

# Chemical Erosion of Diamond-Coated Graphite under Low-Energy Hydrogen Atom Irradiation

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We experimentally investigate chemical erosion of polycrystalline graphite targets coated with boron-doped diamond (BDD) using an induction plasma containing low-energy, high-atomic-hydrogen flux. Chemical erosion is drastically suppressed by diamond coating the graphite target. The chemical sputtering yield for the BDD layer is about two orders of magnitude lower than that for the graphite target. After exposure in low-temperature hydrogen plasmas, however, the surface morphology of the BDD target is significantly modified. The polycrystalline diamond is eroded near the grain boundary, and many pits with diamond-like shapes are observed on the crystal surface. X-ray photoelectron spectroscopy and Raman spectroscopy reveal that the hydrogen atoms penetrate into the BDD target to a depth of at least  $\sim 20$  nm.

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In using graphite materials as plasma-facing components (PFCs) in fusion devices such as divertor tiles, it is recognized that the high chemical erosion rate due to the presence of hydrogen fuel and the related carbon dust formation will cause severe performance degradation in future fusion reactors. Diamond, which is a typical carbon crystal and has an  $sp^3$  electronic structure, offers several features including an extremely high thermal conductivity, a low chemical reactivity, [1] and an increase of electrical conductivity that is possible through boron doping, making it superior for use as a PFC in fusion reactors. To date, several experimental efforts have focused on diamond erosion by energetic-ion irradiation [2, 3], but little effort has been devoted to low-energy atomic hydrogen. In the present research, boron-doped diamond (BDD) is tested for erosion by low-temperature ( $\sim 1$  eV), high-flux hydrogen neutrals to check its suitability for divertor tiles used in a detached divertor.

The experiments were performed in an Ar/H<sub>2</sub> mixture plasma generated by an inductively coupled plasma (ICP), which has the characteristic features of high neutral particle flux  $\Gamma_H \sim 10^{23}$ - $10^{24}$  m<sup>-2</sup> · s<sup>-1</sup>, low ion flux  $\Gamma_i \sim 10^{19}$ - $10^{20}$  m<sup>-2</sup> · s<sup>-1</sup>, and low electron temperature  $T_e \sim 1$  eV [4]. The working gas pressure is  $\sim 4$  kPa. An approximately 30-nm thick polycrystalline BDD layer covers an isotropic graphite, where the boron doping rate is  $\sim 0.5\%$ . We deposit a BDD coating on an isotropic graphite target using hot-filament chemical vapor deposition (CVD). We vary irradiation time from 60 to 1200 minutes to examine the

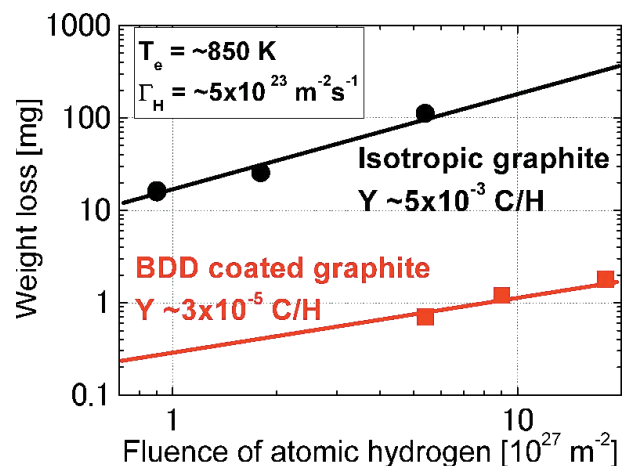


Fig. 1 Dependence of target weight loss on fluence of atomic hydrogen; here,  $Y$  indicates the chemical sputtering yield by hydrogen atoms.

effect of target erosion due to hydrogen fluence into the target. The surface temperature is measured through a quartz window using a radiation thermometer.

Figure 1 shows the weight loss of the BDD and the graphite targets as a function of atomic hydrogen fluence. Here, atomic hydrogen fluence is estimated using the results of an electromagnetic fluid simulation under local thermodynamic equilibrium conditions [5]. The erosion of both the graphite and the BDD target increases linearly with increasing hydrogen fluence. The chemical sputtering yield ( $Y$ ) is estimated from the target weight loss

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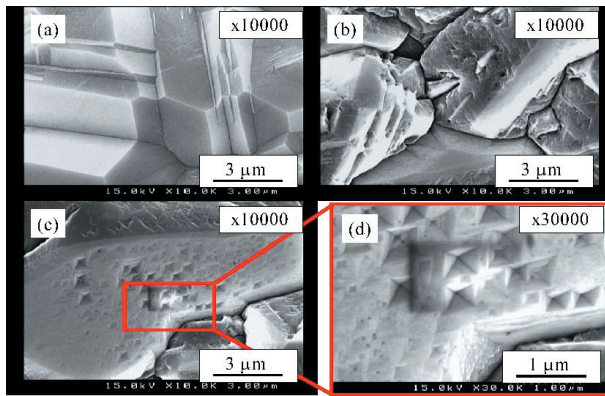


Fig. 2 SEM images of the surface of BDD coated graphite targets (a) Before exposure in plasma and (b) and (c) After exposure in plasma. (d) Enlarged view of (c).

and the atomic hydrogen fluence. The chemical sputtering yield of the BDD target is about  $3 \times 10^{-5}$  C/H (etching rate  $\sim 0.4 \mu\text{m/h}$ ), whereas that of the graphite target is about  $5 \times 10^{-3}$  C/H (etching rate  $\sim 0.1 \text{ mm/h}$ ). We found that the chemical sputtering yield of the BDD target is about two orders of magnitude lower than that of the graphite target in this experiment.

Figures 2(a)-(d) show the morphology of the BDD target, imaged using a scanning electron microscope (SEM). After exposure to the Ar/H<sub>2</sub> mixture plasma at a surface temperature,  $T_s \sim 850 \text{ K}$ , the morphology of the BDD surface is modified significantly, as shown in Figs. 2(b) and (d). The grain boundaries of polycrystalline diamond are eroded, and their surfaces are carved in the shape of small crystals. To investigate the effects of hydrogen irradiation on BDD crystal structure, we performed x-ray photoelectron spectroscopy (XPS) and Raman spectroscopy. The peak separation in the C1s spectrum obtained by XPS shows two peaks corresponding to an sp<sup>3</sup> carbon at 285.6 eV (i.e., diamond), an sp<sup>2</sup> carbon at 284.3 eV (i.e., graphitic carbon), and/or a CH bond at 284.2 eV. We believe that the peak at 284.2–284.3 eV comes from the CH bond because it is known that the outermost carbon atoms in as-deposited diamond crystals are terminated by hydrogen atoms. This hydrogen bond is very resistant to replacement by other bonds [6]. After exposure to the plasma, the increase in the CH peak can be clearly seen in the BDD surface layer. This result implies that the diamond structure on the BDD surface layer becomes amorphous because of irradiation by atomic hydrogen.

Figure 3 shows the depth profile of the CH/sp<sup>3</sup> peak ratio from the C1s spectra obtained from argon-ion etching at an energy = 1000 eV. Before exposure, it appears that an almost mono-atomic layer of the BDD surface is terminally-bonded by atomic hydrogen. This terminal bonding is attributed to the hydrogen plasma irradiation when the diamond film is formed. After atomic-hydrogen irradiation, the CH bond concentration is clearly enhanced

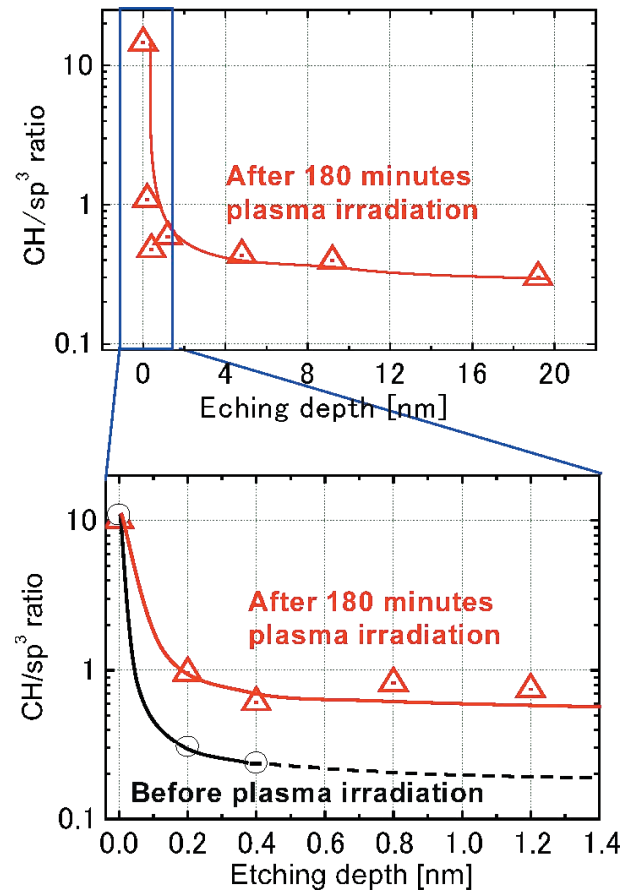


Fig. 3 Depth profile of the CH/sp<sup>3</sup> ratio from the C1s spectrum, obtained from argon-ion etching with an ion energy of 1000 eV.

to a depth of at least  $\sim 20 \text{ nm}$  from the BDD surface, but the CH bond decay characteristics are almost unchanged. However, Raman spectroscopy measurements with  $\sim 1 \mu\text{m}$  of depth resolution do not show any significant change in the BDD structure, with only the D-band at  $1333 \text{ cm}^{-1}$  being identical in the unexposed and exposed BDD targets.

In summary, the chemical erosion of polycrystalline BDD targets has been studied using an ICP containing, low-energy, high atomic-hydrogen flux plasma. The chemical sputtering yield for the BDD target is about two orders of magnitude lower than that of the graphite target. The polycrystalline diamond is eroded gradually but significantly by plasma irradiation, and its surface is carved in the form of small crystals. In addition, on the BDD surface, pits in the shape of crystals grow with exposure time. Ref. [7] suggests that characteristic pits on the diamond surface are related to the anisotropic etching by the hydrogen plasma due to crystal defects. From XPS and Raman spectroscopy measurements, we conclude that the hydrogen atoms penetrate into the BDD target to a depth of at least 20 nm. Detailed analysis of the polycrystalline diamond erosion will soon be presented in a future report.

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