

# Measurement of deuterium and tritium retentions on the surface of JT-60 divertor tiles by means of nuclear reaction analysis

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## Abstract

It is important to measure retentions and depth profiles of deuterium and tritium on plasma facing materials to understand plasma-wall interactions. The nuclear reaction analysis (NRA) has been established using a deuteron beam with an adjustable energy. By means of the NRA, depth profiles of deuterium and tritium on the surface of JT-60 divertor graphite tiles irradiated with DD burning were obtained. The NRA method showed that the average concentrations of the deuterium retained in the surface of two different were  $1.3 \times 10^{16}$  and  $0.3 \times 10^{16}$  cm<sup>-2</sup>.  $\alpha$ -particles from the T(d,  $\alpha$ )n reaction were measured from a dome unit tile of JT-60U W-shaped divertor and the measured activity of tritium was about 18 kBq/cm<sup>2</sup>. The peak energy of the  $\alpha$  spectrum obtained with the NRA was about 1.2 MeV. This result implies that energetic tritons were implanted in the divertor graphite tile.

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## 1. Introduction

It has been reported that the retention and the depth profile of tritium in the carbon tiles used for the W-shaped divertor in the DD experiments in JT-60U have been measured by means of imaging plates. The energetic triton from DD experiments implants in part of the dome unit tile of the JT-60U W-shaped divertor with energy about 0.5 MeV [1,2]. Analyses of the retentions of the tritium and deuterium are valuable to obtain some information for study of the interactions of fusion plasmas and wall. There are several possible methods for the measurement of retentions using ion beam analysis. Especially, the nuclear reaction analysis (NRA) method is very effective for the simultaneous absolute measurement of the depth profiles of D and T retained in the materials. However, as we have few methods of mea-

surement of D and T retentions and depth profiles on the plasma facing materials by means of NRA, the objective of this paper is to establish a methodology for measurements of the D and T profiles on the plasma facing materials by means of NRA and to verify the methodology with carbon used as the JT-60U divertor.

## 2. Experimental methods

The measurement has been carried out using the deuteron accelerator of the Fusion Neutron Source in Japan Atomic Energy Research Institute (JAERI-FNS) [3]. Fig. 1 shows the schematic view of the experimental set-up for NRA. The probe beam is a deuteron beam and its energy and current density are adjusted between 250 and 350 keV and 0.1–1  $\mu$ A/cm<sup>2</sup>, respectively. In case of carbon samples, the detectable depth is about 1.5  $\mu$ m by 350 keV deuteron [4]. The size of the beam is to 6.5 mm in diameter by an aperture. The number of incident charged particle is directly measured from the samples with a positive bias supply (about 150 V).

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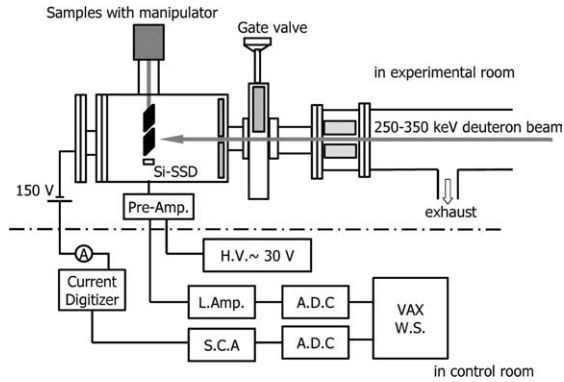


Fig. 1. Schematic view of the experimental set-up for NRA in JAERI-FNS.

Samples were prepared from two JT-60 graphite tiles. The JT-60 samples examined were two carbon tiles labeled as 'A' and 'B' that had been used in the divertor plates of JT-60. The sample A was CFC carbon (CX-2002U) used as the inner baffle plate and the sample B is isotropic carbon (PCC-2S) used as a dome unit tile of JT-60U W-shaped divertor-baffle respectively [2]. They were located and exposed to DD burns, glow discharges and other discharges between June 1996 and November 1998. After all the discharges, the pre-analysis of the samples using liquid scintillation counter (LSC) showed that the tritium concentrations were 3 and 60 kBq/cm<sup>2</sup> for the samples A and B, respectively. The tiles were cut to the size of 20×20×2 mm plates and both set on the movable sample holder in the analyzing chamber at the same time. Moreover, a standard carbon sample, which is clear density, was also used to calibrate the method of the NRA.

A silicon solid state detector (SSD) with a deposition layer thickness of 200 μm, was used to detect high energetic charged particles emitted from the samples. The solid angle for the detection was 1.1×10<sup>-2</sup> sr. The effective area of the detector was covered with a 6 μm thick aluminum foil to suppress scattered deuterons. The incident angle and the detection angle were 45° and 90° respectively. The energy resolution was checked with an α-particle emitter (Am-241: E<sub>α</sub> = 5.486 MeV) and the FWHM was below 40 keV.

D(d,p)T and T(d,α)n are used to measure the deuterium and tritium in the samples. The cross-section data of D(d,p)T and T(d,α)n were referred to the fusion evaluated nuclear data library (FENDL-2) [5,6]. The experimental data evaluated by Maxson and Sornborger were used for the <sup>12</sup>C(d,p)<sup>13</sup>C cross-section and the S-factor [7]. Ziegler's data compilation and the SRIM-code, a Monte-Carlo simulation program were used to estimate the stopping power in the solids [4].

From the yield of each spectrum, retentions and depth profiles can be estimated by the following Eqs. (1)–(3):

$$E_2 = k_{(E,\theta_{\text{out}})} \times \left\{ E_0 - \left( \frac{dE}{dx} \right)_1 \frac{x_1}{\sin \theta_{\text{in}}} \right\} - \left\{ E_1 - \left( \frac{dE}{dx} \right)_2 \frac{x_1}{\sin(\theta_{\text{out}} - \theta_{\text{in}})} \right\} - \left( \frac{dE}{dx} \right)_3 \Delta x_2, \quad (1)$$

$$N_{(x)} = \frac{\int_E^{E+\Delta E} Y dE}{\int_x^{x+\Delta x} \left( \frac{dE}{dx} \right)_1^{-1} dx \cdot \left\langle \frac{d\sigma}{d\Omega} \right\rangle \Delta \Omega \frac{\phi}{\sin \theta_{\text{in}}}}, \quad (2)$$

where E<sub>0</sub>, E<sub>1</sub> and E<sub>2</sub> are incident deuteron energy, emission-particle energy by nuclear reaction at a depth point x<sub>1</sub> and detection energy, (dE/dx)<sub>1</sub>, (dE/dx)<sub>2</sub> and (dE/dx)<sub>3</sub> are stopping powers for each charged particles in the samples and in the Al (6 μm) screening foil, k<sub>(E,θ<sub>out</sub>)</sub> represents kinematics factor taking into account Q-values, x<sub>1</sub> and Δx<sub>2</sub> = depth point from the sample surface and unit depth in the sample taking into account the detector resolution, θ<sub>in</sub> and θ<sub>out</sub> are incident angle and detection angle, N<sub>(x)</sub> is areal density, dσ/dΩ is differential cross-sections, ΔΩ is solid angle (sr) and φ is the number of incident deuterons.

The cross-sections of D(d,p)T and <sup>12</sup>C(d,p)<sup>13</sup>C reactions can be described by the following equation:

$$\sigma_{(E)} = \frac{S_{(E)}}{E} \exp \left( -31.29 Z_1 Z_2 \left( \frac{\mu}{E} \right)^{-\frac{1}{2}} \right), \quad (3)$$

where S<sub>(E)</sub> is astrophysical S-factor in MeV barn, E is incident energy in MeV, Z<sub>1</sub> and Z<sub>2</sub> are atomic numbers of incident and target particles and μ is reduced mass. Especially, the S-factor data of the T(d,α) reaction was used the value of Ref. [6].

### 3. Results and discussion

#### 3.1. Charged particle spectra

Fig. 2 shows the charged particle spectra of sample B and the standard carbon sample. The thick line and the narrow line show the spectra of the sample B irradiated with 350 keV and 250 keV deuterons, respectively. The dotted line shows the spectrum of the standard carbon sample at 350 keV d-beam energy. The number of incident deuterons was used to normalize these yields. The peak at about 2.95 MeV arises from protons from D(d,p)T reaction and the 2.66 MeV peak arises from protons by the <sup>12</sup>C(d,p)<sup>13</sup>C reaction. The triton, associated with charged particle produced by D(d,p)T reaction, causes a peak at about 0.58 MeV. Each peak has tails that

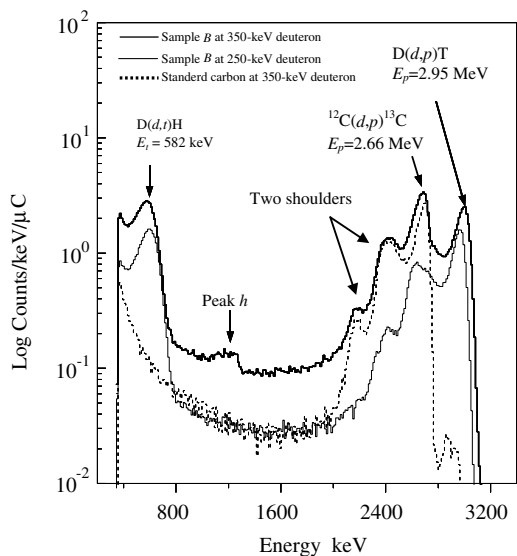


Fig. 2. Charged particles spectra of sample B and the standard carbon sample. Heavy line and narrow one are spectra of sample B by 350 and 250 keV deuteron bombardments respectively. Dotted line is the spectrum of the standard carbon sample by 350 keV.

reflect depth profiles of the deuterium and carbon in sample B. Moreover, two shoulders between the  $^{12}\text{C}(d,p)^{13}\text{C}$  proton and continuous peak (peak h) near 1.2 MeV appeared in all the samples and the standard carbon. The origins of the shoulders are not yet identified. They could arise from protons scattered at the edge of the detector. A possible improvement of the method would be an aperture placed in front of the detector with a diameter smaller than the sensitivity area of the detector.

Peak h appeared only in the sample B irradiated with the 350 keV deuteron. The only candidate reactions which could produce this peak are  $^{16}\text{O}(d,p)^{17}\text{O}$  ( $E_p = 1.3$  MeV) and  $\text{T}(d,\alpha)n$ . However, the  $^{16}\text{O}(d,p)^{17}\text{O}$  reaction can emit the proton at 2 energies ( $E_p = 1.3$  and 2.0 MeV) depending on whether it is accompanied by a  $\gamma$  emission or not. But since we observe only one peak, we think it is due to  $\text{T}(d,\alpha)$ .

### 3.2. Deuterium

From Eqs. (1) and (2), the depth profiles of deuterium in the samples were estimated. Figs. 3 and 4 show the depth profiles of deuterium in the samples A and B respectively. The left vertical axes are the areal carbon densities ( $N_{\text{C}(x)}$ ) measured by  $^{12}\text{C}(d,p)^{13}\text{C}$  reaction. The right vertical axes are the ratio ( $N_{\text{D}(x)}/N_{\text{C}(x)}$ ) of areal densities of deuterium measured by  $\text{D}(d,p)\text{T}$  reaction to the areal density of the standard carbon or the graphite of the samples. The each ratio of D depth profile in Fig. 3 is based on the areal density of the standard carbon.

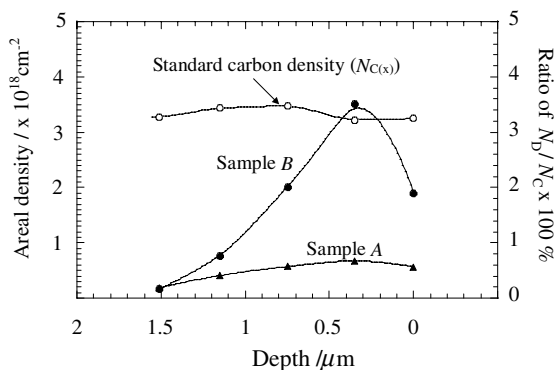


Fig. 3. The areal density of standard carbon and the ratio of D areal densities to the areal density of standard carbon in the each sample. Open circle shows the areal density of standard carbon and closed triangle and circle show the ratio to sample A and B respectively.

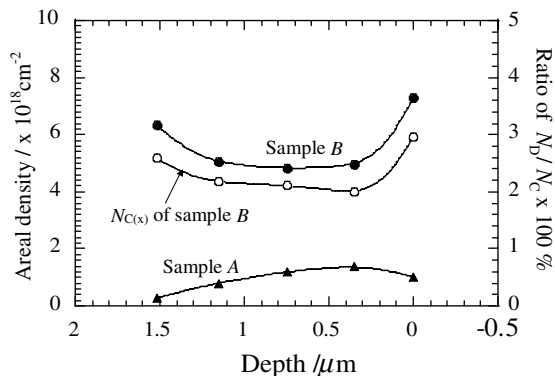


Fig. 4. The areal density of carbon in a sample and the ratio of D areal densities to the areal density of carbon in the each sample. Open circle shows the areal density of the graphite in sample B and closed triangle and circle show the D areal densities to the areal the graphite in sample A and B respectively.

The areal density of the standard carbon gives the constant profile as a function of depth and its average value to total retention was about  $3.7 \times 10^{18} \text{ cm}^{-2}$ . It was found in Fig. 3 that the sample B has a maximum D retention at the surface ( $\sim 0.5 \mu\text{m}$ ), which was about  $1.3 \times 10^{16} \text{ cm}^{-2}$ . The distribution of the sample A was relatively uniform and its average retention was about  $0.3 \times 10^{16} \text{ cm}^{-2}$ . Their tendencies of the D depth profile were in good agreement with the previous measurement by means of a secondary ion mass spectroscopy [8].

The each ratio of D depth profiles in the sample A and B in Fig. 4 is based on the areal densities of the graphite of the sample A and B, respectively. Since the C depth distribution in the sample A showed the same constant profile, the tendency of D depth profile in the sample A was similar to the result in Fig. 3. However, the D depth

profile in the sample B had a different tendency, compared with the result in Fig. 3. Also it was found that the C depth profile of the graphite in the sample B is different from the standard carbon case. Moreover, the D depth profile in the sample B appeared the same tendency of the C depth profile of the graphite. From the result, it could be suggested that the difference of each D depth profile of the sample B in Figs. 3 and 4 were caused by the unclear chemical condition on the surface of the sample B made by the DD burning. It has been reported that a re-deposition layer of 60  $\mu\text{m}$  thickness on the dome top divertor was measured by means of a scanning electron microscopy [9]. From the report, it is considered that the uncertainty of the carbon density in such an instoichiometric re-deposition layer made the stopping power be unclear and caused the results of the anomalous C depth profile and D depth profile.

### 3.3. Tritium

As mentioned the above, the peak h is thought to be due to  $\alpha$ -particles emitted from the T(d, $\alpha$ )n reaction. However, if there is tritium on the surface of the sample, we should obtain peak h at 2.1 MeV. There is an energy difference between 2.1 MeV and the measured energy (1.2 MeV). We think that there is a localized tritium distribution in deeper regions rather than the surface. We calculated the radioactivity of retained tritium from the yield of h and the formula (4).

$$A = \frac{Y_h}{\frac{d\sigma}{d\Omega} \Delta\Omega \frac{\phi_d}{\sin\theta_{in}}} \frac{\ln 2}{T}, \quad (4)$$

where A and  $Y_h$  are Bq/cm<sup>2</sup> of tritium and the net yield of peak h and T is tritium half-life ( $3.89 \times 10^8$  s). The used value of the DT cross-section was 0.41 barn/sr at 109 keV. Calculated value was about 18 kBq/cm<sup>2</sup>. As compared with preliminary measurements by means of LSC, the value obtained by NRA was smaller than one third of the LSC value (60 kBq/cm<sup>2</sup>). The underestimated value and no peak in the sample B at 250 keV deuteron NRA (see Fig. 2) could support few tritium in 1.5  $\mu\text{m}$  depth from the surface of the sample B and the existence of a deeper tritium distribution, which has been referred by Ref. [1].

As a future work, we should consider the increasing of the incident energy of deuteron beam to investigate the tritium density in the deeper region.

## 4. Conclusion

We have established the method of NRA to obtain depth profiles of D and T retentions in JT-60U carbon tiles. Our results of measurement showed that the sample B has a maximum D retention at the surface ( $\sim 0.5 \mu\text{m}$ ) and the maximum retention was about  $1.3 \times 10^{16} \text{ cm}^{-2}$ . The distribution of the sample A was relatively uniform and its average retention was about  $0.3 \times 10^{16} \text{ cm}^{-2}$ , respectively. And also, it is assumed that the instoichiometric re-deposition layer on the surface of the sample B caused the result of the anomalous C depth profile and D depth profile.

It was found that tritium in the sample B predicts is accumulated in deeper area. From this result, it is concluded that the energetic tritons from DD experiments implants the part of the dome unit tile of JT-60U W-shaped divertor with keeping its energy about 0.5 MeV.

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