



Non-destructive tritium measurements of Mk IIA divertor tile by BIXS

M. Matsuyama ^{a,*}, N. Bekris ^b, M. Glugla ^b, N. Noda ^c,
V. Philipps ^d, K. Watanabe ^a

^a *Hydrogen Isotope Research Center, Toyama University, Gofuku 3190, Toyama 930-8555, Japan*

^b *Tritium Laboratory, Forschungszentrum Karlsruhe, D-76021 Karlsruhe, Germany*

^c *National Institute for Fusion Science, Oroshi-cho, Toki-shi, Gifu 509-5292, Japan*

^d *Institute for Plasma Physics, Forschungszentrum Jülich, Association EURATOM, Trilateral Euregio Cluster (TEC), 52425 Jülich, Germany*

Abstract

β -ray-induced X-ray spectrometry was applied to determine the amounts and depth profiles of tritium captured by graphite tiles used in the PTE1 campaign and by carbon fiber composite tiles in the DTE1 campaign of JET. Although the X-ray spectra observed for both carbon tiles consisted of characteristic and bremsstrahlung X-rays, the intensities and shapes of the X-ray spectra were quite different between these tiles. The analyses of the X-ray spectra showed that the amount of tritium retained in the latter tile was on average 15 500 times greater than that in the former tile, and the tritium depth profile extended to 60 μm for the latter tile compared to 5 μm for the former tile. In addition, characteristic X-rays corresponding to Cl, Cr, Fe, Ni and Mo appeared in the latter tile, where Ni showed the highest concentration, indicating that considerable erosion and deposition of vacuum vessel materials took place in the DTE1 campaign.

© 2003 Elsevier Science B.V. All rights reserved.

PACS: 52.40.Hf

Keywords: Amount and depth profile of tritium; Metallic impurities; Non-destructive measurement; β -ray-induced X-ray spectrometry; JET divertor tiles

1. Introduction

Non-destructive and quantitative measurements of the amount and depth profile of tritium retained on/in plasma-facing materials (PFMs) of magnetic fusion devices are of great importance to ensure safety for maintenance work and waste processing of the PFMs containing high level tritium. In addition, these data are indispensable to estimate fuel particle recycling on the surface of PFMs. From these viewpoints, a variety of techniques have been applied so far to measure hydro-

gen isotopes retained on/in the PFMs: for example, a nuclear reaction analysis [1], a solid state charged particle detector [2], an accelerator mass spectrometer [3], a full-combustion method [4], an imaging method [5], a charged-coupled device camera [6], and so on were employed for this purpose.

Recently, a new tritium measuring method was applied to assay one of ALT-II belt limiter tiles exposed to D-plasmas in TEXTOR [7]. The new method is called β -ray-induced X-ray spectrometry (BIXS) [8,9]. By this method, a trace amount of tritium produced by D–D fusion reactions could be quantitatively evaluated from the observed X-ray spectra, and it was shown that the amounts and depth profiles of tritium were different from spot to spot on the tile surface. From these observations, it was concluded that BIXS is applicable to

* Corresponding author. Tel.: +81-76 445 6926; fax: +81-76 445 6931.

E-mail address: masao@hrc.toyama-u.ac.jp (M. Matsuyama).

evaluation of PFMs of working machines and is a promising method for non-destructive measurements of tritium retained on/in materials.

The purpose of the present study is to examine the feasibility of BIXS for the measurements of areal concentration and near surface depth profile of tritium retained at high levels on carbon tiles exposed to D–T plasmas in JET.

2. Experimental

2.1. Carbon tiles for measurements

Two kinds of JET carbon tiles were examined in this study: (i) a graphite tile (004/2–20) exposed to tritium plasmas during the PTE1 campaign and (ii) a 2-D carbon fiber composite (CFC) tile (HZ-DTE1/1BN7) used in the Mk IIA divertor during the DTE1 campaign. For the 004/2–20 tile tritium depth profiles have been already determined using a full-combustion method by Penzhorn et al. The total amount of tritium was evaluated to be 13.8 ± 3.5 kBq/cm², and about 99% of the total tritium was retained within the region of 1 mm beneath the surface [10]. On the other hand, the HZ-DTE1/1BN7 tile contained high level tritium [11], and a front view of the tile is shown by picture (A) in Fig. 1.

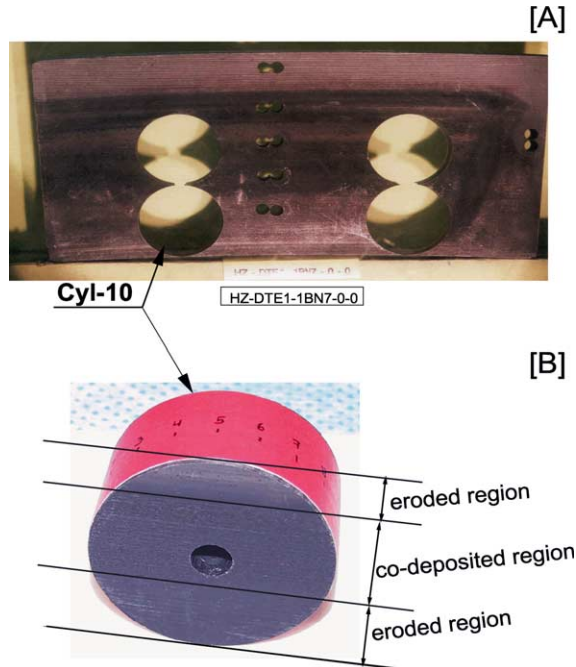


Fig. 1. Photographs of (A) the plasma-facing surface of the divertor tile (HZ-DTE1/1BN7) and (B) the cylindrical sample (Cyl-10) prepared from the tile. Sizes of the tile and the Cyl-10 sample were $(359\text{--}381) \times 181$ mm and 58.7 mm $\varnothing \times 32$ mm, respectively.

This tile was one of the outer divertor tiles in the Mk IIA configuration, and the actual position is described elsewhere [11,12]. Picture (B) shows a cylindrical sample (described as Cyl-10: 58.7 mm in diameter, and 32 mm in thickness) cored out from the HZ-DTE1/1BN7 tile. This cylinder sample was used for tritium measurements by the present technique. The plasma-facing surface of the Cyl-10 sample contained both eroded and co-deposited regions as shown in Fig. 1(B).

2.2. Measurements of the β -ray-induced X-ray spectra

Measurements of β -ray-induced X-ray spectra were carried out using a portable X-ray measuring device at Tritium Laboratory in Karlsruhe (TLK). The X-ray device was equipped with a high purity Ge detector with an ultra-thin beryllium window for efficient detection of low energy X-rays below 18.6 keV. Both the measuring sample and the front surface of the Ge detector were put into a polyethylene bag, and then the plasma-facing surface of the sample was placed opposite to the Ge detector at a distance of 4 mm. The bag was filled with argon gas, used as a working gas to convert β -rays emitted from surface layers of the sample to characteristic X-rays of argon. The flow rate of argon was controlled 1.2–1.3 l/min during measurement. Prior to the first measurement, the background spectrum of the X-ray detector at TLK was measured for 15 h to assess the contamination level of the detector after the measurements. The X-rays from the graphite tile 004/2–20 were accumulated for 20 h on account of its low tritium activity, while for the Cyl-10 sample the accumulation period was set to only 1 h.

For comparison with the reported value [10], measurements of the graphite tile 004/2–20 were conducted at first. Subsequently, nine spots along the circumference on the plasma-facing surface of the Cyl-10 sample were measured to examine the effects of erosion and co-deposition. The tritium depth profiles in both graphite and CFC tiles were estimated by analyzing the observed X-ray spectra with the aid of computer simulation.

3. Results and discussion

3.1. Example of the observed X-ray spectra

Fig. 2 shows an example of the X-ray spectra observed for the graphite tile 004/2–20. The closed circles represent the background level obtained just before the tritium measurement. Two kinds of X-ray peak were observed for the 004/2–20 tile: one is a sharp intense peak of Ar($K\alpha$) X-rays and the other is a considerably weak spectrum of bremsstrahlung X-rays. The intensity of the former peak gives information about the amount of surface tritium, while shape of the latter one gives

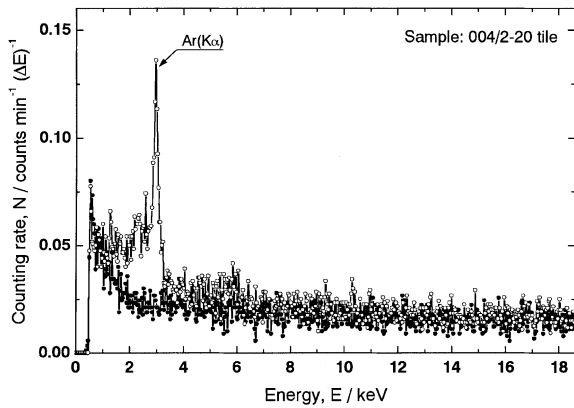


Fig. 2. Example of the X-ray spectra observed for the graphite tile 004/2–20 (open circle) and the background spectrum (closed circle).

information about the depth profile of tritium in the bulk. The X-ray intensity of the former peak was integrated over the spectral features and it was determined to be 0.50 counts/min, which corresponds to the surface activity of 1.3 kBq/cm² [10,13]. This activity was 10–20 times greater than that evaluated for the ALT-II belt limiter tiles exposed to D-plasmas in TEXTOR [7]. On the other hand, the integrated intensity of the bremsstrahlung X-ray spectrum was 3.55 counts/min, which was 30–90 times greater than that of bremsstrahlung X-rays observed for the ALT-II limiter tiles. The difference in the X-ray intensities is mainly due to the different composition of fuel particles between two fusion experimental devices, TEXTOR and JET. In addition, the different increasing ratio of the characteristic X-ray intensity to the bremsstrahlung X-ray intensity indicates a distinct depth profile of tritium in the tiles. In the present measuring technique, the detectable depth of tritium is estimated to be about 1 mm from attenuation of the bremsstrahlung X-rays in carbon materials.

An example of the X-ray spectra observed for the Cyl-10 sample is shown in Fig. 3. The spectral intensity was quite different from that shown in Fig. 2: the intensities of Ar(K α) and bremsstrahlung X-rays significantly increased by about 5000 and 9000 times, respectively. From the intensity of Ar(K α), the amount of surface tritium was estimated to be 6.3 MBq/cm². Furthermore, new characteristic X-ray peaks appeared along with the Ar(K α) X-rays, which were ascribed to Cl, Cr, Fe, Ni and Mo. However, no disturbance in the measurements by activated products was observed in the energy region of interest (0–18.6 keV), in spite of the accumulation of metallic impurities.

The spectra observed for Cyl-10 sample mainly consisted of a sharp intense peak of Ar(K α) X-rays and a considerably large broad spectrum of bremsstrahlung X-rays. Although intensities of the Ar(K α) and bremsstrahlung X-rays slightly differed from spot to spot on

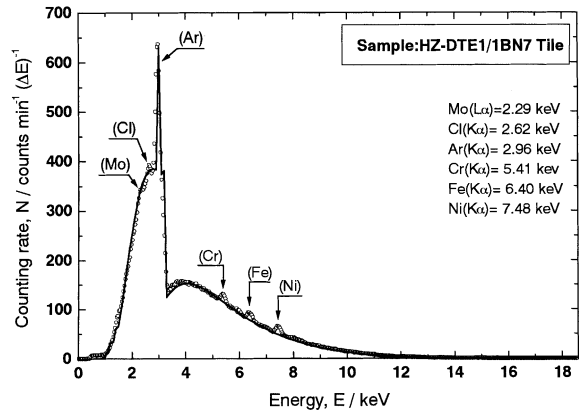


Fig. 3. Example of the X-ray spectra observed for the CFC tile 1BN7: open circles and the solid line represent the observed and simulation spectra, respectively.

the plasma-exposed surface, shape of the X-ray spectra observed for the nine spots was almost same. Both the intensity and shape indicate that a large amount of tritium is retained not only on the surface but also in the bulk of the Cyl-10 sample. In addition, the presence of metallic impurities suggests that surface components of vacuum vessel materials were eroded by the plasma and they deposited on the surface of the outer divertor tiles in the duration of the D–T fusion experiments.

3.2. Depth profiles of tritium in both carbon tiles

To estimate depth profiles of tritium, the observed X-ray spectra were analyzed by means of computer simulation. The tritium depth profiles were basically deduced by matching simulated spectra derived for various depth profiles to the observed spectra. In this simulation, it was assumed that the surface of a sample is perfectly flat, although the real surface of the tile is rough owing to erosion by the plasma. The detailed procedures of the simulation are described elsewhere [8]. The comparison between the simulation and observed spectra is illustrated in Fig. 3. The excellent agreement between the two spectra could only be obtained by assuming a particular depth profile of tritium. This indicates that the depth profile of tritium in a sample can be uniquely determined by computer simulation.

The tritium depth profiles determined by computer simulation for both the present carbon samples were shown in Fig. 4. The depth profiles were considerably different between the two carbon samples: namely, for the graphite tile 004/2–20 most of the tritium was retained within a surface layer of 5 μ m, while for the Cyl-10 sample the depth profile extended to 60 μ m from the surface. For both carbon tiles, it was seen that a fairly large amount of tritium is retained near surface region. This result consists with the data reported by Penzhorn

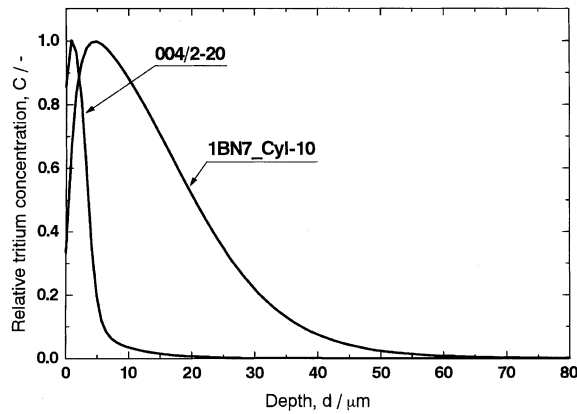


Fig. 4. Tritium depth profiles determined for both graphite and CFC tiles by computer simulation.

et al. [11]. The difference of these depth profiles can be attributed to difference in the structure and temperature of the carbon tiles, and operating conditions of the fusion device. The total amount of tritium within the detection depth of the Cyl-10 sample was estimated to be 214 MBq/cm² [10,13], taking into account the intensity of bremsstrahlung X-rays and the geometric area of the depth profile. It was found that the tritium inventory in the Cyl-10 sample was 15 500 times greater than that in the graphite tile used for the PTE1 campaign.

3.3. Tritium distribution on the surface and in bulk of the Cyl-10 sample

The plasma-facing surface of the Cyl-10 sample contained both erosion and co-deposition regions as shown in Fig. 1. To examine the tritium distribution on the sample surface, nine spots along the circumference on the plasma-facing surface were measured. The local profiles of Ar(K α) and bremsstrahlung X-ray intensities are shown in Fig. 5. The spot numbers in the abscissa correspond to the following regions of the sample surface: the numbers 1, 7, 9, 15, and 17 are in the eroded region, and the others in the co-deposited one. The intensity distribution of Ar(K α) and bremsstrahlung X-rays was almost uniform. This indicates that there is no significant spatial variation in the amount of tritium retained on the surface and in the bulk of the Cyl-10 sample, although the erosion and co-deposition regions were observed on the plasma-facing surface of the Cyl-10 sample.

3.4. Distribution of impurities on the surface of the Cyl-10 sample

The BIXS spectra for the Cyl-10 sample indicated the presence of metallic impurities on the surface as shown in Fig. 3. Impurities were assigned to Cl, Cr, Fe, Ni, and Mo. The deposition of these impurities except Cl should

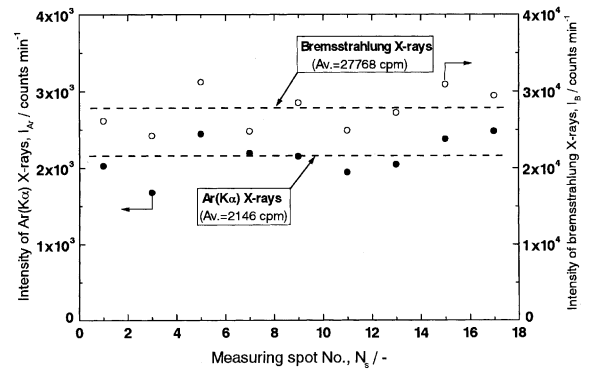


Fig. 5. Local distribution of Ar(K α) and bremsstrahlung X-ray intensities on the surface of Cyl-10.

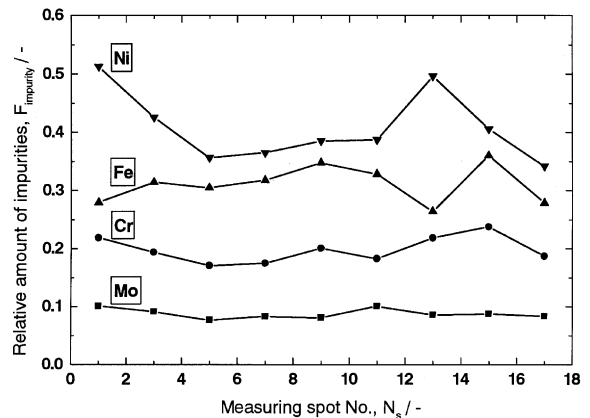


Fig. 6. Local distribution of metallic impurities deposited on the Cyl-10 sample.

be due to the erosion of vacuum vessel materials by plasma impact and their deposition on the outer divertor tile in the duration of D–T fusion experiments. The distribution of the relative amount of those impurities is shown in Fig. 6 except Cl. Among those impurities, nickel was the most dominant species. This implies that vacuum vessel materials consist of a Ni-based alloy contained Mo. Furthermore, although the distribution of Ni and Fe appears to be anticorrelated each other, the reason is not clear and further investigations are required. The presence of such transition metals on the PFMs affects the retention and reemission behavior of tritium [14,15].

4. Summary

BIXS was applied to evaluate tritium inventory in two kinds of carbon tiles: one was a graphite tile (004/2–20) exposed to tritium-containing plasma in the PTE1 campaign, and the other was a cylindrical sample (Cyl-10) cored out from the Mk IIA divertor tile (DTE1/1BN7) used in the DTE1 campaign in JET. For both

carbon tiles, the observed X-ray spectra consisted of characteristic and bremsstrahlung X-rays, but the intensities as well as shape of the spectra were quite different. From analyses of the X-ray spectra, the amount of tritium retained in the Cyl-10 sample was determined as 214 MBq/cm², which was 15 500 times greater than that for the 004/2–20 tile. The difference in the shape of the X-ray spectra clearly suggested that the tritium depth profile extended to 5 µm for the PTE1 tile compared to 60 µm for the DTE1 tile. In addition, additional characteristic X-rays were observed for the DTE1 tile, indicating accumulation of several kinds of metallic impurities on the surface. Those impurities were assigned to Cl, Cr, Fe, Ni, and Mo. This suggests erosion of vacuum vessel materials by plasma particles and their deposition on the surface. Furthermore, no increase in the background level after all the measurements was observed, indicating contamination of the detector surface was negligibly small even after non-destructive measurements of the tiles containing high level tritium.

Acknowledgement

The authors wish to thank staff of the Hot Cells in Forschungszentrum Karlsruhe for preparation of the cylindrical CFC sample.

References

- [1] W.R. Wampler et al., *J. Vac. Sci. Technol. A* 6 (1988) 2111.
- [2] W.R. Wampler, B.L. Doyle, *Nucl. Instrum. and Meth.* 349 (1994) 473.
- [3] M. Friedrich et al., *Nucl. Instrum. and Meth. B* 161–163 (2000) 216.
- [4] R.-D. Penzhorn et al., *Fusion Eng. Des.* 49&50 (2000) 753.
- [5] K. Miyasaka et al., *J. Nucl. Mater.* 290–293 (2001) 448.
- [6] C.A. Gentile, J.J. Parker, S.J. Zweben, *Fusion Sci. Technol.* 41 (2001) 551.
- [7] M. Matsuyama et al., *J. Nucl. Mater.* 290–293 (2001) 437.
- [8] M. Matsuyama, K. Watanabe, K. Hasegawa, *Fusion Eng. Des.* 39&40 (1998) 929.
- [9] M. Matsuyama, S. Ueda, K. Watanabe, *Fusion Eng. Des.* 49&50 (2000) 885.
- [10] R.-D. Penzhorn et al., *J. Nucl. Mater.* 279 (2000) 139.
- [11] J.P. Coad et al., *J. Nucl. Mater.* 290–293 (2001) 224.
- [12] R.-D. Penzhorn et al., *J. Nucl. Mater.* 288 (2001) 170.
- [13] M. Matsuyama, T. Murai, K. Watanabe, *Fusion Sci. Technol.* 41 (2002) 505.
- [14] K. Ashida, K. Ichimura, K. Watanabe, *J. Vac. Soc. Jpn.* 30 (1987) 294.
- [15] K. Ashida, K. Ichimura, M. Matsuyama, K. Watanabe, *J. Nucl. Mater.* 148 (1987) 217.