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The Use of Isotopically-Enriched Carbon Probes for Erosion/Deposition Measurements in the ASDEX Divertor*

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Abstract

An isotopic marker technique has been developed and used to measure near-surface erosion and carbon and impurity deposition on graphite samples in the ASDEX divertor chamber. Papyex graphite strips were enriched to ~20% ^{13}C over the first 1000 Å by ion implantation. The implanted ^{13}C can be clearly distinguished from ^{12}C of the host by Rutherford backscattering using 2.7 MeV ^4He . Carbon erosion and deposition were determined from changes in the depth distribution of the ^{13}C , and impurity accumulation was also monitored in the backscattering spectrum. The ^{13}C profiles remained sharp in spite of high heat fluxes to the samples (sufficient to melt stainless steel), and the results showed clear evidence of simultaneous erosion and deposition of carbon and impurities. During 2.4 MW neutral injection, net carbon erosion exceeded 250 Å/s in the divertor throat. High carbon erosion was also observed during periods of substantial Fe and Ti impurity accumulation on the samples. The results demonstrate the applicability of the ^{13}C technique for measuring erosion/deposition at high-heat-flux surfaces such as limiters or divertor plates.

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1. Introduction

Erosion of plasma-side surfaces may impose severe limitations on component lifetimes in future fusion reactors. For steady-state operation, erosion due to sputtering will dominate, and calculations¹ for FED/INTOR suggest net erosion rates of centimeters per year at the limiter. Total erosion (neglecting redeposition) is much higher and can approach 50 cm/yr. The local balance between erosion and deposition is clearly critical in minimizing material loss at high particle fluxes. In this work, we report the development and application of an isotopic marker technique for determining erosion/deposition at graphite surfaces exposed to high particle fluxes in fusion devices.

There have been several recent measurements of erosion in the plasma edge using surface probes,²⁻⁵ laser-induced fluorescence,⁶⁻⁹ and thin-layer activation.¹⁰ The present technique is unique in that it utilizes a marker which is chemically identical to the host and allows monitoring of erosion, redeposition, and impurity accumulation. The measurements were performed in the highest flux region of the ASDEX divertor where the samples encountered reactor-relevant heat and particle fluxes.

2. Experimental

Papyex graphite strips were implanted at room temperature with 35 keV ^{13}C ions from a high current ion implantation accelerator at Oak Ridge National Laboratory. Implants were typically at normal or 45-degree incidence to fluences of $2\text{--}4 \times 10^{17}/\text{cm}^2$. The depth distribution of the implanted ^{13}C was determined using Rutherford backscattering of 2.7 MeV ^4He ions. A backscattering spectrum for a sample implanted at normal incidence is shown in Fig. 1. The ^{13}C peak corresponds to a local concentration of ~ 30 atomic percent ^{13}C at $\sim 1000 \text{ \AA}$ depth.

Individual graphite strips were mounted on a cylindrical drum which could be rotated behind a molybdenum-edged slit in a stainless steel cover can. This arrangement allowed integrated or time-resolved exposures to be made by rotating the drum between or during discharges, respectively. In ASDEX, the ^{13}C -enriched probes intercepted a cross section of the diverted flux at normal incidence in front of the upper divertor plate. The probes were centered in the divertor throat and received a peak flux approximately ten times the flux at the divertor plates which intercept the field lines at grazing incidence. Power and particle fluxes to the probes were estimated to be as high as 2 kW/cm^2 and $10^{19}/\text{cm}^2\text{-s}$. Exposures were made for a variety of ASDEX operating conditions including discharges with 2.4 MW neutral injection and 80 kW of lower hybrid heating.

Several laboratory experiments were performed to qualify the ^{13}C marker for use under the above conditions. The ^{13}C depth profile was monitored using Rutherford backscattering during resistive heating of a probe. The profile was unchanged below 2000 K, but the ^{13}C peak disappeared at higher temperatures due to evaporation or C self-diffusion. Sputtering experiments at 1800 K using incident hydrogen ions indicated that the ^{13}C distribution was not affected by simultaneous heat and particle bombardment at plasma edge energies. Ion beam mixing of ^{13}C and ^{12}C was limited to the range of the bombarding ions ($\sim 150 \text{ \AA}$ for 100 eV H) which is much smaller than the $1000 \text{ \AA}$ ^{13}C implant depth. The chemical equivalence of the marker and host make this system ideal for self-diffusion and ion mixing studies.¹¹

Following exposure in ASDEX, the samples were again analyzed for ^{13}C profiles and impurities using Rutherford backscattering. It was found that the location of the ^{13}C peak with respect to the ^{12}C surface was the most convenient

measure of carbon erosion or deposition. This can be seen in Fig. 2 where backscattering spectrum for an as-implanted sample is compared with spectra from the same sample following erosion of 350 Å of ^{12}C and deposition of 500 Å of ^{12}C in two separate exposures in ASDEX. The ^{13}C peak shows little change in shape in spite of the erosion and deposition at high heat fluxes. Changes in the depth of the ^{13}C distribution could be determined to ~ 50 Å.

3. Results and Discussion

Spatially-resolved erosion/deposition results for a ^{13}C -enriched probe with an integrated exposure to six ASDEX discharges with 80 KW of lower hybrid heating are shown in Fig. 3. These data were taken in the divertor throat and correspond to main plasma parameters of $\bar{n}_e = 2.8 \times 10^{13}/\text{cm}^3$ and $I_p = 300$ kA. Impurity (O, Fe) accumulation shows a minimum at the center of the divertor throat where the maximum erosion would be expected. Wall impurities (O, Fe) are skewed toward the wall side of the separatrix while divertor impurities (Ti) show a minimum at this location. This is consistent with efficient scrapeoff of wall impurities entering the plasma edge and low erosion of the divertor plate for flux surfaces nearest the wall.

The ^{13}C -implanted region for this probe was not centered in the divertor throat and C erosion data are available only on the plasma side of the separatrix. The maximum C erosion was observed closest to the separatrix and corresponded to ~ 80 Å/discharge (~ 40 Å/s). Deposition of impurities reduced net erosion 10-20% below the C values depending on location. These results indicate that the accumulation of ~ 100 monolayers of impurities does not eliminate C erosion and, therefore, that the impurities must be mixed with the C in the near surface region.

Time-resolved erosion/deposition results for discharges with 2.4 MW neutral injection are shown in Fig. 4. These data are for main plasma parameters of $\bar{n}_e \approx 3 \times 10^{13}/\text{cm}^3$ before injection (dropping to $1 \times 10^{13}/\text{cm}^3$ after injection) and $I_p = 300$ kA. The results are integrated over ten discharges by indexing the probe rotation to the timing sequence for each discharge and correspond to a probe location in the center of the divertor throat.

In Fig. 4, C erosion increases rapidly in the beginning of the discharge to exceed $250 \text{ \AA/s}$ (erosion of the entire ^{13}C layer) at 0.6s. This large erosion rate is observed in spite of the accumulation $\sim 60 \text{ \AA}$ of Fe and Ti on the probe, again indicating mixing of the impurities in the near surface. During neutral injection, erosion remains high but impurity accumulation ceases. Following injection, net deposition of C is observed along with a slight accumulation of Fe and Ti. The shift from C erosion to deposition corresponds to a reduction in plasma density which could result in lower heat fluxes in the divertor throat. C erosion could be greatly reduced at lower sample temperatures. Averaged over the discharge, the results in Fig. 4 correspond to Fe and Ti accumulation of $\sim 2 \text{ \AA/s}$ and net C erosion of $\sim 80 \text{ \AA/s}$. This compares reasonably with the integrated results for the lower power discharges of Fig. 3.

It was apparent from blackening and deformation of the exposed samples that surface temperatures were high. This was also obvious from the occasional melting of the stainless steel can surrounding the samples. Evidence for high surface temperatures can also be seen in the surface morphology which developed in the samples during erosion. In particular, the micrograph for 1.5s into the discharge (Fig. 5) shows a surface structure characteristic of the sputtering of Papyex above 1500 K and is not observed for low temperature sputtering. It is important to note that the surface structures shown in Fig. 5

did not significantly alter the shape of the ^{13}C distribution as observed by Rutherford backscattering.

4. Conclusions

The erosion/deposition results presented above represent a first attempt to determine graphite erosion performance under reactor-relevant edge conditions. The exposures in ASDEX were probably not sufficient to "condition" the samples (in the sense that graphite limiters in tokamaks are conditioned by long-term exposure to the plasma). Accordingly, the present erosion results may be modified by additional surface conditioning. Nevertheless, the present study supports several general conclusions:

1) Extrapolated to steady-state, the erosion rates observed in the divertor throat would correspond to $\gtrsim 50$ cm/yr or ~ 5 cm/yr for the grazing incidence surface of a limiter or divertor plate. These values are consistent with net erosion estimates for FED/INTOR.¹

2) The simultaneous deposition of plasma impurity ions, at least at high surface temperatures, does not substantially reduce graphite substrate erosion for typical impurity fluxes in the plasma edge. This result is consistent with laboratory measurements of the sputtering of graphite at high temperature during simultaneous Ti^{12} and Ni^{13} evaporation on the sample, and requires mixing of the impurities in the near surface region or diffusion of the C through the impurity layer.

3) The use of ^{13}C -enriched graphite probes is an effective means for measuring erosion/deposition at high heat/particle fluxes in the plasma edge. It is expected that similar techniques will prove to be useful for monitoring erosion in other materials.

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Figure Captions

- Fig. 1. Rutherford backscattering spectrum for a graphite sample implanted at normal incidence with 2×10^{17} ^{13}C atoms/cm² at 35 keV. ^4He backscattering at 2.7 MeV clearly resolves the ^{13}C peak from the ^{12}C substrate even though the ^{13}C is 1000 Å below the ^{12}C surface. Heavier impurities on the surface would appear at higher backscattered energies as indicated for ^{16}O .
- Fig. 2. Comparison of backscattering spectra for a ^{13}C -implanted sample before exposure in the ASDEX divertor, following ^{12}C erosion of 350 Å, and following ^{12}C deposition of 500 Å. The position of the ^{13}C distribution with respect to the ^{12}C surface changes with ^{12}C erosion and deposition. This sample was implanted at 45° which accounts for the broader ^{13}C depth distribution compared to Fig. 1.
- Fig. 3. Spatially-resolved erosion/deposition results for a ^{13}C -probe spanning the divertor throat in ASDEX. The data are integrated over six discharges with 80 kW of lower hybrid heating. The plasma side of the separatrix is to the left.
- Fig. 4. Time-resolved erosion/deposition in the center of the divertor throat for ten ASDEX discharges with 2.4 MW of neutral injection at 1.1 - 1.3s. The maximum erosion rates correspond to removal of the entire ^{13}C layer.
- Fig. 5. Scanning electron micrographs of the exposed samples of Fig. 4 corresponding to several times during the discharge. The surface structure at 1.5s resembles structures developed during the sputtering of graphite above 1500 K.

RBS SPECTRUM OF ^{13}C IMPLANTED GRAPHITE

Fig. 1

BACKSCATTERING SPECTRA OF ^{13}C -IMPLANTED GRAPHITE

Fig. 2

Fig. 3

Fig. 4

Surface Morphology of Graphite Probes Exposed at Various Times in the ASDEX Divertor

(2.4 MW NI at 1.1–1.3 sec)

– 0.3 sec

0.6 sec

1.5 sec

2.1 sec

Fig. 5