

The Effects of Negative Carbon Ion Beam Energy on the Properties of DLC Film

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Abstract

The effects of negative carbon ion beam energy on the bonding configuration, hardness and surface roughness of DLC film prepared by a direct metal ion beam deposition system were investigated. As the negative carbon ion beam energy increased from 25 to 150 eV, the sp^3 fraction of DLC films was increased from 32 to 67%, while the surface roughness was decreased. The films prepared at 150 eV showed the more flat surface morphology of the film than that of the film prepared under another ion beam energy conditions. Surface roughness of DLC film varied from 0.62 to 0.22 nm with depositing carbon ion beam energy. Surface nano-hardness increased from 12 to 57 Gpa when increasing the negative carbon ion beam energy from 25 to 150 eV, and then decreased when increasing the ion beam energy from 150 to 200 eV.

Key words: Diamond-like carbon, Ion bombardment, Nanohardness, AFM

1. Introduction

Since Diamond-like carbon (DLC) films have a mechanical hardness, low friction coefficient, chemical inertness, and optical transparency, high quality thin DLC films have various applications including micro electro mechanical devices and protection layer of cutting tools, moldings, and magnetic storage medias¹⁻³. It is well known that the sp^3/sp^2 atomic carbon ratio in DLC film is strongly related to the quality of DLC films and in general, the higher the sp^3/sp^2 ratio, the closer the DLC film properties approach those of diamond⁴. Therefore, many deposition methods have been considered to increase the sp^3 carbon content in DLC film and among of them, ion beam deposition methods such as direct negative metal ion beam deposition (DMIBD), mass selected ion beam deposition and plasma immersion ion implantation have been recommended as advanced DLC deposition techniques⁵⁻⁷. In this study, DLC films were prepared by DMIBD under different negative carbon ion beam energy conditions and then, the effects of negative

carbon ion beam energy on the bonding configurations and properties of DLC films were investigated. The DMIBD method uses a solid caesium pellet as a primary Cs^+ ion source. The primary Cs^+ ions were extracted from the pellet and then sputtered a graphite target. Sputtered carbon atoms were changed into negative carbon ions by surface ionization⁸ and then accelerated towards the Si substrate because the target is biased negatively, while the substrate is electrically grounded.

Since there was no need for plasma gas in the DMIBD, secondary carbon ions lost little of their kinetic energy by thermalization before deposition. In this work, the dependence of the sp^3 content of DLC film on negative carbon ion energy was considered using XPS and the effects of ion beam energy on the surface morphology and hardness of the film was also considered.

2. Experimental

DLC films were prepared using a direct negative carbon ion beam deposition system in a high vacuum setup with a base pressure of 8.0×10^{-5} Pa. The unique

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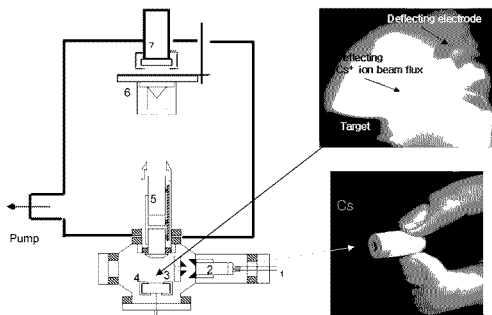


Fig. 1. Schematic diagram of the DMIBD system.

1. Pellet bias electrode, 2. Cs pellet, 3. Cs⁺ ion deflecting electrode, 4. Graphite target, 5. Focusing optic electrode (Einzel lens), 6. Substrate, 7. Heater.

properties and basic operation of the DMIBD methods have been described in previous publications^{9,10}. The operating pressure was about 1.3×10^{-3} Pa. As a sputtering target, graphite target with a diameter of 1-in. and a thickness of 0.25-in. was used. All DLC depositions were performed at a 20 cm distance between target and substrate. Fig. 1 shows a schematic diagram of the direct negative carbon ion deposition system and shows the deflecting primary Cs⁺ ion beam flux. Primary Cs⁺ ions were extracted from the Cs pellet by biasing high voltage, which was applied across the pellet. The Cs⁺ ion beams emitted were deflected to the target by a positive deflecting voltage. During deposition, the Cs⁺ ion dose and bombarding energy onto the target were kept constant at 3×10^{14} ion/cm² and 3 keV, respectively from the previous study¹⁰. As substrates, a 2-in Si (100) wafer was used.

Although the substrates were not heated intentionally, the substrate temperature, which was measured with a thermocouple, reached approximately 70°C due to radiation from the heating element of the primary Cs ion source. The deposition time was adjusted to obtain a film thickness of 300 nm. The thickness was measured from the several defined edges with a surface profilometer.

After deposition, in order to characterize the bonding states of carbon atoms in the film, XPS (Physical electronics) with an Al K α monochromatic excitation source with energy of 1486.6 eV was employed at

350 W. XPS curve fittings were performed after Savitsky-Golay smoothing¹¹ and a Shirley background subtraction¹², by a fitting method employing a mixed Gaussian-Lorentzian function. The nanohardness of DLC films were obtained by using a Hysitron Triboscope nanoindenter connected to a Digital Instruments Nanoscope IIIa Atomic force microscopy (AFM). The system is equipped with a cube corner diamond tip. The measured depth was 30 nm. AFM analysis was also performed for the characterization of surface morphology and roughness of the film prepared with different negative carbon ion beam energy.

3. Results and Discussion

Fig. 2 shows the typical XPS spectra of DLC films prepared under negative carbon ion beam energy of 50 eV. The trace of oxygen in the XPS spectra is not clearly investigated. However, it may be due to the ambient gas during deposition.

In order to consider the effects of negative carbon ion beam energy on the bonding configuration of DLC film, the plasmon energy loss from XPS analysis was observed. Fig. 3. shows the change of XPS spectra of carbon 1s core peak of carbon films prepared under different negative ion beam energy from 25 to 200 eV. As shown in Fig. 3, the π plasmon loss peak decreased as the negative carbon ion

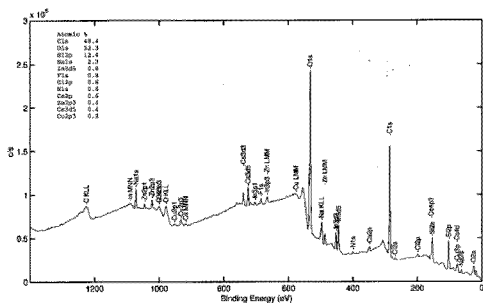


Fig. 2. XPS spectra of DLC film prepared at the ion beam energy of 50 eV by direct negative carbon ion beam deposition.

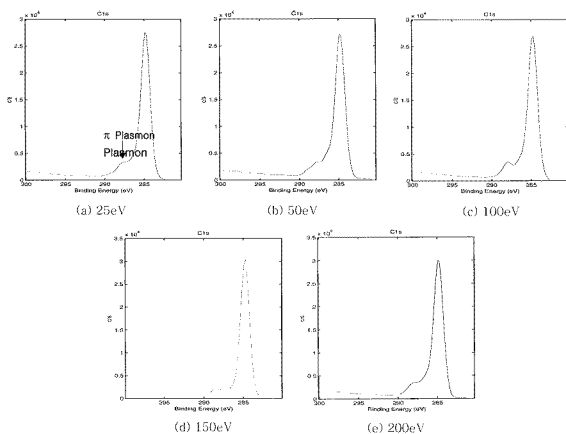


Fig. 3. XPS spectra of DLC films prepared at different negative carbon ion energy.

energy increased. This result meant that the sp^3 bond fraction increases with increasing carbon ion energy (≤ 150 eV)²³. In the DMIBD, the ionized carbons transferred their kinetic energy directly to the growing DLC film, thus it could achieve an efficient kinetic energy transfer because there was no necessity for an

auxiliary gas ion source to the kinetic energy transfer.

Fig. 4 shows the sp^3 fraction estimated from the XPS spectrum as a function of carbon ion beam energy. The sp^3 fraction of DLC films was deduced from XPS fitting for C1s core peak, consisting of peaks due to diamond (285.2 eV), graphite (284.4

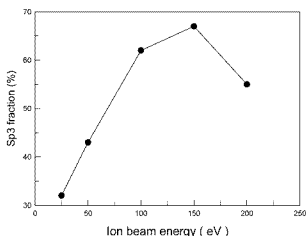


Fig. 4. The variation of sp^3 contents in DLC films with negative carbon ion energy.

eV), CO-contaminated (286.5 eV), and SiC (283.7 eV) phases. Since the area of each peak was related to the concentration of the corresponding phase, the sp^3 content was estimated by taking the ratio of diamond peak area over the sum of diamond and graphite peak areas. Each component is a convolution of a Gaussian and a Lorentzian method, and the contribution of the background was approximated by the Shirley methods.

As the ion beam energy increased from 25 to 150 eV, the sp^3 fraction appeared to increase from 32 to 67%. However, DLC film prepared at 200 eV had a lower sp^3 fraction than that of the film prepared at 150 eV. A similar increase and decrease of the sp^3 bond fraction has been observed and explained in detail by Lifshitz *et al.*^{14,15)} and Hofsaess *et al.*¹⁶⁾

Fig. 5 shows the AFM images of DLC films prepared at ion beam energy of 50, 150, and 200 eV, respectively. Each AFM image is comprised of an identical scan size of $2 \mu\text{m} \times 2 \mu\text{m}$ and the vertical scale was set at 20 nm for AFM measurements. Fig. 6(a) shows the surface image of the film prepared at 50 eV. The surface roughness was higher than that of the bare Si substrate. At 150 eV, the energetic carbon ion bombardments enhanced the mobility of carbon adatom on the surface, which made the film smoother than that of the film prepared at low ion beam energy. However, at 200 eV, the surface roughness of the film increased. According to Y. Lifshitz *et al.*¹⁴⁾, radiation enhanced diffusion (e.g., due to atomic displacement) is a reasonable way to by which the surface roughness would increase and the diamond like properties would simultaneously be suppressed. Table 1 shows the dependence of the surface roughness

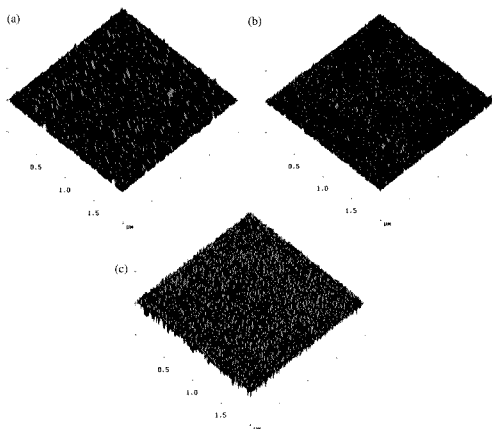


Fig. 5. AFM images of DLC films prepared at the ion beam energy of (a) 50, (b) 150, (c) 200 eV.

Table 1. The dependence of the surface roughness (RMS) of a DLC film on the ion beam energy

Ion beam energy	25	50	75	100	150	200
RMS (nm)	0.62	0.53	0.38	0.29	0.22	0.41

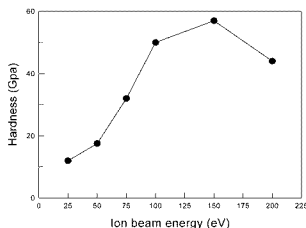


Fig. 6. Influence of the negative carbon ion energy on the nanohardness of DLC film.

of a DLC film on the ion beam energy.

The nanohardness of the films was measured as shown in Fig. 6. The surface nanohardness increased with an increase of the negative ion beam energy from 25 to 150 eV, then decreased with an increasing of the negative ion beam energy from 150 to 200 eV.

4. Conclusions

In order to investigate the effects of negative carbon ion beam energy on the bonding configuration and surface morphology of DLC film, the films were prepared on a Si(100) substrate by a direct negative metal ion beam deposition system at different levels of ion beam energy. As the ion beam energy increased from 25 to 150 eV, the sp^3 fraction in DLC films increased from 32 to 67%. But the DLC film prepared

at 200 eV had a lower sp^3 fraction than that of the film prepared at 150 eV. Hardness also increased from 12 to 57 Gpa with carbon ion beam energy (≤ 150 eV) but the higher ion beam energy ($=200$ eV) have lead to a lower hardness of the film. The surface roughness also related with ion beam energy. Surface roughness of DLC film varied from 0.62 to 0.22 nm with depositing carbon ion beam energy.

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