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He^+ irradiation temperature influence on the structure and nanohardness of hydrocarbon films

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ABSTRACT

Polymer-like hydrocarbon films were irradiated with 100 keV He^+ or annealed at sample temperatures varying from 25 to 600 °C. The effects of sample temperature on the structure and nanohardness of hydrocarbon films are investigated by atomic force microscopy (AFM), AFM-based nanoindentation, Fourier transform infrared spectroscopy, and Raman spectroscopy. Analysis shows that annealing results in the decrease in the nanohardness of hydrocarbon films from 4.0 GPa to 0.55 GPa while He^+ irradiation at an elevated sample temperature results in the formation of dense diamond-like carbon films with nanohardness up to 20.0 GPa. This indicates that polymer-like hydrocarbon films can be transformed into the hard diamond-like carbon films with a relatively low H content on vacuum vessels of fusion devices due to the energetic bombardments at an elevated wall temperature.

1. Introduction

Tritium retention caused by the cooperation of gas-phase hydrogen (H) or hydrocarbon (HC) species is an important issue for the effective and safe operation of current and planned fusion devices, such as ITER, and for the design of fusion energy systems. Indeed, hydrocarbon coatings, which are tens of microns thick, were observed on plasma-facing materials (PFMs) in current fusion devices due to redeposition of eroded HC species [1,2]. Depending on the location in ITER, hydrocarbon films deposited inside the vacuum vessels will be subjected to different levels of low-energy (<500 eV) He/H ions or neutral flux, neutron and gamma radiation as well as bombardment by ions produced in the residual gas and accelerated by surrounding electromagnetic fields [3]. Various surface processes including surface adsorption and growth, chemical erosion, ions-assisted sputtering, are induced by low-energy ions or neutral species [4]. Surface reactions of reactive neutral species, such as hydrocarbon radicals can lead to deposition of polymer-like hydrocarbon films in remote areas of fusion devices [5,6].

Surface irradiation of PFMs by energetic (>100 keV) ions or neutrons can significantly affect the structures and properties of redeposited hydrocarbon materials [7–9]. After the energetic bombardments, the properties of hydrocarbon films, such as conductivity, trapping sites, hydrogen contents are obviously altered. Previously, we have evaluated the performances of the hydrocarbon films irradiated with 60–140 keV at room temperature [10]. Measurements show that He^+ irradiation leads to a decrease in their surface roughness and H contents, and an obvious increase in their hardness. However, during the normal operation of ITER, the expected surface temperature of vacuum vessels can be up to >600 °C [11]. In this study, we focus on the He^+ irradiated hydrocarbon films at elevated sample temperature. The effects of He^+ irradiation temperature on the structure and properties of hydrocarbon films are systematically investigated.

2. Experimental

2.1. He^+ irradiation of hydrocarbon films at elevated sample temperature

Plasma-enhanced chemical vapor deposition was used utilized to deposit amorphous hydrocarbon films on Si single crystal substrates. This method is based on atmospheric dielectric barrier discharge (DBD) deposition principle and has been described in details previously [12]. The vacuum-tight discharge chamber with gas volume of $\varphi 100 \times 10$ mm mainly consists of a stainless-steel

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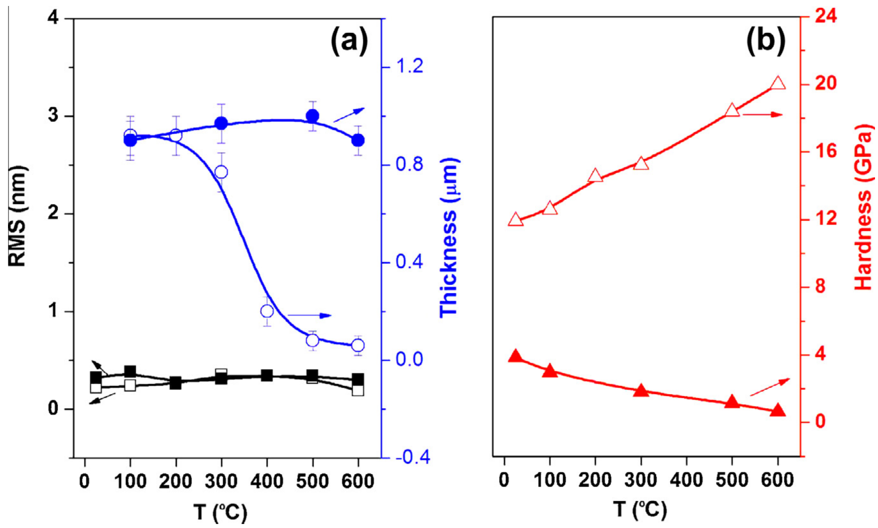

Fig. 1. (a) Thickness and RMS roughness of both annealed (solid symbols) and He^+ irradiated (hollow symbols) hydrocarbon films as a function of sample temperature and (b) nanohardness of both annealed (solid symbols) and He^+ irradiated (hollow symbols) hydrocarbon films as a function of sample temperature.

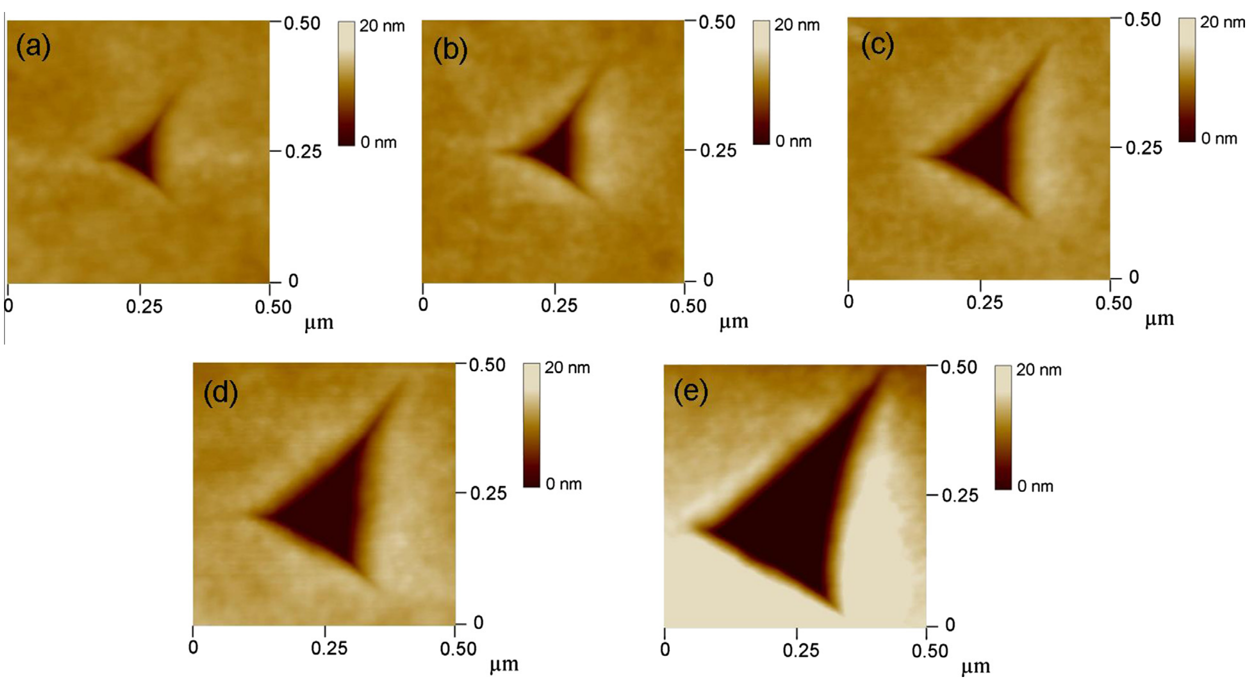

Fig. 2. The representative AFM images with indentation impressions for the hydrocarbon films annealed at the sample temperature of (a) 25 °C, (b) 100 °C, (c) 300 °C, (d) 500 °C and (e) 600 °C. All indentation impressions are generated with the single vertical diamond-tip movement using the same loading force of 40 μN.

ground electrode and a parallel 5 mm thick glass dielectric barrier plate. The stainless-steel high voltage electrode is attached to the outside of the glass plate at atmospheric pressure in order to prohibit the high-voltage breakdown around the glass. This deposition is performed at a CH_4 pressure of 100 Pa and a gas spacing of 5 mm. The repetition frequency of AC power supply and applied peak voltage are 5 kHz and 12 kV, respectively. Plasma deposition lasts for 100 min, which results in the 1.0 μm-thick hydrocarbon films with a good adherence on the Si substrates. This hydrocarbon film shows the very smooth surface with RMS roughness of 0.68 nm and a Knoop hardness of 4.0 GPa.

Then, hydrocarbon films were irradiated with 100 keV He^+ at the constant ion fluence of 3.0×10^{16} ions/cm^2. The ion irradiation system was described elsewhere [13]. The incident angle of the ion beam is 90°, and the substrate temperature during irradiation varied between 25 and 600 °C. TRIM calculations from Monte Carlo code based on the binary collision approximation [14] indicated a He^+ projected range of 590 nm. To analyze the effects of He^+ irradiation and substrate temperature on hydrocarbon films, annealing tests were also performed for hydrocarbon films without no He^+ irradiation. Annealing temperature was also changed from 25 to 600 °C.

2.2. SEM, FTIR and Raman analyses

Field emission scanning electron microscopy (SEM Hitachi S-4800, Japan) is utilized to characterize these hydrocarbon films. The SEM measurements are performed mainly on specimen cross