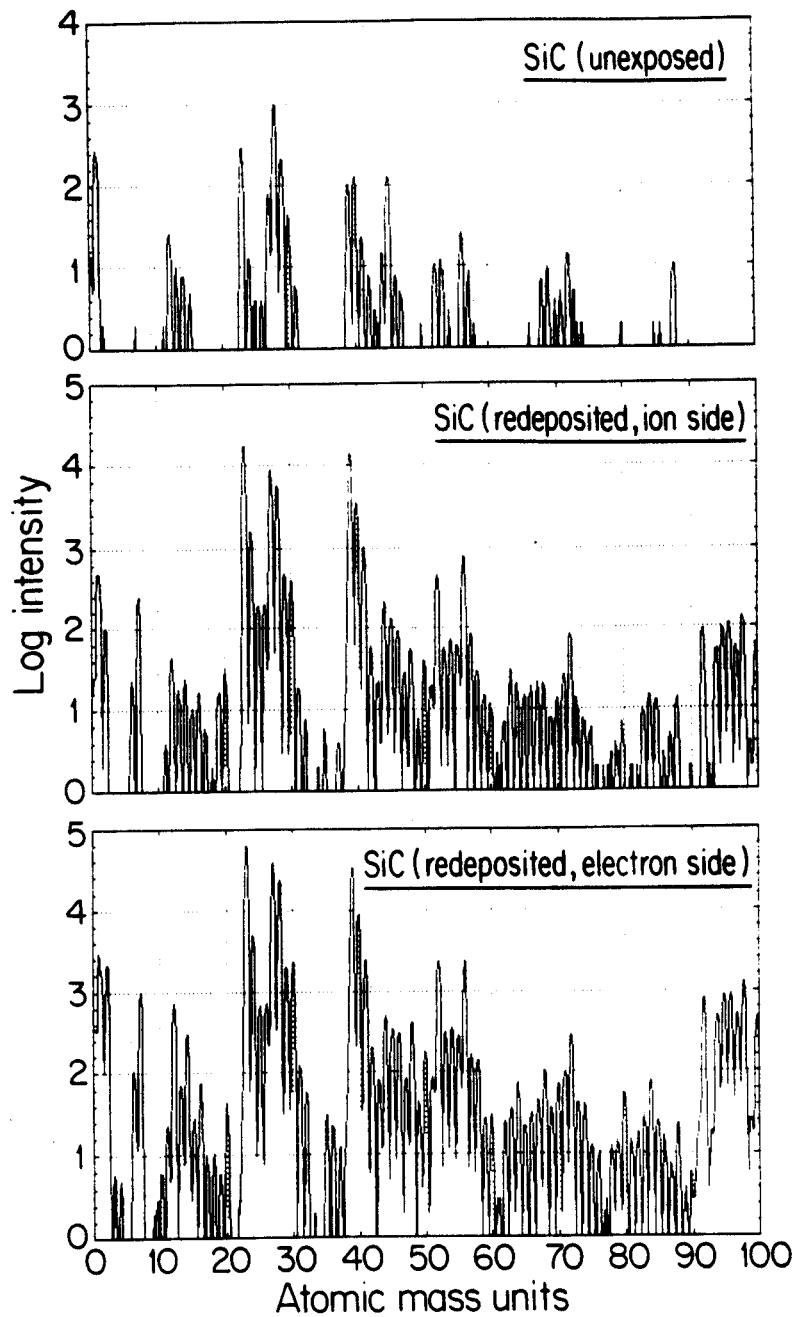


Fig. 8: Positive ion mass spectrum; unexposed SiC-coated graphite limiter (top); SiC-coated graphite limiter redeposited on the ion side (middle); SiC-coated graphite limiter redeposited on the electron side (bottom).