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Tritium uptake in graphite tiles exposed to EAST plasma and then tritium gas

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ABSTRACT

Tritium exposure experiments were carried out for three kinds of EAST SiC coated doped-graphite (SiC/C) samples, one from the original graphite tiles without being irradiated, and the other two from erosion and deposition areas of first wall after the 2009 campaign in EAST. β-ray-induced X-ray spectrometry (BIXS) was used to characterize the exposed samples. It is showed that the significant amount of tritium was absorbed in the surface of deposition sample in comparison with that of original sample, which was also supported by the results of imaging plate (IP) measurements. In addition, it was found that drastic decrease in tritium retention appeared by lowering exposure temperature, and the trapped tritium was maintained stably with time. Computer simulation is used to analyze the details of depth profile of tritium in different kinds of samples.

1. Introduction

EAST was operating with the SiC/C tiles as the sole plasma facing material (PFM) until 2011, and as divertor PFM for next several years [1,2]. Before W/Cu-PFMs being used in EAST, PWI studies on the SiC/C-PFM are very important especially for long pulse discharge and semi-detachment experiments. And the experiences on SiC will give great evidence for PWI on W-PFM. In our preliminary work, some basic properties of SiC coatings and behavior with exposure to HT-7 plasmas have been investigated [3,4], and the adsorption/retention of hydrogen isotopes had been investigated by β-ray-induced X-ray spectrometry (BIXS) comparing with doped graphite and W coatings fabricated by vacuum plasma spraying [5]. To better understand the behavior of the SiC/C-PFMs in different erosion/deposition areas in EAST, it is of great importance to accumulate the basic data such as the amount of adsorption and depth profiles of tritium in the PFMs. Therefore, we exposed SiC/C samples including original surface (un-irradiated), erosion surface and deposition surface (exposed to plasma in the erosion and deposition areas of EAST) to tritium gas, and then made use of a BIXS and an imaging plate (IP) method to measure the relative characterization. Simulation is also used to analyze the depth profile of tritium absorbed in the samples.

2. Experimental

2.1. Samples for measurements

Three kinds of samples were prepared for this study: (i) original surface (Ori), (ii) erosion surface (Ero) and (iii) deposition surface (Dep). The first kind was cut from the surface of SiC/C tiles without the plasma exposure. The graphite tiles are produced at the Institute of Coal Chemistry, Chinese Academy of Science (ICCCAS). The bulk is doped graphite (GBST1308, 1%B, 2.5%Si, 7.5%Ti) and the SiC coated plasma-facing surface is fabricated by chemical vapor infiltration (CVI). The latter two were SiC/C samples irradiated at the bottom of the outer target and dome tile near low field side (LFS) of EAST, represented erosion-dominated and deposition-dominated areas, respectively.

All of the samples (3 pieces for each kind) were cut into a thickness of 3 mm using a diamond line cutter with a feed speed of 1.5 mm/min and linear velocity of 1.16 m/s. For each kind of the samples, two pieces were cut into 15 mm × 15 mm and the other one was cut into 15 mm × 20 mm for different exposure conditions. All of the samples were rinsed in acetone, and then were evacuated thoroughly below 1×10^{-4} Pa at room temperature by a turbo molecular pump. After that, samples were baked at 500 °C for 4.5 h under high vacuum conditions (10^{-4} Pa), while pumping of the samples was continued using an ion pump in a closed system, with raising the temperature stepwise to 150, 250, 350 and 500 °C during 3 days for release of impurity gases. The time needed for each stage is decided by the pressure.

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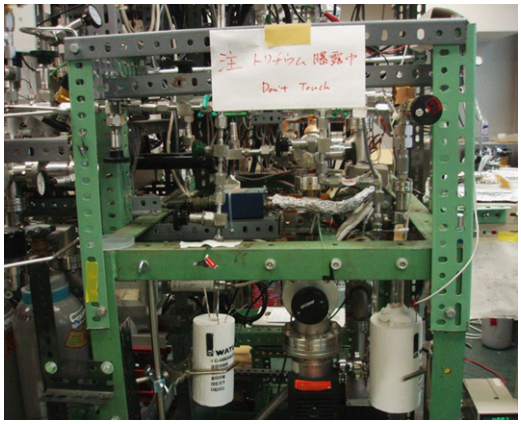

Fig. 1. Tritium exposure device.

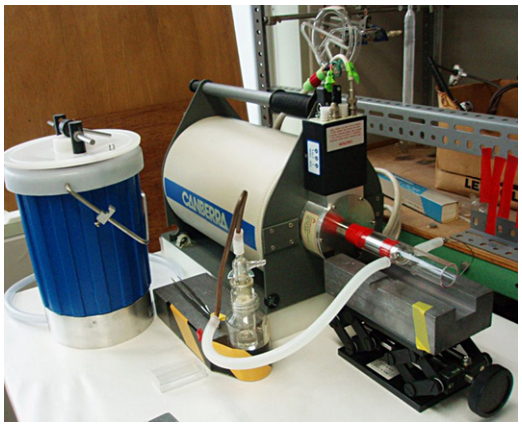

Fig. 2. BIXS measurement device.

2.2. Experimental methods

2.2.1. Tritium exposure experiments

Tritium exposure experiments of the samples were carried out by a tritium exposure device shown in Fig. 1. 400 °C and 100 °C were selected in this experiment corresponding to the temperatures of the tiles during long pulse discharge and normal discharge, respectively. Pressures of tritium gas diluted with deuterium were set at 3 Torr and 5 Torr, respectively. Tritium concentration in the gas was about 16%-T, as shown in Table 1.

2.2.2. BIXS measurement

The BIXS measurement is proposed by Matsuyama et al. [6] for the purpose of in situ measurements of tritium concentration distribution. It is a kind of non-destructive measurements based on the measurement and analysis of X-ray spectra induced by the β-rays from tritium trapped in/on materials by Si/Ge semiconductor detector (Ge in this study) under Ar atmosphere, and can measure the tritium amounts retained on the surface layers and in bulk independently [7]. The depth profile of tritium in the bulk could be clarified by computer simulation by comparing with the intensity and profile of the bremsstrahlung X-ray peak [6]. The measurement depth is about 100 μm. Liquid nitrogen is used for reducing the background noise (Fig. 2).

As for SiC/C samples, intensity of the characteristic X-rays of Ar gives information about the amount of tritium retained on surface and in sub-surface layers, and the tritium amounts retained in bulk could be evaluated by the intensity of characteristic X-rays of elements in samples besides C, and the bremsstrahlung X-rays [8]. Each spectrum of the Ori, Ero and Dep samples was obtained by measuring for more than 15, 3 and 1 h, respectively to minimize the statistic error.

2.2.3. IP method

The IP is a radiation image sensor based on photo-stimulated luminescence. The IP can detect tritium distributed within the depth of about 3.5 μm from the surface of the carbon-based tile [9]. Before the IP measurement, one piece of each kind of the sample exposed in 400 °C was cut into small pieces of 15 mm × 2 mm × 3 mm by a micro cutter (MC-201N, MARUTO) with a linear velocity of 0.375 m/s. It costs about 5 min for the Ori and Ero samples, and more than 20 min for the Dep sample. The cross sections of the samples were then polished with polishing paper. The polishing direction was kept along the surface to minimize tritium contamination from different tritium retained area. After polishing, samples were put into a darkroom together with a series of standard samples prepared by ARC in 2005, contacting with the surface of IP face to face. Surfaces of the samples were covered with adhesive tape to reduce the effect of large amount of tritium estimated to be retained on surface. About 2 h later, the plate was read by a FLA-700-reader with a scanning for 5 min.

Table 1
Tritium concentration of the gas released from getter material (Zr–Ni alloy).

Temperature (°C)	Pressure (Torr)	T-concentration (%)
400	3.0	16.0
100	5.0	16.4

3. Results and discussion

3.1. X-ray spectra

Fig. 3 shows the X-ray spectrum for the Ori surface. Two kinds of X-ray peaks were observed for this sample. One is sharp intense peaks of characteristic X-rays, whose maximum intensity appeared at 1.73 keV and 2.96 keV, respectively, corresponding to Si(Kα) and Ar(Kα). The characteristic X-ray from carbon atoms is low energy that could not be detected by the device. The other is a considerably broad weak spectrum of bremsstrahlung X-rays, who reaches a maximum at about 6 keV.

Fig. 4 shows the spectrum of the Ero surface. Two sharp peaks appeared at the energy of 1.73 keV and 2.96 keV, corresponding to Si(Kα) and Ar(Kα). There are many weak sharp peaks that could be observed, such as Ca(Kα) at 3.68 keV, S(Kα) at 2.965 keV, Ti(Kα) at 4.52 keV, Cr(Kα) at 5.44 keV, Fe(Kα) at 6.40 keV, and Cu(Kα) at 8.04 keV, etc. Ti is a dopant element in the bulk. Fe and Cr may be

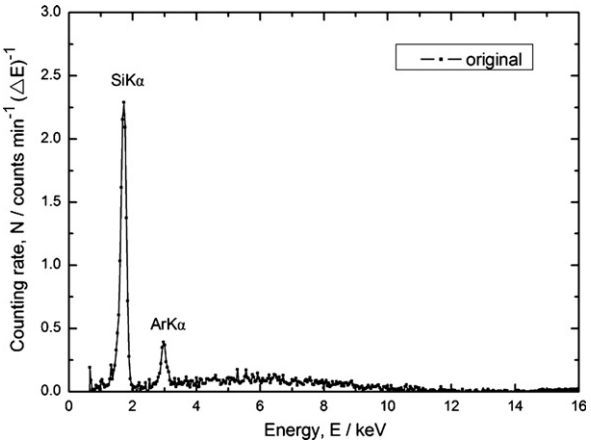

Fig. 3. X-ray spectrum for Ori exposed in 400 °C.