

A STRIKING EFFECT OF TRITIUM RETENTION IN GRAPHITE PROTECTION TILES

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The work presents the first complete depth profiling distributions measurements of tritium retention in carbon protection tiles employed in fusion devices. An unexpected Tritium retention on the backside of the protection tiles was determined that is dependent on the tile thickness. On the tile sidewalls the retention increases from the surface towards the backside. The accumulation of tritium on the backside was found to be ca. 3 orders of magnitude greater than in the bulk. The experimental data were determined and confirmed by Accelerator Mass Spectrometry (AMS) and Full Combustion followed by liquid scintillation counting of tritium.

Keywords: Accelerator Mass Spectrometry, tritium profiling, tritium absorption in graphite

1. Introduction

The heavier isotopes of hydrogen namely deuterium and tritium are fuels of the fusion process, and their retention must be avoided by all means in a tokamak. Many methods have been used to prevent, recover and analyze tritium in a variety of plasma facing materials. In order to withstand the huge temperature during the fusion reactions, the fusion reactor vessel is lined up with carbon protection tiles. Tritium retention and removal in such tiles has been extensively reported [1-7]. However, a complete depth proofing of the accumulated tritium in the Carbon Fiber Composite (CFC) protection tiles in the situation of tritium high pressure exposure has not yet been measured.

This paper presents the results of complete depth profiling measurements of the tritium retention in such CFC carbon tiles after the bombarded with tritium gas. Measurements were performed by the use of the highly sensitive analyzing methods for tritium: The Accelerator Mass Spectrometry (AMS) and the Full Combustion (FC) followed by liquid scintillation counting of tritium [8-13].

On the backside of protection tiles where the thermal and dynamic encumbrances were kept by far smaller than in front side some questionable and striking effects of tritium retention were observed that will be presented below.

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2. Experimental

AMS and FC were measuring the tritium retention in plasma facing components (PFC) for more than 10 years ago. Both methods are highly effective and sensitive in measuring the tritium content in carbon samples and have been used in many applications for the fusion experiments and for many other applications [14].

However, investigation of the tritium retention that extends over the entire depth length of the tiles has not yet been performed due to the extremely long measuring time required by such analyze. In graphite, AMS explores the content of tritium with a speed of about 15 $\mu\text{m}/\text{h}$ and the FC cannot handle samples heavier than few grams that restrict the sample thickness to about 1-2 mm. Most information of the tritium retentions relies on measurements of the surface concentrations accumulated on the PF's. Based on such information, it was believed that the accumulated tritium concentration has its maximum on the surface of the tile, then drops rapidly towards the inside of the tile [15] and remains at a low concentration value up to the end of the tile depth length. Diffusion should be the triggering force and responsible for the distribution in the bulk.

AMS and FC, which are complementary methods since the former determines the tritium retention on a nanometer-scale inside the material and the former on an mm-scale, were applied to get complete information of the tritium retention over the entire length of the tiles and to determine the retention on the sidewalls, also.

Both methods require samples of reduced size and therefore cuts were performed on the tiles as cylinders with diameters of 16 mm. Then, the hollow cut samples were cut into slices 1 mm thick (see Fig. 1).

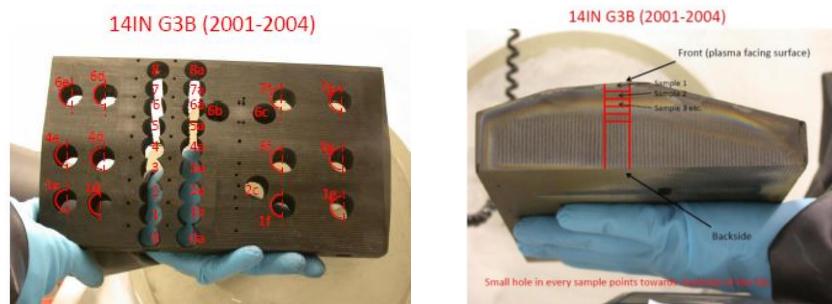


Fig. 1: The measured tile and the various locations of the cuts. The corresponding drilled cylinders for the AMS and FC measurements are 4e, 6e, 1f, 3f and 7f.

For the AMS analyze small cuts of slices (7 x 7 x 2 mm) were loaded into the target holder of the vacuumed ion source. There, the bombardment of the sample material with a Cs133 accelerated and focused ion beam produces desired

ions. Depending on the elapsed time, the sputtered negative ions are then electrically extracted from a progressively larger depth of the produced crater in the sample. The extracted ions are accelerated and analyzed by use of electric and magnetic filters in order to select only the desired ion species.

For tritium measurements the other negative ions with mass 3 (the hydrogen-deuterium molecule HD⁻ and the hydrogen molecule H₃⁻) are selected and eliminated by the injector-analyzing magnet and then accelerated by a tandem accelerator to the positive high voltage in the MeV range. After the tandem accelerator a large analyzing magnet selects the particles according to their masses and ion charges to eliminate the molecular fragments (HD⁻ and H₃⁻), which are broken in the tandem stripper terminal foil, counting ultimately only the tritium atoms. The particle detector is placed at the end of the AMS analyzing system that registers the concentration depth profile by counting the atoms one by one. The depth profiling of the concentration of tritium in the bulk of the sample is performed by taking advantage of the sputtering process performed by the focused Cs beam into the carbon material of the sample.

As the sputter beam is advancing into the bulk of the sample this it produces a crater. The analysis is done step by step and the data acquisition system will record a time distribution of the concentration measured by the detector during the entire sputtering process.

After the AMS experiment the time scale is converted to the depth scale by use of an optic profilometer that measures the produced crater depth and its geometric form. These data will also be used to calculate the correction of the crater rim effects on the depth profile. In this way AMS is measuring continuously the concentration of the element into the depth of a sample and its resolution can go down too few nanometers.

On the other hand, the complementary FC method coupled to the Liquid Scintillation Counting exploits the radiochemical character of the tritium atoms as it measures the beta particles emitted during the normal beta-decay of the tritium atoms. In order to measure the low beta disintegration energy (average energy 5.9 keV), without any absorption in the carbon host, the entire sample material will first be combusted using an oxidation apparatus proposed by Vance and described elsewhere [16]. The tritium released during the combustion process is oxidized to water and then captured in the downstream water bubblers. Water aliquots were taken from each of the bubblers using calibrated syringes. These aliquots were mixed with scintillation cocktail and analyzed to determine the total tritium concentration using a liquid scintillation. In this way, the measured tritium concentration will give the average T concentration value over the entire mass of the sample and will be expressed in Bq/g. The thickness of the slices determines the depth resolution of FC measurement.

Finally, since both experimental analyzing methods are relative measuring methods they have to use for calibration standard samples.

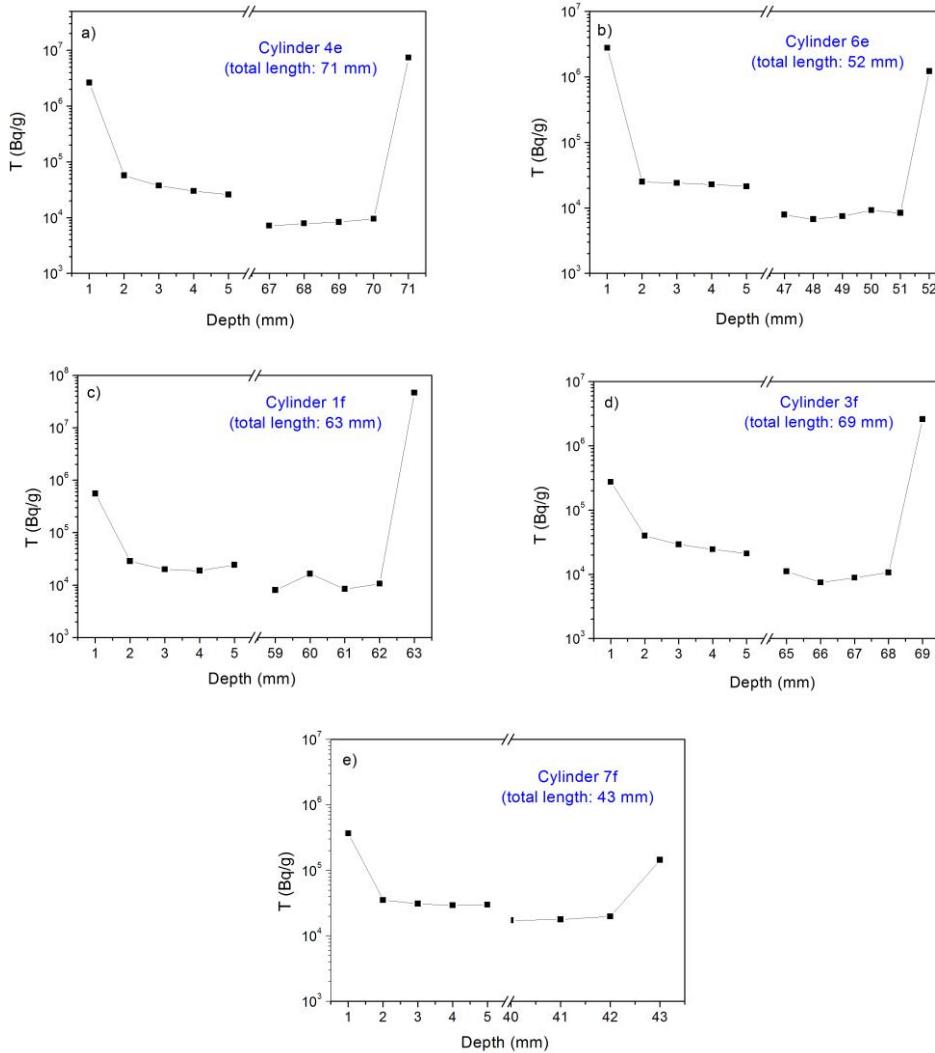


Fig. 2: The depth profiles of tritium accumulation in investigated tile as measured in five drilled different cylinders (1f, 3f, 7f, 4e, 6e). Since the tile has a saddle like shape the height of the drilled cylinders are different, extending between 43 to 71 mm. In each of the spectra the first point was measured from the surface slice and the last point from the backside (the last 1 mm slice).

Fig. 2 (a, b, c, d, e) presents the averaged results of AMS and FC for the integrated depth distribution of tritium retention in five cylinders (4e, 6e, 1f, 3f, 7f) that were drilled from tile 14ING3B of the Joint European Torus (JET)

divertor. The averaging interval of the experimental data was 1 mm. At the end (bottom) of each tile the T concentration distributions show increased tritium retention.

Cylinders 4e and 6e were drilled from the left half-side of the tile and cylinders 1f, 3f and 7f from the right side, as shown in Fig. 1.

To emphasize is that the right tile half surface part was exposed to a tritium removal procedure by laser ablation. However, independent of the amount of removed tritium from the tile surface (92%), all five explored cylinders have shown the same variation trend of their T-depth profile: 1) the maximum of the tritium concentration is at the surface of the tile; 2) then, follows an exponential decrease towards the bulk; 3) inside of the tile the T-concentration remains constant at a low value for more than 30-40 mm; 4) finally, a peaking of the tritium retention occurs in the last 1-2 mm of the tile. This enhanced accumulation of tritium on the backside of the PFCs was clearly determined by both analyzing methods and also it was found in all performed measurements.

Since the plasma-facing surface of the divertor tile has a saddle shape (see Fig. 1) some of the drilled cylinders are longer than others. Measurements showed that the tritium retention on the backside of the divertor is dependent on the distance from the surface. Compared to the average concentration in the bulk the tritium accumulation measured in the backside of long cylinders ($h = 60-75$ mm, 1f, 3f, 4e) is about 3 orders of magnitude higher. However, the shorter cylinders ($h = 40-55$ mm, 7f, 6e) had smaller tritium retention. For example, the thickness of cylinder 7f is only 43 mm and it was drilled from the curved region of the tile, which is in a shadowed location in respect to the plasma flux, with lower tritium input. The measured tritium accumulation in back of the tile was therefore smaller (see 7f in Fig. 2 and in Fig. 3).

In order to compare the retention effect in the different drilled cylinders (different locations) from the protection tile, Fig. 3 shows a plot were all tritium depth profiles were presented on an arbitrary depth scale.

A contamination from the storage in a closed package would have had the effect of an equal tritium accumulation on surface and backside what is not at all the case as shown in Fig. 3. In Fig. 4 values are shown of the tritium concentrations retention in the last back slices of the divertor tile. These values are different by one up to two orders of magnitude.

The tritium removal by laser ablation that was applied to the right half tile had no consequences on the tritium retention or on the amount of the retained tritium in the backside of the tiles. This is due to the fact that the detritiation was performed after the exposure of tiles to the tritium fluxes. For the same reason the bulk values are also similar. The only persuading dependence remains the dependence on the tile width, as could be seen in Fig. 2.

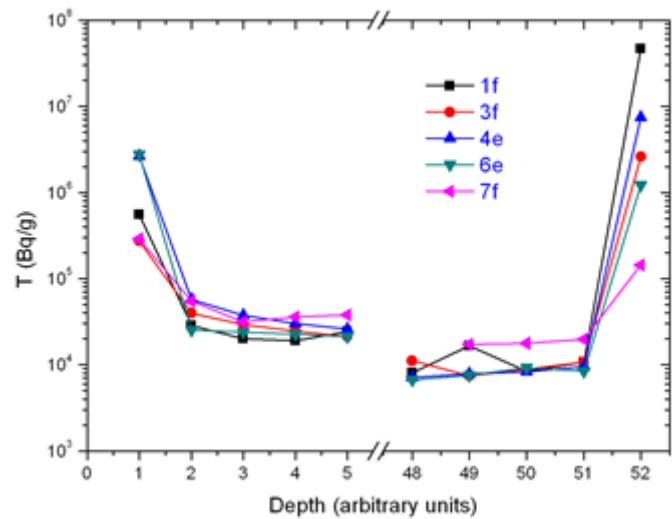


Fig. 3: Comparison of tritium retention distributions in the protection tile.

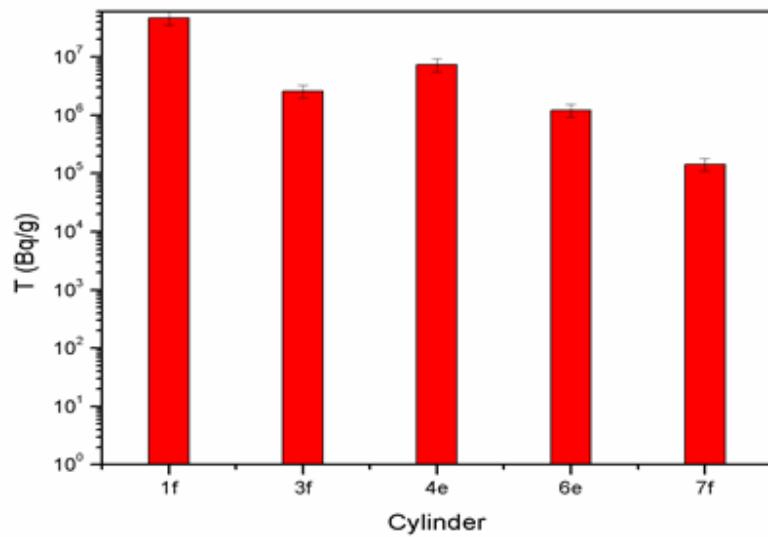


Fig. 4: Comparison of the tritium - concentration end-values, measured in the last mm of the backside of the protection tile.

The intriguing accumulations of tritium on the backside of the JET tiles made us to explore the tritium retention on the sidewalls of the divertor tile, also. Those parts of a tile are even more hidden to the plasma exposure, as is their surface. However, a narrow inter-space remains in-between the tiles and could be tritium contaminated due to the inflow of the fuel exhaust produced by the large vacuum pumps located behind the divertor tiles. To test this assumption two slices were cut (1h and 6h, see Fig. 5a) from the sidewall of the same tile. Then, slices were further cut into 1mm thin layers, noted as layer I and layer II (Fig. 5b).

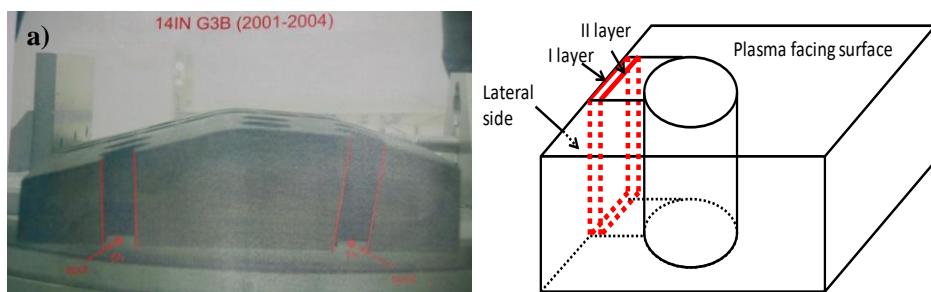
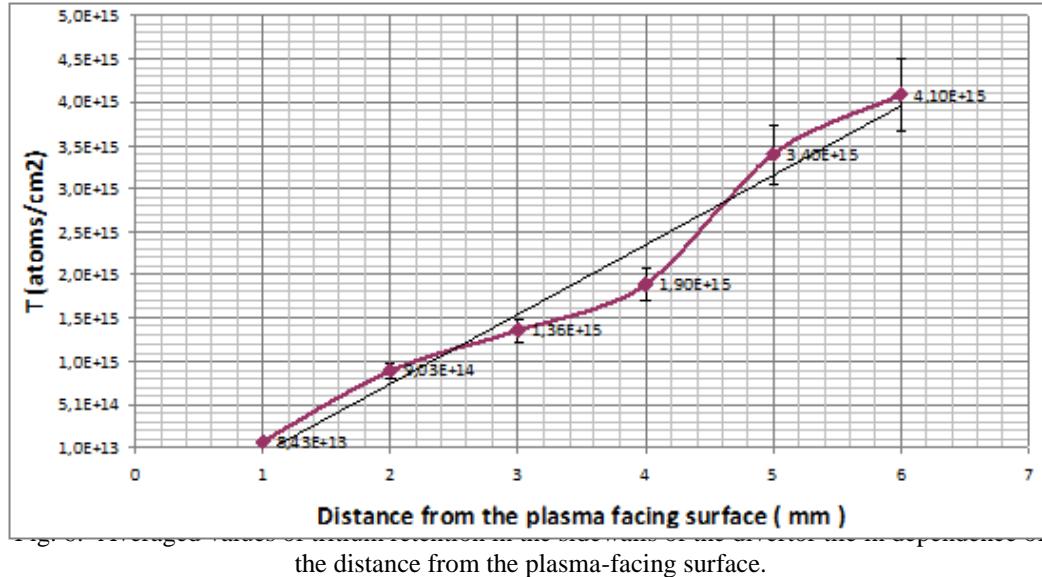


Fig. 5: a) Sample cuts from two locations on the sidewall of test tile.b) Schematic drawing of a side cut from a divertor tile showing the two layers of 1 mm.

Finally, each layer was cut in 6 parallel stripes to the surface and of same dimensions.

If a tritium contamination would exist on the outer of the sidewall this should be revealed by the measurements of the same tritium concentration in all stripes of the first cut layers. However, the tritium concentration content in the stripes was similar for the two positions 1h and 2h. For each stripe the tritium retention values are increasing with the distance from the surface. Therefore, in Fig. 6 only the averaged tritium retention values are shown for the two outer layers cut from the sidewall of the tile. The values are expressed in atoms/cm² and start increasing nearly linear from the plasma-facing surface towards the backside. The difference between the values at ends of the tile is about two orders of magnitude. The steady increasing of tritium retention in the measured strips shows that such an effect is the result of diffusion of a dynamic interaction of the tile side surface with the fuel flow, but has nothing in common with a contamination in a steady regime produced by a constant tritium gas pressure.



the distance from the plasma-facing surface.

The measurements in the second layer showed smaller values of tritium retention, at level of about 104 Bq/g. Such values are similar to concentrations measured for the tritium retention in the bulk of the tile (Fig. 3) and are three orders of magnitude lower than the retention values at the backside of the tile (Fig. 4).

This effect looks to be a general trend of the CFC protection tiles.

3. Conclusions

The tritium retention in CFC protection tiles was experimentally investigated by the FC and by the AMS analyzing methods. Results were used in common for evaluating the experimental data and drawing conclusions. All measured data in this work have shown clearly an enhanced accumulation of tritium on the backside of the divertor tile in comparison to the value in its bulk and on its surface.

The difference between tritium retention in the bulk and in the backside of the tile is about 3 orders of magnitude. Tritium retention was measured for five positions on the backside of tiles. Each retention value was different. Furthermore, the accumulation in the backside showed to be proportional to the thickness of the tile. Measurements have shown that the supplementary heating from the laser ablation had no noticeable effect on the tritium accumulation.

Since all these divertor tiles were made of a porous carbon material (CFC structure) the tritium molecules and in general the molecular hydrogen can

penetrate along open pores reaching the end of the tile width. At temperatures below 500 K tritium becomes trapped in the carbon the structure and accumulation run to saturation in those regions [7]. In fusion reactors the base plate and the shaft supporting the protection tile are water-cooled. Therefore, the low temperature in this region could be the reason of accumulation. If this is true, a simple way to overcome the effect would be to warm up the base plate and to perform an aspiration of the tritium excess in that region.

Since contamination from the storage in a closed package was initially suspected for the retention effect, measurements were also done for the tritium retention in the sidewall of one of the divertor tiles. Results have shown that the retention on the side wall also varies, increasing from the plasma facing side of the tile towards its backside and cannot be attributed to contamination from storage in a closed package or box.

The absorption of the fuel by the vacuum pumps placed behind the system could be responsible for the side contamination. Concentration depth profiling of tritium retention measured on perpendicular direction to edge sides reveal the same retention values in the bulk as measured in the initial measurements performed on a direction perpendicular of the plasma-facing surface.

All the above results converge to the conclusion that carbon CFC protection tiles are exposed to tritium retention/contamination on all sides. However, the backside tritium accumulation is the most dangerous effect and should be avoided by all means.

A highly improving decision was to change the materials used for future production of tiles from carbon or carbon tungsten coated to exclusively use of solid tungsten as will be used at the International Thermonuclear Experimental Reactor (ITER).

R E F E R E N C E S

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