

Surface Analysis for the TFTR Armor Tile Exposed to D-T Plasmas Using Nuclear Technique

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Abstract. Quantitative measurements of hydrogen isotope, lithium isotope and other impurities were performed on the surface of the plasma facing component exposed to D-T plasma in Tokamak Fusion Test Reactor. The sample tile was analyzed with deuteron-induced nuclear reaction analysis, imaging plate, full combustion and gamma-ray measurement methods. The tritium retention on the side surface of the sample tile was 2 times as large as that of the plasma facing surface and its areal distribution increased toward the plasma facing surface. So carbon co-deposition with hydrogen isotopes occurred on tile surfaces without direct plasma contact. The tritium depth profile was different from deuterium one on the side surface, which reflects the history of injected deuterium and tritium fuels in the D-T experiments. The retained T/D was in good agreement with the total T/D retained in torus. The averaged tritium retention in the near surface region was estimated to be 50 % of the whole tritium retention in the bulk. The half amount of tritium remained near the surface and did not diffuse more deeply, which gives a bright prospect for tritium safety. On the other hand, the retained isotope ratio of lithium-6 to whole lithium was 3.1 times as large as the natural isotope ratio. The ratio relates to the injection of 95.6 % enriched lithium-6 pellets to avoid resonant absorption of RF heating power in some campaigns. The lithium retention was the same order of magnitude as the deuterium retention, suggesting that lithium deposited as lithium-deuteride. No impurities such as carbon-14 or radioactive metal nuclei were detected.

1. Introduction

Fuel and impurity particles show complicated behavior on the surface of plasma facing components (PFC) in fusion devices. Its study is required for designs of the fuel recycling, plasma control, etc. Moreover, it is also important to understand tritium behavior in terms of safety management of the tritium inventory under D-T plasma operations.

In the case that carbon-based materials are used as PFC, co-deposited layers of carbon compounds including hydrogen isotopes or other impurities accumulate on PFC surfaces due to plasma-wall interactions [1-6]. The analysis of the PFC surfaces exposed to D-T plasmas is necessary to understand behavior of fuel and impurity particles under the burning plasma.

This paper investigates hydrogen isotope depth profiles, lithium isotope retentions and other impurity concentrations on PFC exposed to D-T plasma in Tokamak Fusion Test Reactor (TFTR), Princeton Plasma Physics Laboratory.

Deuterium-tritium plasma experiments were performed from 1993 to 1997 in TFTR. Operations of TFTR were performed in contact with an inner toroidal bumper limiter. The limiter was divided into 20 bays (labeled A-T) in toroidal direction. Each bay composed of tiles with 4 columns (labeled A-D) in toroidal direction, 24 rows (labeled 1-24) in poloidal direction. Tiles were made of 4-dimensional coarse weave carbon-fiber composite (4D-CFC), 3-dimensional fine weave CFC or isotropic graphite. The outer vacuum vessel was covered with 304 stainless steel and graphite tiles. Co-deposition and flaking were observed at some areas due to the contact of plasma with the bumper limiter [1]. Lithium conditionings on the surface of PFC were performed by the lithium pellet injection for plasma performance improvement [3, 8-10]. After the experiments, air ventilation and a bake out were performed

to clean the PFC surfaces.

2. Experimental Procedure

A 4D-CFC tile placed at K bay, column C, row 16 (KC-16) was used as a sample. The corner of the tile was cut into a piece with the dimension of 15 mm × 20 mm × 10 mm. Three surfaces were planes facing the vacuum and other surfaces were cutting planes.

2.1. Deuteron-induced Nuclear Reaction Analysis Method

Depth profiles of hydrogen and lithium-6 in the TFTR tile exposed to D-T plasmas were measured with deuteron-induced nuclear reaction analysis (deuteron-NRA) method for the first time. The experimental setup is shown in FIG. 1. The deuteron beam produced by the deuteron accelerator in the Fusion Neutronics Source facility, Japan Atomic Energy Agency, was steered to the chamber at the end of the 80° beam line. The deuteron beam through the aperture of 6 mm in diameter was bombarded on the target at an incident angle of 30°. Emitted charged-particles from the target were detected at the detection angle of 90° using a silicon solid state detector (Si-SSD) with depletion layer thickness of 200 μm. The solid angle subtended by the Si-SSD is defined to 1.1×10^{-2} sr by an active area of 25 mm². The energy calibration was corrected based on a ²⁴¹Am alpha source (5.486MeV). A screening foil of aluminum was used to reject scattered deuterons.

The depth resolution of 0.75 μm was obtained under the present setup using proton spectrum of ¹²C(d,p)¹³C reactions in a graphite as a standard sample. Considering this value, depth resolutions of 0.25 μm and 0.50 μm were expected for tritium and deuterium measurements, respectively. Incident deuteron energies of 150, 200, 250, 300 and 350 keV were chosen. Typical incident deuteron fluence was 8.1×10^{19} deuterons/m². An organic scintillator NE213 was also used to monitor neutron emission from the sample.

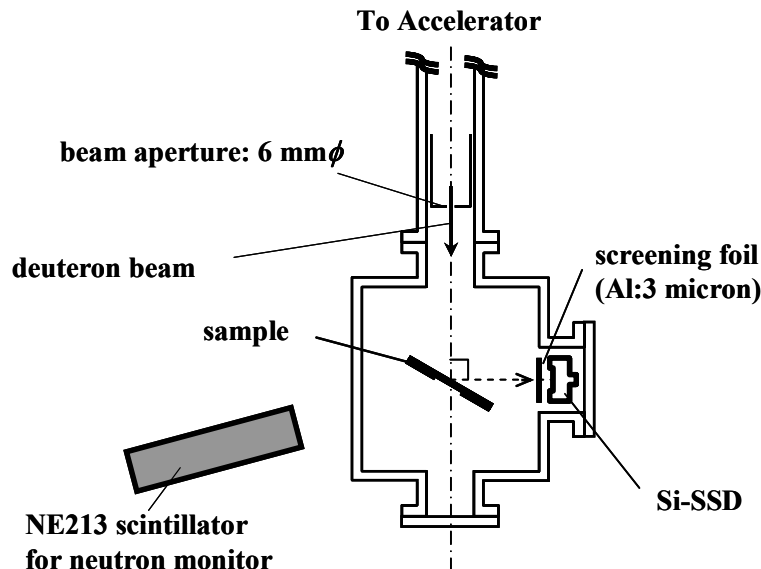


FIG. 1. Schematic view of the experimental setup for deuteron-NRA method.

2.2. Imaging Plate Method and Other Methods for Impurity Retention Measurement

Beta-rays emitted from the sample was recorded to measure tritium areal distribution using an imaging plate (IP) predominantly composed of a BaFBr:Eu photo-stimulable phosphor [11].

Since an IP's surface was covered with a polyethylene terephthalate film of $0.9\ \mu\text{m}$ in thickness to avoid tritium contamination, photo-stimulated luminescence (PSL) intensity corresponded to amount of tritium retained from the surface to $0.7\ \mu\text{m}$ in depth. Note that the contribution of bremsstrahlung X-rays to the PSL intensity was three orders of magnitude less than that of β -rays. The imaging plate was exposed to the radiation from the sample for three minutes. In consideration of an IP's fading characteristic, the tritium image was read from IP in 60 minutes after the exposure. Spatial resolution of the IP reader was set to be $100 \times 100\ \mu\text{m}$.

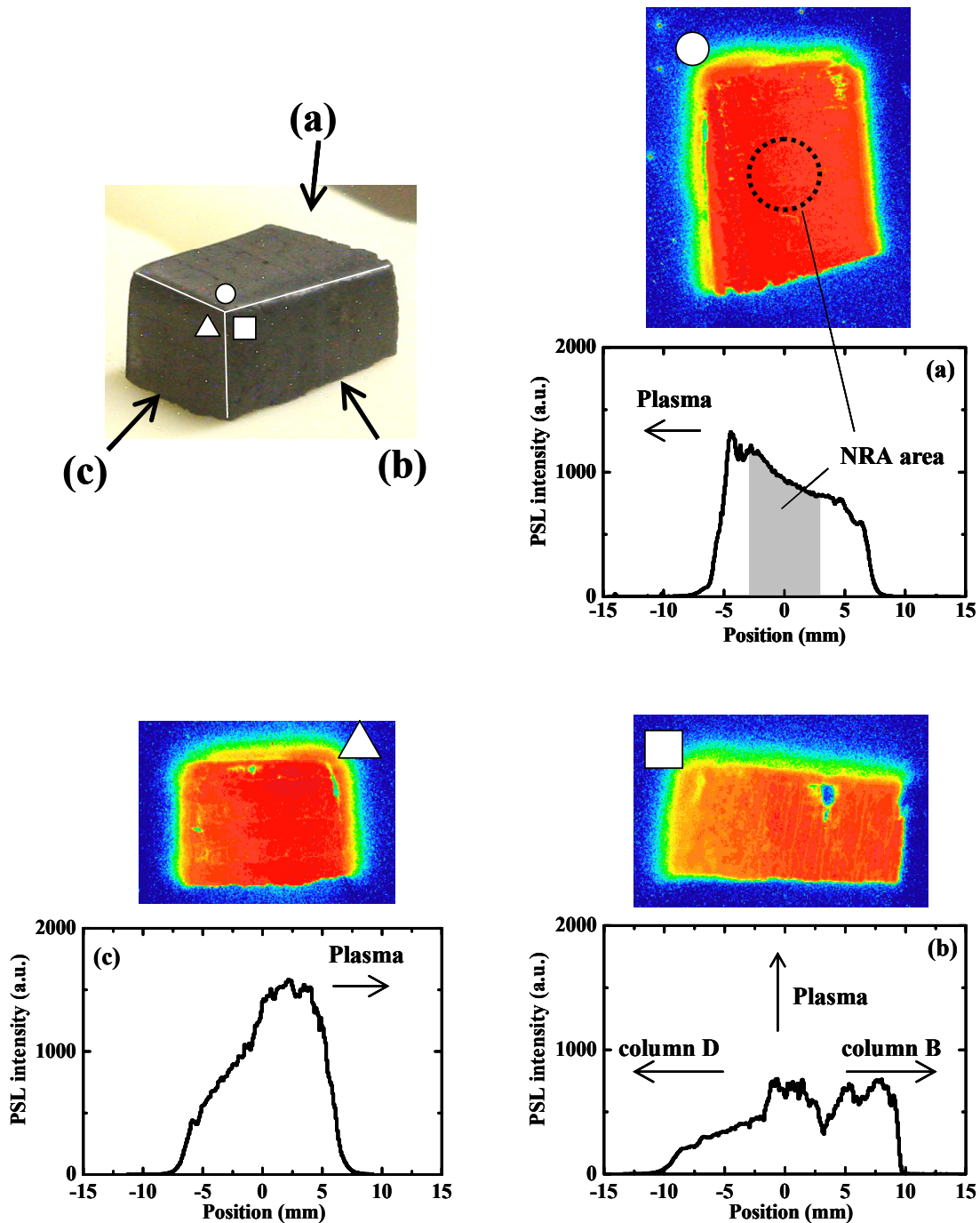


FIG. 2. Photograph of the sample, areal distributions of tritium and PSL intensity profiles on side surfaces (a), (c) and a plasma facing surface (b). Markers of circle, square and triangle in the photograph correspond to those in the areal distributions. Tritium around gray area in (a) was measured by deuteron-NRA method, as is indicated in FIG. 4(a).

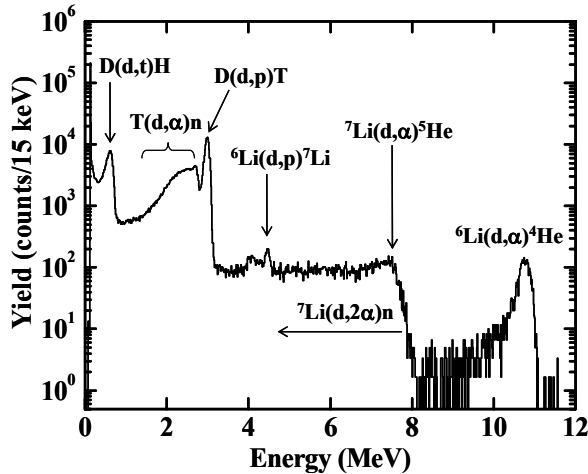


FIG. 3. Typical energy spectrum observed with Si-SSD by means of 350 keV deuteron-NRA. Charged-particles produced by T+d, D+d and Li+d reactions were used to estimate depth profiles.

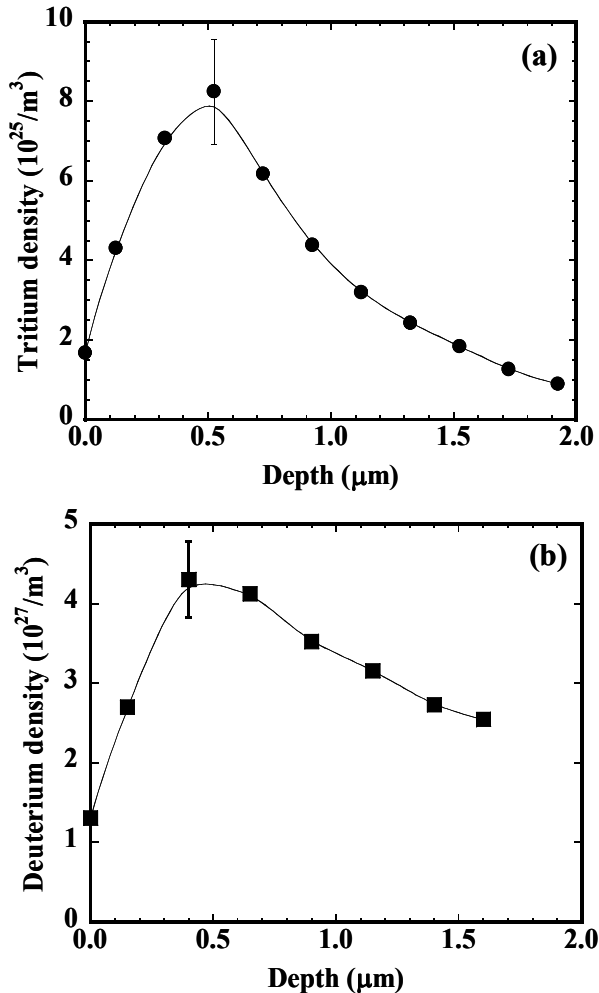


FIG. 4. Depth profiles of (a) tritium and (b) deuterium in the side surface. These profiles were calculated using the T(d,α)n and D(d,t)H spectra. The solid lines are intended as a visual aid.

A high pure germanium detector was prepared to measure gamma-rays from radioactive nuclides originating in metal impurities. The whole tritium and carbon-14 in the sample were measured by a full combustion method.

3. Results and Discussion

3.1 Hydrogen Isotope Retention

Tritium areal distributions on a plasma facing surface and two side surfaces by the IP method are shown in FIG 2. Lateral profiles of the PSL intensity were normalized with the longitudinal width in the figure. The amount of tritium on the both side surfaces increases toward the plasma facing surface. The tritium retention of each side surface is 2 times as large as that of the plasma facing surface. Co-deposition with hydrogen isotopes occurred on tile surfaces without direct plasma contact because tritium retentions of the both side surfaces were larger than that of the plasma facing surface.

Tritium and deuterium depth profiles in the side surface (FIG. 2(a)) with larger tritium retention were measured using deuteron-NRA method. A typical energy spectrum obtained by the 350 keV deuteron-NRA is shown in FIG. 3. Alpha-particles from the T(d,α)n reaction were observed together with tritons and protons from the D(d,p)T reaction predominately. We also identified three small peaks of the ${}^6\text{Li}(d,p){}^7\text{Li}$, ${}^6\text{Li}(d,\alpha){}^4\text{He}$ and ${}^7\text{Li}(d,\alpha){}^5\text{He}$ reactions together with a continuum spectrum of the ${}^7\text{Li}(d,2\alpha)n$ three-body break-up reaction. Associated neutrons of the D+T reaction and neutrons from the D(d,n) ${}^3\text{He}$ reactions were monitored with the NE213 scintillator.

Tritium and deuterium depth profiles in the side surface are shown in FIG. 4(a) and (b), respectively. The profiles were estimated using energy loss and yield of charged-particles produced by the T(d,α)n and D(d,t)H reactions. Broadening on depth profiles caused by the depth resolution in the measurement system was corrected by

unfolding technique with a Gaussian response function. The tritium profile has a peak at $0.5 \mu\text{m}$ with a density of $8.2 \times 10^{25} \text{m}^{-3}$. The deuterium broadly distributed up to a depth of $1.5 \mu\text{m}$ with a peak density of $4.4 \times 10^{27} \text{m}^{-3}$. Both tritium and deuterium densities decrease at the surface as the result of tritium removal treatment. Figure 5 shows a depth profile for the ratio of tritium to deuterium density (T/D). The T/D profile is not constant. Since fuels with a wide range of T/D were introduced to the torus, it is considered that T/D in the sample reflects the plasma operating condition.

In addition, the amounts of the retained tritium and deuterium from the surface to $1.5 \mu\text{m}$ were $7.6 \times 10^{19} \text{m}^{-2}$ and $5.5 \times 10^{21} \text{m}^{-2}$, respectively. The 1.7×10^{23} atoms of tritium and 1.4×10^{25} atoms of deuterium were retained in the torus [9]. The averaged T/D of 0.01 in the sample agreed with the total retained T/D of 0.01 in the torus.

Using the results of both deutron-NRA and imaging plate methods, estimated tritium retentions were $3.4 \times 10^{19} \text{m}^{-2}$, $3.1 \times 10^{19} \text{m}^{-2}$ on both the side surfaces and $1.5 \times 10^{19} \text{m}^{-2}$ on the plasma facing surface. The tritium retention in the bulk was estimated to be $1.6 \times 10^{20} \text{m}^{-2}$ as the result of the full combustion method. It was found that 50 % of the whole tritium remained near surface region and did not diffuse more deeply, which gives a bright prospect for tritium safety.

3.2. Lithium Isotope and Other Impurity Retentions

A lithium-6 depth profile at the side surface of the sample tile was also evaluated from the ${}^6\text{Li}(d,\alpha){}^4\text{He}$ spectra, as is shown in FIG. 6. The profile indicates the maximum density of $2.2 \times 10^{26} \text{m}^{-3}$ around the depth of $1.0 \mu\text{m}$, which is the same tendency as hydrogen isotope in

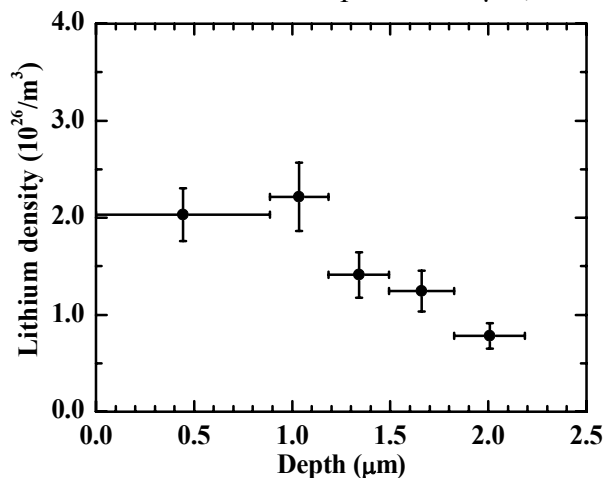


FIG. 6. Depth profile of lithium-6 estimated with ${}^6\text{Li}(d,\alpha){}^4\text{He}$ yields for each incident deuteron energy.

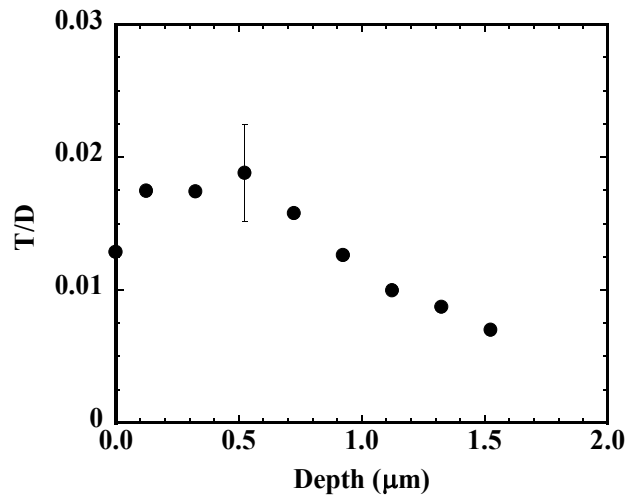


FIG. 5. The ratio of tritium to deuterium as a function of depth. In deeper region the amount of retained tritium was relatively low.

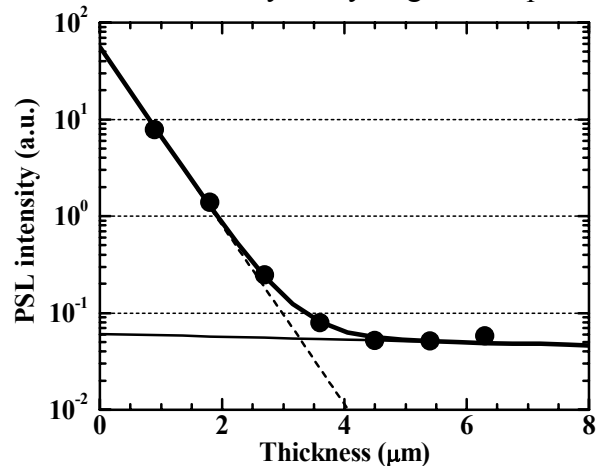


FIG. 7. Photo stimulated luminescence intensity as a function of thickness of polyethylene terephthalate films. The solid, dashed curves are exponential approximation ones. Thick curve is a sum of them.

consideration of a broader depth resolution. Lithium isotope retention was estimated with a ratio of the ${}^6\text{Li}(d,\alpha){}^4\text{He}$ to the ${}^7\text{Li}(d,\alpha){}^5\text{He}$ yields in FIG. 3. The lithium-6 and lithium-7 retentions were $4.1 \times 10^{20} \text{ m}^{-2}$ and $1.4 \times 10^{21} \text{ m}^{-2}$ in the range from the surface to $2.0 \mu\text{m}$. The retained isotope ratio of lithium-6 to whole lithium was 0.232, which is 3.1 times as large as the natural isotope ratio 0.075. The ratio relates to the injection of 95.6 % enriched lithium-6 pellets to avoid resonant absorption of RF heating power in some campaigns [8, 10]. The lithium retention was the same order of magnitude as the deuterium retention, suggesting that lithium deposited as lithium-deuteride. In addition, the retained D/Li ratio of 3.1 implied that hydrogen isotope was retained as not only lithium-hydride but also carbon compounds.

Attenuation of radiation intensity passing through films with different thickness was recorded using IP to identify retained radioactive nuclei other than tritium. Figure 7 shows the PSL intensity as a function of thickness of polyethylene terephthalate films. The intensity with two components, *i.e.* the rapid attenuation component of β -rays and the slow one of X-ray, exponentially decreased with increasing thickness of the film. The β -ray emitter was only tritium in the side surface because other components were not observed in FIG. 7. Although cobalt-56 and cobalt-60 were retained at lower rows of bay K in TFTR [12], no impurities such as carbon-14 or radioactive metal nuclei were detected by the full combustion or gamma-rays measurement methods. The concentration of retained impurities was less than the detection limit (10 ppb) for both measurements in KC-16.

4. Summary

The methods of the deuteron-NRA, IP, full combustion and gamma-rays measurement were applied to surface analysis for the TFTR tile exposed to D-T plasmas to measure hydrogen isotope, lithium isotope and other impurities. A large amount of tritium was retained on the side surfaces of the tile and its depth profile was different from deuterium one. The tritium profile had a peak near surface, whereas deuterium profile became broader. The difference of both absolute densities reflects the history of injected deuterium and tritium fuels in the D-T experiments. Co-deposition with hydrogen isotopes occurred on tile surfaces without direct plasma contact because the tritium retentions of the side surfaces were 2 times as large as that of the plasma facing surface. Thus these profiles resulted from the co-deposition of hydrogen isotopes with eroded carbon under the TFTR plasma operations. In addition, half amount of the whole tritium in the sample was retained near surface region, which gives a bright prospect for tritium safety. The lithium retention was the same order of magnitude as the deuterium retention, suggesting that lithium deposited as lithium-deuteride. Other impurity concentrations were less than sub ppb in the sample.

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References

- [1] SKINNER, C.H., et al., "Studies of tritiated co-deposited layers in TFTR", *J. Nucl. Mater.* **290-293** (2001) 486.
- [2] BEKRIS, N., et al., "Tritium depth profiles in 2D and 4D FC tiles from JET and TFTR", *J. Nucl. Mater.* **313-316** (2003) 501.
- [3] SKINNER, C.H., et al., "Plasma wall interaction and tritium retention in TFTR", *J. Nucl. Mater.* **241-243** (1997) 214.

- [4] SHU, W.M., et al., "Tritium decontamination of TFTR D-T plasma facing components using an ultra violet laser", *Fusion Sci. Technol.* **41** (2002) 690.
- [5] PENZHORN, R.D., et al., "Tritium profiles in tiles from the first wall of fusion machines and techniques for their detritiation", *J. Nucl. Mater.* **279** (2000) 139.
- [6] TANABE, T., et al., "Tritium retention of plasma facing components in tokamaks", *J. Nucl. Mater.* **313-316** (2003) 478.
- [7] OCHIAI, K., et al., "Measurement of deuterium and tritium retentions on the surface of JT-60 divertor tiles by means of nuclear reaction analysis", *J. Nucl. Mater.* **329-333** (2004) 836.
- [8] MANSFIELD, D.K., et al., "Observations concerning the injection of a lithium aerosol into the edge of TFTR discharges", *Nucl. Fusion* **41** (2001) 1823.
- [9] SKINNER, C.H., et al., "Long term tritium trapping in TFTR and JET", 28th EPS Conference on Contr. Fusion and Plasma Phys. ECA(Europhysics Conference Abstracts) **25A** (2001) 1621.
- [10] HOGAN, J.T., et al., "Lithium effects in plasmas", *Nucl. Fusion* **37** (1997) 705.
- [11] SUGIYAMA, K., et al., "Tritium profile in plasma-facing components following D-D operation", *J. Nucl. Mater.* **329-333** (2004) 874.
- [12] SUGIYAMA, K., et al., "Measurement of Tritium Surface Distribution on TFTR Bumper Limiter Tiles", *Phys. Scri.* **T108** (2004) 68.