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# Controlled irradiation of CFC samples in the scrape-off layer of Tore Supra

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#### Abstract

The first experiments with a mobile sample holder in Tore Supra are described. It exposes 10 CFC samples to direct irradiation by the scrape-off layer plasma. The plasma parameters are measured simultaneously by two Langmuir probes, and the temperature of the samples by embedded thermocouples. The cumulated irradiation dose during the first brief campaign was enough to exceed the classical saturation of the ion stopping zone, as verified by thermodesorption spectroscopy and nuclear reaction analysis. Scanning electron microscopy of some of the samples was performed before and after irradiation in order to investigate the evolution of the surface structure due to ion bombardment. © 2004 Elsevier B.V. All rights reserved.

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# 1. Introduction

Carbon-based materials are widely used as plasma facing components in fusion devices. Data related to long term plasma-carbon interactions, and in particular, carbon fiber composites (CFC), are needed given its possible use in the next step tokamaks [1]. To improve understanding it is useful to develop dedicated systems that can simultaneously expose test samples and measure their precise irradiation history.

The fast-scanning probe system that is installed on the Tore Supra tokamak operates routinely and reliably

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in all discharge scenarios, making up to ten plunges per discharge. It offers the facility to expose samples in a controlled manner to parallel particle and energy fluxes that are comparable to the poloidal fluxes expected in the ITER divertor. The probe regularly intercepts ion flux  $\Gamma_{\parallel,i}$  up to 10 A/cm<sup>2</sup> and energy flux up to 50 MW/  $m^2$  at normal magnetic field incidence (Fig. 1). Even though the fluxes are comparable, it is important to note that the density is much lower in the Tore Supra SOL than at the ITER divertor strike point, and the temperature is higher. A sample holder that can be easily mounted on the existing probe drive has been constructed and tested. It is described in Section 2. A complete history of the sample irradiation during the probe's first experimental campaign and measurements of the deuterium retention are given in Section 3.

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Fig. 1. The range of edge plasma parameters obtainable in Tore Supra. The points are from all discharges of one experimental campaign, including many values of magnetic field, central density, and additional heating power, at all radial positions attained by the fast-scanning probe.

# 2. Sample probe

The fast-scanning probe drive is situated on top of the torus. It plunges vertically along R = 2.534 m. The plasma major radius is typically R = 2.4 m and the minor radius is usually a = 0.72 m. The toroidal magnetic field (3-4T) and plasma current (0.6–1.6 MA) are oriented in the negative toroidal direction. Real-time feedback is employed to control the probe position with respect to the last-closed flux surface (LCFS). Before the discharge, the desired plunge depth is passed to a subroutine of the poloidal field program which controls the probe hydraulic drive. The probe can make a plunge of up to 46cm below its rest position at an average speed of 1–2m/s, and attain its target with a precision of ±5mm. The tip of the probe spends roughly 50–100 ms in the high flux layer.

The sample probe consists of two stainless steel trays that each house five samples whose dimensions are  $9 \text{ mm} \times 9 \text{ mm} \times 2 \text{ mm}$ . A hole of 0.5 mm diameter is drilled into each sample to accommodate a thermocouple. Five of the samples receive flux from the 'A-side' (electron or downstream side) of the holder, and five from the 'B-side' (ion or upstream side). The samples are numbered '1' to '5' from the deepest position. The ion flux and electron temperature profiles are measured by two directionally-sensitive Langmuir probes at the tip of the sample holder. The ion flux density and the electron temperature are always lower on the A-side than those on the B-side. Our system presents the advantage of exposing several samples to controlled discharge conditions, and being able to extract them during the experimental campaign for analysis. The spacing of the samples is such that four orders of magnitude variation of the incident fluence are obtained. The leading samples (A1 and B1) are exposed to intense irradiation in high flux, high temperature SOL plasma, while the trailing samples spend their time in colder less dense layers.

The trays are protected from plasma irradiation by a 5 mm thick CFC cylindrical housing into which orifices of 7 mm diameter are pierced such that only the samples are irradiated. The kinetic code XOOPIC [2] was used to simulate the ion flow through the orifices. Due to deflection of ion orbits in the magnetized sheath onto the inner wall of the orifice, a certain fraction of the incoming flux does not strike the sample [3]. This effect must be taken into account in order to be able to obtain accurate estimates of the collected deuterium fluence. The ion flux to the sample is typically reduced to 70–90% of the value at the leading edge of the orifice. The irradiation fluence during one plunge to sample B1 is typically a few  $10^{17}$  D<sup>+</sup> ions.

Knowledge of the temperature of the sample is crucial to understanding the mechanisms of deuterium trapping and surface modification. Three chromel–alumel thermocouples are embedded in samples A1, A3, and B1 which are initially at the same temperature as the tokamak ( $120^{\circ}$ C) but then heat up slowly during a day of plasma discharges. Brief exposure to plasma causes a short-lived temperature increase that lasts only as long as the irradiation. The maximum increase of surface temperature during the incident heat pulse rarely exceeds  $100-200^{\circ}$ C.

# 3. First results

During the campaign of autumn 2003 the probe made a total of 213 plunges during 66 discharges. The cumulative ion fluence and the distribution of ion impact energy were calculated using the Langmuir probes (Fig. 2 and Table 1). The upstream–downstream flux asymmetry is larger than the temperature asymmetry, leading to the possibility of making interesting comparisons and eventually to distinguish between flux and energy effects. For example, samples A1 and B2 collected the same number of deuterium ions, but the average ion implantation energy on A1 was comparable to B1. The thermal history of the most irradiated sample B1 is shown as a function of the cumulative fluence in Fig. 3.

The SEM micrographs show a variation of the degree of surface modification both from sample to sample, and across the diameter of the wetted area of a given sample. For example, SEM micrographs of the pyrolytic graphite matrix at the center of samples A1 and A2 are shown in Fig. 4, before and after irradiation (see Table 1 for the irradiation conditions). The signature of the irradiation



Fig. 2. Cumulated distributions of ion implantation energy estimated from Langmuir probe data for all samples. The distributions are normalized to give the total irradiation dose when integrated over energy.

Table 1

Total irradiation dose and mean ion implantation energy estimated from Langmuir probe data for all samples

Sample	Dose [D <sup>+</sup> ]	Mean implantation energy [eV]
Al	$8.1 \times 10^{18}$	400
A2	$2.1 \times 10^{18}$	200
A3	$4.7 \times 10^{17}$	90
A4	$6.8 \times 10^{16}$	70
A5	$2.8 \times 10^{15}$	40
B1	$2.3 \times 10^{19}$	430
B2	$8.3 \times 10^{18}$	290
B3	$2.2 \times 10^{18}$	190
B4	$2.8 \times 10^{17}$	130
B5	$1.0 \times 10^{16}$	60

intensity is the smoothing of sharp edges and the merging of grains smaller than a characteristic dimension, especially in regions where the pyrolytic graphite matrix is initially observed. On this particular region of sample A1 all features smaller than roughly  $0.5\,\mu$ m have vanished, whereas on A2 they remain essentially intact. One can suggest that erosion due to ion bombardment plays the main role in surface modification, while redeposition of carbon atoms can not be fully excluded at the moment. The basic characteristics of the surface



Fig. 3. Peak surface temperature of sample B1 as a function of the cumulated incident fluence, calculated as the sum of the sample temperature measured before the discharge and the theoretical temperature increase due to the irradiation as measured by the Langmuir probe.

structure evolution resemble what is observed in laboratory experiments [4].

After removal from the tokamak the surface distribution of deuterium concentration on samples B1 and B3 was estimated by nuclear reaction analysis (NRA). A 1.5 MeV <sup>3</sup>He ion beam  $(10 \times 10 \mu m^2)$  was used to produce  $D({}^{3}He,p)^{4}He$  reactions. The retention profile across the irradiated surface was measured in steps of 200 µm. Both samples show a decay of the deuterium concentration near the edge of the magnetic shadow of the orifice (Fig. 5). The shape of the concentration profile coincides with the kinetic ion orbit calculations which predict that 10-30% of the incident flux is lost to the inner wall of the orifice due to the combined action of the finite ion Larmor radius and the polarization drift inside the magnetized sheath. The amount of retained deuterium in the central region is much higher than the amount of particles that we expect to be collected in the saturated ion implantation zone. That is why one can believe that the radial profile in fact illustrates a proportionality between incident fluence and retention in the deep layers of the sample.

The total deuterium retention was estimated for each sample by thermodesorption spectroscopy. A radiating tungsten filament was used to increase the sample temperature linearly at a rate of 10 K/s up to 1500 K. The measured quantities of desorbed D<sub>2</sub> of the B samples, along with the NRA measurements, are compared with typical values from the literature in Fig. 6. The values seem reasonable, especially for the most irradiated samples B1–B3. Samples B4 and B5 give rather low results that could perhaps be explained by a number of possible measurement uncertainties. The retention is underestimated because only the desorbed molecular deuterium is counted; the contributions of HD and deuterocarbons are not yet included. The samples were exposed to air for several weeks before TDS, as well as to the water leak



Fig. 4. SEM micrographs of the pyrolytic graphite matrix at the centers of samples A1 and A2 before and after plasma irradiation. The displayed areas are roughly  $8 \mu m \times 8 \mu m$  and  $5 \mu m \times 5 \mu m$  respectively.



Fig. 5. Radial profile of retained deuterium fluence on samples B1 (squares) and B3 (diamonds). The narrow depression at r = -2mm corresponds to a scratch, or a mark left by the machining process. The full curve is the theoretical incident ion flux, integrated over the experimental campaign. It is normalized to fit onto the B1 data.



Fig. 6. Retained fluence as a function of irradiation dose as measured by the Langmuir probe. The results of TDS (full squares) and NRA (stars) are compared with results from the literature [5–7].

that terminated the experimental campaign of autumn 2003. It is expected that the desorbed molecular deuterium should be reduced by a factor 2 at most due to interaction with oxygen during the detrapping process, with the remaining deuterium appearing in an increased amount of hydrocarbon desorption [8]. The ion flux to the samples is somewhat overestimated because we neglect secondary electron emission by ion impact, but this will only account for a few 10% of the measured current. The contribution of ionized impurities to the total current is unknown, and can only be evaluated by sophisticated impurity transport calculations. Samples B4 and B5 received very low power, so we expect temperature excursions no greater than a few degree celsius, and consequently no desorption during the experiments. Finally, we note that the samples, like any CFC component mounted inside the tokamak, were not subject to any special treatment before the experiments. During the TDS, each sample released a huge amount of hydrogen, independent of its irradiation history (for example B5 released three orders of magnitude more  $H_2$  than  $D_2$ ). The release of HD molecules from samples B4 and B5 was also much higher than  $D_2$ . We need to verify if this hydrogen was introduced into the samples before, during, or after exposure, and evaluate whether its presence could have an effect on the deuterium retention at weak fluences.

# 4. Conclusions

At a time when decisions are being taken about what materials to use in ITER, it is important to be able to conduct plasma irradiation experiments in realistic, controllable conditions. We have described here the first experiments with a simple mobile sample holder that can expose 10 samples to the edge plasma of Tore Supra, while providing simultaneous measurements of ion flux and electron temperature, as well as the bulk temperature of the samples. The novelty of this probe is evident: for this first short campaign the probe was used as a standard Mach probe for physics studies, while at the same time gathering information about plasma–CFC interaction. The initial measurements of deuterium retention appear to be consistent with the literature. In future experiments we will try to investigate the process of the CFC structural modification under high intensity tokamak plasma irradiation, the features of the deuterium retention in tokamak conditions, and in particular, the details of the surface distribution of implanted deuterium. It will be possible to test ITER-relevant materials such as tungsten.

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