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# Hydrogen trapping in carbon film: From laboratories studies to tokamak applications



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#### ABSTRACT

During the DITS (Deuterium Inventory in Tore Supra) project, inner vessel walls of Tore Supra tokamak have been loaded with deuterium during a specific plasma experiment based on long pulse operation. Then, and in order to determine where the deuterium used as a plasma fuel was lost in the plasma facing components, the Tore Supra limiter had been dismounted and post mortem analysis had been performed on samples coming from different places corresponding to erosion or deposited area. In this the thermo-desorption spectrometry (TDS) studies are presented. All the TDS spectra present independently to their origin the same behaviour e.g. 5 desorption bands at 560 K, 820 K, 1050 K, 1238 K and 1375 K. These bands correspond to different trapping processes in carbon material which are identified with the help of laboratories experiments. Finally, the spectra are fitted by a rate equation model in which one of the main parameter which is the exponential prefactor is experimentally evaluated  $(2.7 \cdot 10^{15} \text{ s}^{-1})$ . This gives the possibility to unambiguously determine the trapping energies for the 5 trapping processes: 1.8 eV, 2.8 eV 3.6 eV, 4.2 eV and 4.7 eV. The model uses a dispersion in energy around these values due to the complex structure of Carbon Fibre Composite.

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# Introduction

In a fusion device like a tokamak, the plasma is not perfectly confined by the magnetic fields. The in vessel walls are submitted to tremendous fluxes of particles and energy. The material constituting the Plasma Facing Components (PFCs) must fulfil several mandatory conditions and their choice is one of the main challenges for the realisation of a fusion power plant. They have to be conductive in order to evacuate the impinging electrical charges and to be refractory to sustain high temperatures induced by the huge power flow (up to 15 MW/m<sup>2</sup>). The Carbon Fibre Composite (CFC) meets these constraints and during the last decades it was used as PFCs. The interactions between the walls and the plasma induce the trapping of particles in the plasma facing components. Part of these trapped particles can be afterwards released in the plasma. These fluxes (called the recycling fluxes) influence the stability of the plasma edge and thus on the global behaviour of the reactor. Furthermore, the overall tritium inventory in

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the tokamak is limited due to safety constraints. So, it is important to study and model the trapping and the release of hydrogen isotopes in PFCs in order to predict the recycling fluxes and the in vessel tritium inventory. In order to address these points, a dedicated study of fuel retention has been launched in the Tore Supra tokamak operating with CFC plasma facing components.

This is called the DITS project (Deuterium Inventory in Tore Supra) [1]. It is based upon a dedicated procedure which consists first to a heavy deuterium wall loading during dedicated plasma operation. Then, part of the PFCs interacting with the plasma is dismantled and carefully analysed in order to measure the deuterium inventory in the materials. Several post mortem analysis have been undertaken and among them Thermo-Desorption Spectrometry (TDS) [2]. In a tokamak environment, the plasma wall interaction process is far to be homogeneous. That is why some areas are eroded and others covered by materials transported by the plasma from the eroded zone. Thus, onto in vessel components, different interaction patternsare observed from pure erosion area to thick deposition zone. In order to accomplish a global TDS analysis, a large number of small samples have been cut from the PFCs originating from these different zones of the machine.

In the presenting paper, the experimental results obtained during TDS are first detailed. A comparison with previous works [3,4] is proposed to assess the deuterium trapping processes which can be considered during plasma operation in Tore Supra. Complementary experiments, mainly ions implantation in dedicated samples coming from an erosion area followed by TDS, are proposed and analysed. It allows assessing a fundamental parameter e.g. the pre-exponential factor needed for TDS modelling which is proposed at the end of this presentation. Finally, the deuterium trapping energies in the Tore Supra PFCs are determined.

# Experiment

### Samples

The samples investigated here are coming from the Toroïdal Pumped Limiter (TPL) of Tore Supra. TPL is made of CFC-N11 Pan fibres, CFC from Mersen [5]. The sample has been extracted from TPL and then cut following a procedure shown Fig. 1 [2].

In the erosion zone, more 700  $\mu$ m of material had been removed from the PFCs. The sputtered material was redeposited in layers of variable thickness. In "thin deposited



Fig. 1 – Schematic view of cutting TPL sample. From Ref. [2].

zone" a redeposited carbon layer thickness of 10–100  $\mu$ m had been measuredon top of the bulk material and in "thick deposit zone" a carbon layers of 400  $\pm$  100  $\mu$ m has been measured [6]. The temperatures of the different areas during the plasma discharges have been evaluated by infra-red thermography [2]. Tore Supra is an actively cooled machine and the PFCs operating temperature is 100 °C. During plasma discharges, the temperature of the erosion zone remains below 200 °C. The temperature of the thin deposited areas which are far from the plasma stays almost constant (between 100 and 120 °C). This is not the case of thick deposited layers where 500 °C can be easily reached. This discrepancy has been attributed to higher thickness and poor thermal conductivity [7].

14 samples have been extracted from these different zones. The samples have always the same size e.g.  $8 \text{ mm} \times 8 \text{ mm}$  and 0.8 mm of depth. 11 samples were taken from the surface of the PFCS tiles which is facing the plasma discharge and 3 samples from the gap between two adjacent PFCs. Obviously, the latter were not subjected to plasma interaction. The eleven samples from the top of the PFCs surface were divided as follow: 5 samples from eroded zone, 4 samples coming from thin deposit layer and 2 samples coming from thick deposit.

## TDS devices

TDS measurements have been undertaken using a small high vacuum device with a chamber of 30 L. The residual gas pressure in the chamber P ~  $1.10^{-6}$  Pa dominated by H<sub>2</sub>O and H<sub>2</sub>. This device is equipped with 2 pressure gauges and an RGA (Residual Gas Analyser). Before any experiments, the mass spectrometer of TDS device is calibrated with gas (H<sub>2</sub>, D<sub>2</sub>, HD, N<sub>2</sub>, and Ar). The sample to be studied is heated by a heated tungsten filament which is placed close to the sample. The thermocouple is imbedded in the sample allowing a careful control of the temperature ramp up. For TDS measurements the temperature ramp up is 1 K/s.

The preliminary experiments showed that the main fraction (90%) of the D and H atoms are desorbed in the form of D<sub>2</sub>, HD, H<sub>2</sub>. However, due to the pollution of the vacuum vessel by hydrogen, the amount of hydrogen desorbed from the samples has been established following a procedure presented in Ref. [8]. The D inventory obtained by TDS measurements has been compared previously with Nuclear Reaction Analysis (NRA) showing a good agreement between both techniques (10–15% of discrepancy) and thus validating the TDS procedure [2].

#### Implantation devices

It will be shown in the following that complementary TDS studies are required. Three similar samples coming from the eroded zone will be implanted with deuterium in a plasma device at the PIIM laboratory in Marseille. The residual gas pressure in the chamber is below  $10^{-8}$  Pa. Prior to implantation, the samples were degassed at 1200 K. The deuterium implantation energy is 100 eV/D and the implanted ions flux density is ~ $10^{16}$  D<sub>2</sub><sup>+</sup>/m<sup>2</sup>/s. The total irradiation fluence is 5.1  $\cdot 10^{18}$  D<sub>2</sub><sup>+</sup>/m<sup>2</sup>. The temperature is slightly increasing during

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