ACCELERATOR MASS SPECTROSCOPY MEASUREMENTS OF THE DEUTERIUM INVENTORY IN THE TORE SUPRA TOROIDAL LIMITER

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Absolute values of deuterium fuel deposition/retention on the limiter of Tore Supra Tokamak (France) were measured by Accelerator Mass Spectrometry Depth Profiling (AMS-DP). The concentration values found were approximately 100 times lower than previous measurements performed by Nuclear Reaction Analyses (NRA) 3 years ago.

Keywords: Accelerator Mass Spectrometry, deuterium profiling, nuclear fusion, Tore Supra divertor

1. Introduction

Fuel retention inside plasma facing components (PFC) is an important issue for International Thermonuclear Experimental Reactor (ITER) and many experiments were devoted to tritium retention characterization in wall machines [1, 2, 3]. Erosions and subsequent depositions appearing during plasma discharges are the main phenomena that explain why in depositing areas the long-term fuel retention is mainly occurring. PFC erosion/deposition patterns are needed to analyse where and how many particles are eroded or deposited, and how much fuel they contain.

A specific experiment was performed in Tore Supra tokamak from Cadarache, France, dedicated to the search for consistency between wall deuterium inventories estimated from gas balance and post-mortem analysis and to the characterization of the D retention mechanism. The main PFC of Tore Supra (TS) is the Toroidal Pump Limiter (TPL) – a castellated structure composed of 12096 Carbon Fiber Composite (CFC). This major in-vessel component consists of a flat toroidal continuous disk (Ri = 2.2 m, Re = 2.7 m) covering a 7.6 m2 area and made of 576 radial high heat flux elements called "fingers". The enhanced in-vessel configuration of the machine with TPL was a part of Composant Internes Et Limiter (CIEL) project [4, 5]. A photograph of Tore Supra in the CIEL configuration is displayed in Fig. 1.

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Fig 1: Panoramic view of Tore Supra CIEL configuration (left). TPL image showing erosion and co-deposits areas (right) [6]

After an intensive Deuterium (D) wall-loading campaign between 2007 – 2011 consisting in 18000 s of discharges equivalent to 1-year operation in 3 weeks, a sector was dismantled for a detailed study of the coupled carbon–deuterium balance [7, 8] and extensive post-mortem analyses [9, 10, 11]. A complex erosion/deposition pattern related to the periodic magnetic structure of TS is observed. Several tiles extracted from different zones have been investigated using scanning electron microscopy (SEM). Eroded or deposited carbon volumes have been determined for the tile plasma-facing top surfaces as well as for the tile side surfaces inside gaps [10, 12, 13]. An important work was performed post mortem to determine the carbon-deuterium balance [14] and to establish the erosion/deposition mapping on the TPL [15, 16]. Several analysing methods have been applied to investigate the physics at TS like SEM, Con-focal Microscopy, Lock-in thermography, Raman spectroscopy, Transmission Electron Microscopy and NRA [14, 17, 18]. First measurements on dismantled tiles show Deuterium/Carbon ratio ranging from 0.04 to 0.20, on a typical thickness of 15-20 µm and extrapolated to the whole TPL, this yields an amount of $\approx (1.5\pm0.3) \times 10^{24}$ atoms D [7]. In depth-structures and composition of the deposition were determined and the deuterium inventory was deduced from the carbon inventory by assuming a D/D+C ratio of 14% in Tore Supra [15].

We present in this manuscript the results of our post mortem measurements regarding the deuterium retention in different tiles dismounted from TS TPL after the same campaign. Quantity determination of the deuterium retention was performed by applying the Accelerator Mass Spectroscopy (AMS), upgraded to perform depth profiling of fuel concentration applied in fusion experiments [19, 20].

2. Experimental

1.1. Samples

Fig. 2 shows the dismantled sector of the TPL which provided the tiles that were analysed in our study. Fingers 1–32 correspond to 20^0, the period of the main magnetic field (angle between two toroidal coils, 18 coils).

Fig. 2: The LPT of Tore Supra after plasma discharges. Stars are indicating the location from where the sampling was done. Blue, red and yellow stars marked the erosion area, thick deposition and respective, thin deposition.

Each finger is composed of 21 tiles which size along the toroidal direction is between 22 (high field side) and 28 (low field side) mm and along the poloidal direction is 22 mm. Different zones are distinguished: the erosion zones correspond to the shiny zones and the deposition zones can be separated in thick deposit zones (loosely attached deposits, grey zones), and thin deposit loaded or shadowed zones (well attached deposits, dark grey or black zones, resp.). The tile numbering is FxTy for tile y on finger x [10, 16]. Each tile was cut into several fragments indexed with Q1, Q2, Q3 and Q4 (as shown in Fig. 3).

In this study, fragments of the limiters tiles were analysed, dismounted from 4 toroidal positions (fingers: 5, 10, 26 and 27) and from 7 radial positions from various toroidal positions (1, 2, 4, 8, 9, 19 and 20). The positions of samples, measured by AMS, are shown in Fig. 2 through blue, red or yellow stars.
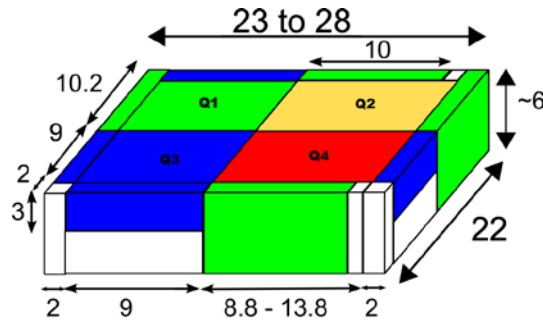
Fig. 3: Diagram of cutting the tiles.

For the AMS post-mortem analyses, fragments of tiles from different regions were cut to small slices (7 x 7 x 4 mm) suitable from mounting into the target holders of the ion source. The fragments indexed with F5T1 and F26T1, from the poloidal edges of the limiter, are curved (2mm radius, see [12].)

The analysed tiles and their description are present in Table 1.

Table 1.

The samples analysed and the area where they were cut (see Fig. 2).

Tiles	Zones	Measured date
F5T1	Erosion zone	April 2013
F5T2Q2	Erosion zone	April 2013
F5T9Q2	Thick deposit	April 2013
F10T19Q3	Thin deposit	April 2013, Nov. 2013, 2014
F26T1	Thick deposit	April 2013
F27T4Q2	Thick deposit	April 2013
F27T8Q1	Erosion zone	April 2013
F27T20Q1	Thin deposit	April 2013, Nov. 2013, 2014

1.2. Techniques

The Accelerator Mass Spectrometry is a highly sensitive analysing method that selects and counts the atoms individually. The sensitivity goes down to about 10^{-16} for the isotope /element ratio. The central part of such an analysing system is a high energy linear accelerator that increases the energy of ions in order to enhance the separation power of a large analysing magnet (Fig. 4).
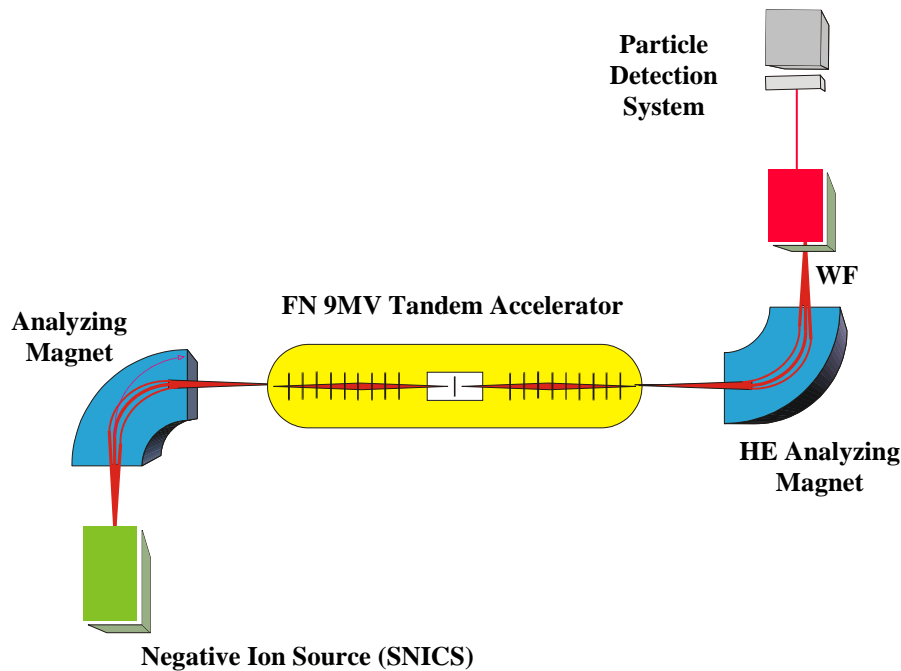
Fig.4: The AMS layout of the machinery at the 9 MV tandem accelerators.

In a nutshell, the analysing machine consists of a Sputtering Negative Ion Source (SNICS), a tandem particle accelerator, several electromagnetic filters (magnetic and electric analysers) and a sensitive charged particle detector. The samples to be analysed are loaded into the SNICS and are bombarded by a $^{133}Cs^+$ accelerated ion beam in order to produce sputtered ions from the target. Only negative sputtered ions are electrically extracted, focused and accelerated to a confined ion beam that is then analysed by electromagnetic filters in order to select the desired type of ions. This is done in a so called "rare isotope ion detector system" that in our experiments consisted in an array of three solid state Si(Au) pin detectors.

Since AMS is a relative analysing method, it requires reference samples for calibration of quantitative measurements. For the determination of the depth profile concentrations of elements in materials, [21, 22] one can take advantage of the ^{133}Cs sputter beam of the ion source that advances into the depth of the sample-target material producing a crater. With the elapsed time, ions will be extracted from a larger depth and the data acquisition system will measure the element concentration in the explored material as a function of time. At the end of the AMS experiment, performing an optic profilometry measurement, we can determine the depth and dimensions of the produced crater. Then, we can easily convert the time scale of the event registration to a depth scale. In this way the AMS concentration depth profile spectrum will be determined. The main problem

to overcome in an AMS-depth profiling experiment is the crater rim effect produced by the ion beam sputtering. That is because ions sputtered from the side walls of the crater should not be considered in the analysed data set since they do not reflect the correct concentration at the bottom of the crater and will falsify the depth profile spectrum. A mathematical unfolding procedure can correct the AMS depth profile distribution for the perturbing contribution added by the secondary ions, when sputtered from the sidewalls of the produced crater [23]. The mathematical procedure is performed at the end of the AMS analysis and uses the crater dimensions determined by optic profilometry (see Fig. 5).

For quantitative deuterium measurements, standard samples and blank samples were used. All results were corrected for crater rim effects during the depth profiling of concentrations. For reference samples and background samples the error was 1% and 3%, respectively.
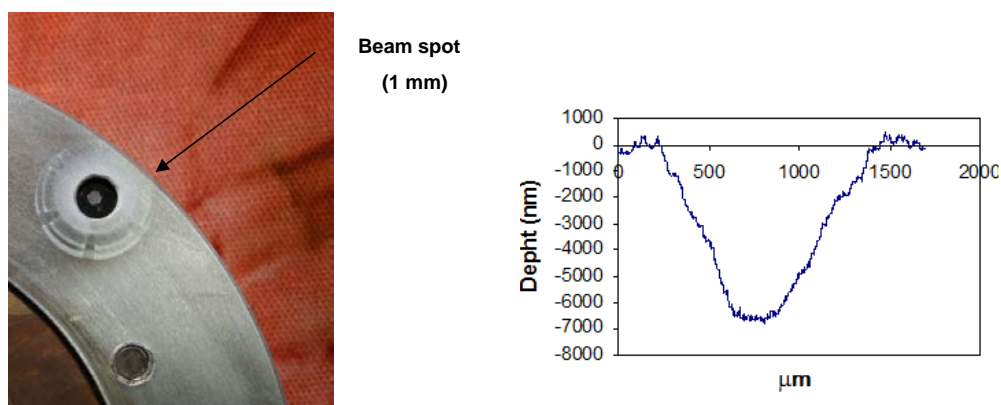

Fig. 5: (Left) The sputter beam spot on the graphite sample containing deuterium. (Right) The sputtered crater determined by optic profilomerty.

3. Results and discussions

Measurements of the deuterium content were done by the use of AMS – DP method, accordingly to the procedure described in section 2.2. For the AMS analyse, the samples were cut from the LTP tiles (see Fig. 2 and Table 1).

The LPT analysed protection tiles were covered by material eroded from the reactor vessel during plasma discharges of Tore-Supra. The exposed tiles were also eroded, and the co-deposition zones alternate with the erosion zones. Carbon co-depositions on tiles were measured to be up to 0.4 mm tick. The thin depositions were evaluated to about 0,1mm thickness [13].

Fig. 6 shows the AMS–DP spectra obtained from thick deposition areas. Since the AMS –DP measurement was done up to 25 mm, the concentration values are

regarding the deuterium captured in the co-deposited layer on the tile. The depth profile shows a flat maximum centred on the depth of about 3-8 μm. The retained deuterium inventory (see Table 2) is the largest for the thick deposition areas. The DP spectra are all similar in shape and concentration values.
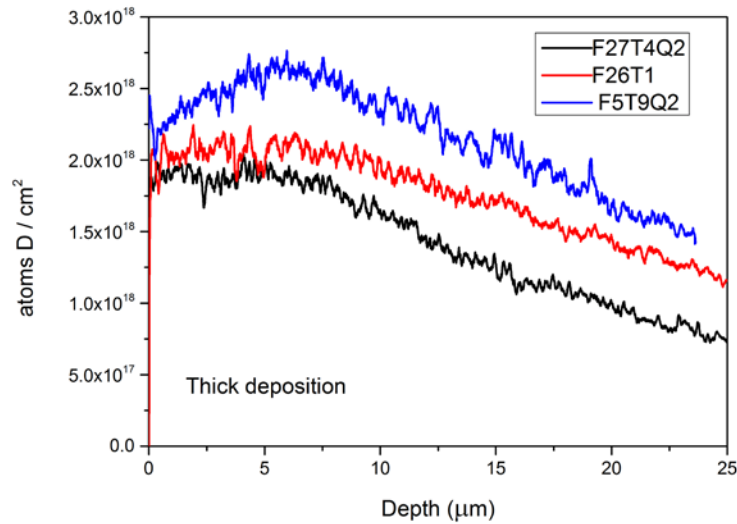

Fig. 6: The AMS-DP spectra of the deuterium concentration in tiles having thick carbon depositions on their surface.

This shows that the co-deposition produced by long term discharges have a continuous source of eroded material in the tokamak vessel following the magnetic field geometry in the machine. In this respect the Tore Supra offer the possibility to investigate the build-up of such co-depositions during long time plasma discharges, as will be at ITER, but in harmless conditions since it uses deuterium and not tritium.

Fig.7 presents the DP spectra for the erosion zones. The spectra have a slow exponential decreasing slope showing that diffusion is the main process in trend. The inventory of accumulated deuterium is 10 times lower than for the tick deposition areas. This difference is in accordance with the measurements performed before by NRA [7].
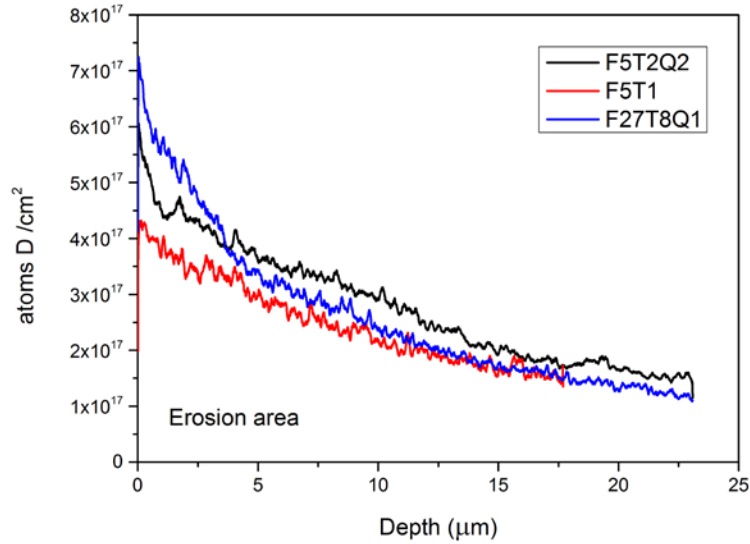
Fig. 7: Concentration depth profile spectra of deuterium in erosion areas.
The tiles from thin area were additionally cut in a several smaller slices. In this way it was
possible to repeat the AMS analyse for the same location after 6 months and after 1 year
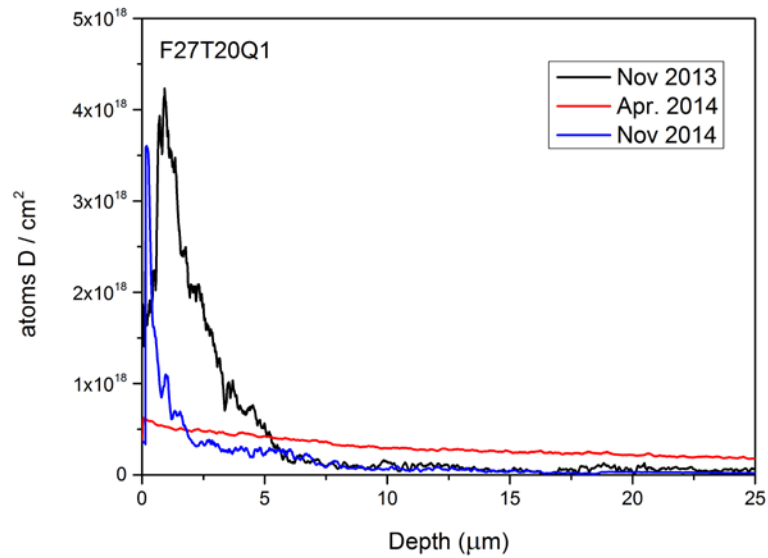(see Fig. 8 and Fig. 9).

Fig. 8: The AMS-DP spectra of the deuterium concentration in tile F27T20Q1 having thin
carbon depositions on their surface. Measurements were done at 6-month distance.
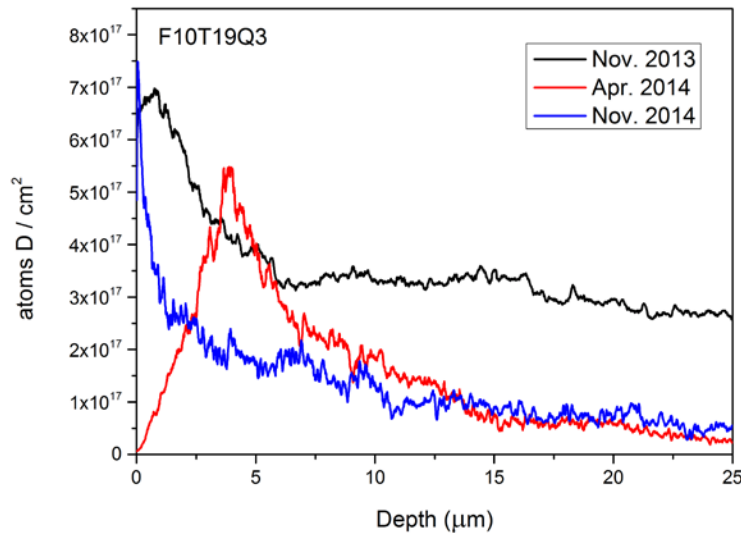
Fig. 9: The AMS-DP spectra of the deuterium concentration in tile F10T19Q3 having thin carbon depositions on their surface. Measurements were done at 6-month distance.

For areas with thin deposition of D, the initial maximum of retention is located on the surface. Towards the inner part of the tile, in the bulk, the retention concentration of D decreased rapidly with a long "tailing" due to the hydrogen isotope diffusion in the CFC structure of the tile. The inventories for deuterium retention in these areas are fluctuating in value more than 20%, indicating that such retentions are randomly distributed. Similar findings were reported in [9].

It was noticed that repeating the measurement after 6 month the deuterium content in the same surface layer decreased. This trend was continuing, and measurements performed after 1 year determined only very low deuterium concentration.

Table 2 presents the measured inventories for the spectra presented in Fig. 6-9 in comparison with NRA measurements [7]. The values of deuterium inventories determined from samples cut from different areas of the TLP are expressed in units of atoms/cm^2. As already mentioned, the difference between global retention of thick deposition and erosion areas is a factor of 10.

The tile from thin deposition areas have values between to the retention inventories of erosion and thick deposition zones. Our results, in absolute values, are about 100 times lower than those measured three years ago by NRA. An explanation for these differences could be the sample degassing during this elapsed time interval.

Table 2: Deuterium inventories measured for AMS – DP spectra

Zones	Tiles	D (atoms/cm²)	Average per zone	NRA measurements
Thick deposit	F5T9Q2	7.92 x10²¹	(6.58±1.45) x10²¹	(3.6±2.9) x10²³
	F27T4Q2	5.04 x10²¹		
	F26T1	6.77 x10²¹		
Erosion zone	F5T1	7.72 x10²⁰	(7.84±0.81) x10²⁰	(3.6±1.3) x10²²
	F5T2Q2	7.10 x10²⁰		
	F27T8Q1	8.70 x10²⁰		
Thin deposit	F10T19Q3	1.09 x10²¹	(1.17±1.10) x10²¹	(1.9±0.3) x10²³
	F27T20Q1	1.25 x10²¹		

4. Conclusions

The deuterium retention on the TPL of the Tore Supra was quantitatively measured by the use of AMS depth profiling analysing method. Samples were cut from tiles extracted during the 2007 – 2011 campaign [7].

The three typical zones on the LTP were also distinguished by the D-concentration depth profiling. These are the erosion-dominated zone, where the ion flux is the highest and where no deposition exists, the thin deposition zone, where deposits are well attached, and the thick deposition zone, where deposits easily flake. They are produced by the magnetic field ripple that produces a non-uniform ion flux onto the TPL. As a consequence, the competition between erosion and deposition leads to either erosion or co-deposition dominated zones. The retention values are characteristic for each zone excepting the thin deposition area where these values are randomly distributed but, the DP spectra showed a pronounced surface peak indicating that deuterium is located at the initial impact point of the plasma with the LPT tile. Unfortunately, the data have poor statistic due to the small number of measured tiles. The retention originates from the deposition of hydrogenated carbon layers produced by plasma bombardment on the vessel walls and the ability of carbon to bond with H isotopes [24]. However, as shown by SIMS measurements also Be, B and Ni contribute to retention. Such surface features on the TPL bring experimental evidence of the ion transport in a magnetized sheath.

The co-deposition is a very disturbing phenomenon in all fusion reactors that has to be solved in order to reduce the fuel retention and the contamination of the machines.

Absolute measured values by AMS were compared with NRA values measured 3 years ago. When comparing the results, a decrease of about 100 times of the retention values was observed. This can be explained by the tritium

degassing during this period of time. It was also noticed that the relative ratios between different retention zones remain similar for both methods.

Finally, since the erosion/co-deposition mapping is a fingerprint of the plasma wall interaction, it will be important to investigate this mapping for the WEST tungsten limiter too.

Acknowledgements

R E F E R E N C E S

[1] *T. Loarer*, Fuel Retention in Tokamaks, J. Nucl. Mater, 390-391 (2009) 20

[2] *T. Loarer, C. Brosset, J. Bucalossi, P. Coad, G. Esser, J. Hogan, J. Likonen, M. Mayer, Ph. Morgan, V. Philipps, V. Rohde, J. Roth, M. Rubel, E. Tsitrone, A. Widdowson*, Nucl. Fusion 47 (2007) 1112–1120

[3] *C. Brosset, H. Khodjab*, Deuterium concentration in deposited carbon layers in Tore Supra, Journal of Nuclear Materials, Volumes 337–339, 2005, Pages 664–668, doi:10.1016/j.jnucmat.2004.10.045

[4] *J.J. Cordier, P. Bayetti, P. Chappuis, A. Durocher, F. Escourbiac, A. Grosman, M. Lipa, R. Mitteau, J. Scholosser, D. van Houtte*, The tore supra Toroidal Pump Limiter : Experience feedback of HHF elements series manufacture, ,doi: 10.1109/fudion.2003.1425866, Conference: Fusion Engineering, 20th IEEE/NPSS Symposium on, (2003)

[5] *J Bernardo, C Fenzi, C Bourdelle, Y Camenen, H Arnichand, João P S Bizarro, S Cortes, X Garbet, Z O Guimarães-Filho, T Aniel*, Density impact on toroidal rotation in Tore Supra: experimental observations and theoretical investigation, Plasma Physics and Controlled Fusion, Volume 57, Number 3

[6] *J. Bucalossi, Y. Corre, A. Ekedahl, P. Monier-Garbet, V. Basiuk, S. Bremond, L. Colas, M. Goniche, F. Kazarian, O. Meyer, P. Moreau, F. Rimini* , High RF power operation issues on Tore Supra, 34th EPS Conference on Plasma Phys. Warsaw, 2 - 6 July 2007 ECA Vol.31F, P-5.113 (2007)

[7] *B. Pégourié, C. Brosset, E. Tsitrone, et al.*, J. Nucl. Mater. 390–391 (2009) 550–555.

[8] *S. Panayotis, B. Pégourié, E. Caprin, D. Douai, J.-C. Hatchressian, V. Negrier, J.-Y. Pascal, S. Vartanian, J. Bucalossi, P. Monier-Garbet*, Journal of Nuclear Materials 438 (2013) S1059–S1062

[9] *E. Tsitrone, C. Brosset, B. Pégourié, et al.*, Nucl. Fusion 49 (2009) 075011.

[10] *T. Dittmar, P. Roubin, E. Tsitrone, et al.*, Phys. Scr. T138 (2009) 014027.

[11] *E. Delchambre et al.*, Characterization of the carbon erosion on the limiter of Tore Supra. Journal of Nuclear Materials 390–391 (2009) 65–67

[12] *C. Martin, B. Pégourié, R. Ruffe, et al.*, Phys. Scr. T145 (2011) 014024.

[13] *C. Martin, R. Ruffe, C. Pardanaud, et al.*, J. Nucl. Mater. 415 (2011) S258–S261.

[14] *B. Pegourie et.al.* J. Nucl.Mater (2013), Journal of Nuclear Materials 438 (2013) S120–S125

[15] *C. Pardanaud, G. Giacometti, C. Martin, R .Ruffle T. Angot, E. Ateou, B. Pegourie, E. Tsitrone, T. Dittmar, C. Hopf, W. Jacob, T. Schwarz-Selinger, P. Roubin*, J.Nucl.Mater.415 (2011) 254-257.

[16] *C. Martin, P. Languille, S. Panayotis, B. Pegourie, H. Roche, C. Pardanaud, G. Giacometti, R. Ruffle, E. Gauthier, X. Courtois, E. Tsitrone, P. Robin*, J.Nucl.Mater. 438 (2013) 771-774.

[17] *E. Gauthier, C. Brosset, H. Roche, E. Tsitrone, B. Pégourié, A. Martinez, P. Languille, X. Courtois, Y. Lallier, M. Salami, Journal of Nuclear Materials 438 (2013) S1216–S1220*

[18] *T. Dittmar, E. Tsitrone, B. Pegourie, I. Cˇadezˇ, P. Pelicon, E. Gauthier, P. Languille, J. Likonen, A. Litnovsky, S. Markelj, C. Martin, M. Mayer, J.-Y. Pascal, C. Pardanaud, V. Philipps, J. Roth, P. Roubin, P. Vavpeticˇ*, Deuterium Inventory in Tore Supra (DITS): 2nd post-mortem analysis campaign and fuel retention in the gaps, Journal of Nuclear Materials 415 (2011) S757–S760

[19] *M. Dogaru, M. A. Calin, C. Stan-Sion,* J. Radioanal. Nucl. Chem 288 (2011) 491-498.

[20] *C. Stan-Sion, M. Enachescu*, Rom J Phys 59, No.9-10 (2014) 930-940

[21] *C. Stan-Sion, M. Enachescu*, Nucl. Instrum. Methods Phys. Res., Sect. B 361 (2015) 250-256.

[22] *C. Stan-Sion, A. Petre*, U.P.B. Sci. Bull. Series A 76 (2014) 265-274.

[23*] M. Enachescu , V. Lazarev, C. Stan-Sion*, J. Phys. D: Appl. Phys. 39 (2006) 2876–2880.

[24] *R. Mitteau, J.C. Vallet, A. Moal, D. Giulhem, J. Schlosser, L. Loarer, B. Riou, A. Grosman, A. Geraud, Ph. Ghendrih, E. Tsitrone, B. Pegourie, J.M Ane*, J.Nucl.Mater. 313-316 (2003) 1229-1233.