



Spectroscopic study of energetic helium-ion irradiation effects on nuclear graphite tiles



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ABSTRACT

Helium ion-irradiation effects on the nuclear graphite tiles were studied in order to understand the structural modifications and damages that can be produced by fusion reaction in tokamaks. The surface morphological changes due to increasing dose of the irradiation were examined by the field-effect scanning electron microscopy, and X-ray photoelectron spectroscopy elucidated the changes in the shallow surface bonding configurations caused by the energetic irradiation. Raman spectroscopy revealed the structural defects and diamond-like carbon sites that increased with increasing irradiation dose, and the average inter-defect distance was found from the Raman peak intensities as a function of the irradiation dose.

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1. Introduction

Graphite is widely used in fusion devices for its strong physical properties and thermal endurance. Study on the effects of the ion irradiation and plasma treatment on nuclear graphite has been carried out for a long time [1,2]. In the Korea Superconducting Tokamak Advanced Research (KSTAR), graphite tiles are used as the first wall protection facing plasma [3]. When generating plasma in a tokamak, energetic helium ions can be released. As graphite tiles face the emitted energetic ions directly, study of their physical property changes following such exposures is essential.

Various types of structural deformation can occur upon He-ion irradiation, such as surface erosions [1,2] and production of various kinds of pores [4,5]. Many works have shown that such disorders in graphite can change the physical properties, resulting in decrease in the electrical conductivity [6] or induction of ferromagnetism [7]. In our previous works, low-energy proton and deuterium irradiation effects on the physical and chemical properties of highly-oriented pyrolytic graphite were systematically studied [6,8]. In this work, energetic He-ion irradiation effects on the graphite tiles were studied by employing Raman and X-ray photoelectron spectroscopy (XPS) measurements.

2. Experiment

The experiments were carried out with the same graphite tiles that are used in KSTAR, which were purchased from Carbone of

America. Helium-ion irradiation was carried out on the graphite tiles at an energy of 10 keV with a fluence of 3.4 mA for 2, 4, or 6 h, the corresponding irradiated samples being denoted by 2H, 4H, and 6H, respectively. The irradiation rate of the helium ions was 5.625×10^{17} ions/cm² h, measured from a Faraday cup detector. The surface morphology changes were examined with the field-emission scanning electron microscopy (FE-SEM) by using a Hitachi S-4700 Instrument. The Raman spectra were measured with LabRam ARAMIS IR2 with a 532-nm diode laser. The XPS measurements were made with X-tool from Physical Electronics Inc. The penetration depths of the He ions were calculated with Stopping and Range of Ions in Matter (SRIM).

3. Results and discussion

Fig. 1 shows the FE-SEM images of the pristine and He ion-irradiated samples. The pristine sample surface appears rather clean with sharp edges of graphite flakes sticking out. The surface was smudged after irradiation for two hours (2H) forming smooth surfaces. Although graphite is strong against thermal exposure, heat energy transferred by the irradiation is high enough to melt the surface. Craters appear in the 4H sample and the surface shows similar damage in the 6H sample, indicating saturation of the He-ion implantation effect.

Fig. 2 shows the C 1s XPS spectra of the graphite tiles before and after the irradiation, fitted with Gaussian functions with the Shirley background being subtracted. The pristine sample exhibits peaks from sp^2 and sp^3 bonds and C–OH bonds at 284.5 eV, 285.2 eV, and 286.5 eV, respectively [9,10]. The surface of the gra-

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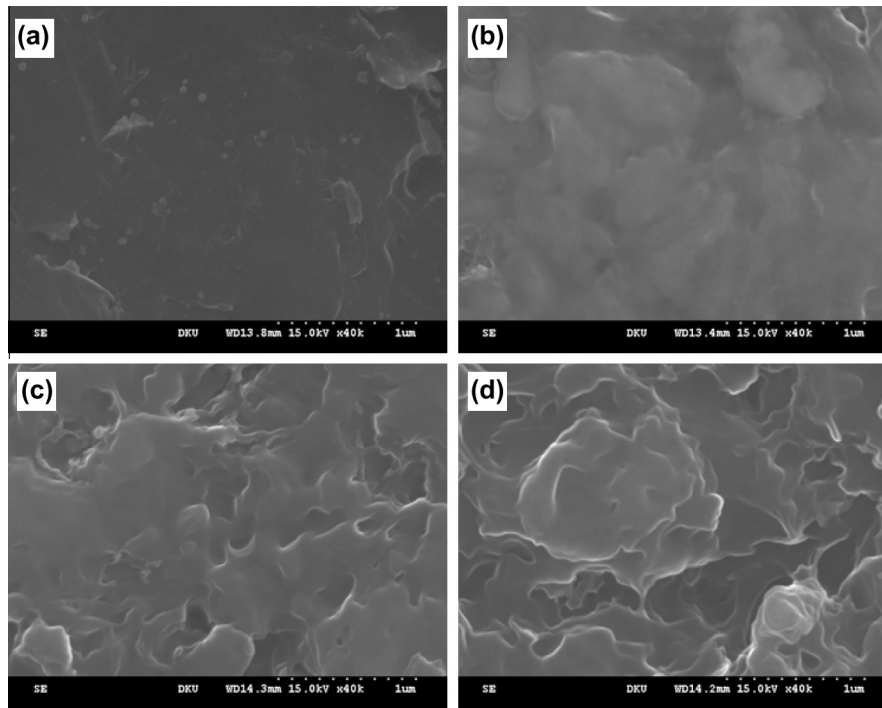


Fig. 1. FE-SEM images of the pristine sample (a), He ion-irradiated samples with irradiation time of 2 h (b), 4 h (c), and 6 h (d), at an energy of 10 keV.

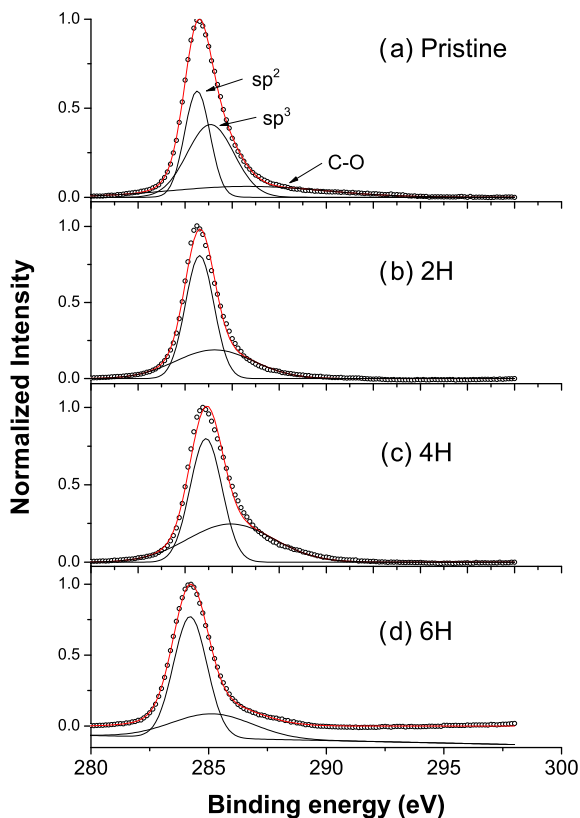


Fig. 2. C 1s XPS spectra fitted with Gaussian components of the pristine (a) and of the samples He ion-irradiated for 2 h (b), 4 h (c), and 6 h (d).

phite tile before irradiation is rather messy and oxidized, but the C–OH bond is polished off on the surface presumably due to the sputtering-off effect following the irradiation. The surface is melted (see Fig. 1) and recrystallized by a low irradiation dose after

irradiation for 2 h (2H) showing reduced sp^3 peak intensity (see Fig. 2(b)) as in pyrolyzed graphite [11], however the sp^3 peak intensity is thereafter increased in the 4H sample, irradiated for 4 h. Thus, overdose of helium-ion implantation would cause surface modification, producing diamond-like carbon (DLC) sites.

The Raman spectra of the pristine and the He ion-irradiated graphite tiles are shown in Fig. 3. The pristine sample exhibited a G peak at 1579 cm^{-1} , a D peak at 1353 cm^{-1} , and a D' peak at 1615 cm^{-1} arising from sp^2 bonds in the small-size domains (see Fig. 3(a)) [12]. The large intensity of the D peak can be attributed to the microscopic disorders and to the edges of the flakes. Drastic increase in the D peak intensity by the irradiation is observed as well as large broadening of both the D and G peaks (see Fig. 3 (b)), indicating increase of the inhomogeneity in the systems after the irradiation. This may seem at odds with the SEM images and the XPS spectra, but may readily be understood by recognizing the fact that the penetration depth of the Raman laser is a few μm deep, which is much deeper than that of X-rays ($\sim 5\text{ nm}$). Recrystallization due to the heat energy may not go deep enough to a few μm .

Disorders in the sixfold rings of graphite lead to broadening of the Raman spectra resulting in Gaussian lineshapes. Continuum electronic scattering, discrete phonon states, and their interference terms are responsible for the asymmetric lineshapes [13]. Generally, a Breit–Wigner–Fano (BWF) line is used for fitting the G peak and a Lorentzian for the D peak (see Fig. 3(a)) [14–16]. The resonance frequency shift and the linewidth of the G peak thus obtained are shown in Fig. 4 as a function of the irradiation time. The G peak was downshifted as the irradiation time was increased (see Fig. 4(a)), indicating softening of the vibrational density of states due to formation of the diamond-like defects with increasing sp^3 bonds [12]. The increase of the G peak linewidth is related to three-dimensional deformation in the system (see Fig. 4(b)) [17]. The resonance frequency of the G peak shows a continuous decrease with increasing irradiation time whereas the linewidth shows saturation after gradual increase up to 4 h of irradiation.

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