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Diamond-like carbon sputtering by laser produced Xe plasma

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1. Introduction

Diamond-like carbon (DLC) films have some excellent materials properties such as high hardness, low surface roughness, and low friction. DLC films also display a high barrier to gas permeation, are good electrical insulators, are relatively chemically inert, and have a high biological compatibility. Because of these properties, DLC films are expected to be used in various applications. We recently proposed a grazing incident mirror coated by such a DLC film in a laser-produced plasma X-ray source (LPX) [1]. We expected that its low surface roughness would be suitable for the total reflection of grazing incident X-rays and its high hardness would give high resistance to laser plasma ion sputtering.

It is anticipated that a LPX, which uses radiation from high-energy-density plasma of a target irradiated by a high-peak-power laser, will be used in industrial applications, especially as an extreme ultraviolet lithography (EUVL) source for the industrial mass production of semiconductors. We have been developing a LPX using a solid Xe target and have demonstrated continuous X-ray generation at 5–17 nm with an average power of 20 W, when the laser pumping power was 100 W at a pulse repetition rate of 320 Hz [2]. However, in the LPX, simultaneous with X-rays, the plasma emits debris mainly consisting of fast ions, which sputter mirror surfaces near the plasma, quickly degrading their reflectivity. A solution to this issue is critical for LPXs to be used in industrial applications. Therefore, to prevent sputtering and to achieve longer mirror lifetimes, we have considered using a mirror coated by a DLC film, a material which is resistant to plasma ion sputtering.

ABSTRACT

The sputtering of diamond-like carbon (DLC) was investigated using Xe ion bombardment from the laser plasma X-ray source (LPX). The LPX we developed uses a solid Xe target and emits UV–X-rays and Xe ions. Using the LPX as an ion source, we measured etching depths of DLC, Ru, and Au films using a quartz crystal microbalance (QCM) to determine their ion sputtering rates at incident angles of 0° and 70°. The calculated results by the SRIM code were able to predict the measured results, except for the case of the DLC film at 0° incident. Our measured result indicated that the DLC sputtering at 0° was ten times larger than previously reported data, in which an ion gun was used. We consider that the difference was a characteristic effect of the laser plasma, and can be explained as a synergistic effect of ion bombardment and UV radiation from the Xe plasma.

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To investigate the ion-sputtering resistance of DLC practically, we used the LPX as a Xe ion source and measured the DLC film etching depth using a quartz crystal microbalance (QCM), in comparison to Ru and Au films that are generally used as mirror materials. Because of the need for degradation studies of ion-thruster-grids [3], many researchers have measured the Xe ion sputtering rate for carbon films. Compared with these previous data, our measured etching depth of DLC films by normal incident ions was more than ten times as large. While the previous etching data were obtained using an ion gun, we used the LPX in our experiment. From this result, we considered that the high sputtering rate was caused by synergistic effects of ion bombardment and UV radiation from the Xe plasma. In this paper, we report the sputtering characteristics of DLC films caused by laser produced Xe ions, and compare the degradation behavior with Ru and Au films.

2. Experiment

A schematic of the experimental setup is shown in Fig. 1. We used a quartz crystal microbalance (QCM) to measure the etching depth on the DLC film resulting from Xe plasma ions sputtering from the LPX. The LPX constitutes a drum system supplying a continuously refreshed solid-Xe-target and a drive laser. The drum target system and the QCM were installed in a vacuum chamber and the drive laser pulses passing through the window were focused perpendicularly onto the target by the lens. The chamber vacuum pressure was 0.2 Pa.

As ions bombard test films coated on the QCM, atoms are ejected from the surface of the film and the resonance frequency of the QCM changes. Thus, we can deduce mass loss, and calculate etching depth, of the film from this frequency change. We placed

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Fig. 1. Experimental setup. The test surface normal was tilted at a 0° (QCM1) and 70° (QCM2) angle with respect to the incident ion axis. The DLC, Ru, and Au sample films were coated on the QCM with thicknesses of 500, 100, and 100 nm, respectively.

two QCM sensors 73 mm from the target point where the incident laser beam creates the Xe plasma. The sensors were arranged on both sides of the incident laser beam so that the laser-Xe target-QCM angle was 45° (see Fig. 1). (The QCM surfaces were not immersed within the plasma column; this is not plasma etching.) The two sensors were tilted such that their surface normal directions were 0° and 70° with respect to the incident ions axis. DLC, Ru, and Au sample films were coated on the quartz sensors with thicknesses of 500, 100, and 100 nm, respectively.

The DLC film used in this study was developed in our laboratory and formed by Ar cluster ion beam assisted deposition using C_{60} as a carbon source [4]. The gas cluster ion beam (GCIB) assisted deposition forms hard DLC films by bombardment-induced high-pressure and high-temperature effects at the surface impact site, as well as ultra low energy effects. This DLC film was found to have a hardness of 50 GPa, which is approximately two times higher than other films deposited using conventional methods. Because of this hardness, we expected that our DLC film would have a high sputtering resistance. The average surface roughness of the film was found to be below 0.5 nm; we considered that this smooth surface was suitable for total X-ray reflection at a grazing incidence. The film was also found to contain a high sp³ C content (diamond bonding) and to contain no H. Recent studies indicate that H-containing DLC films were modified by X-ray irradiation [5]. This was one of the reasons why we chose our DLC film.

As shown in Fig. 2, the drum target system we developed [6] has a cylindrical drum filled with liquid nitrogen, which cools its copper surface to the temperature of liquid nitrogen. Xe gas is directed onto the surface and condenses to form a solid Xe layer. The solid Xe-layer coated drum rotates about the vertical *z*-axis and moves vertically along the *z*-axis, resulting in a spiral motion, supplying a fresh target surface continuously for every laser shot. We also developed a drive laser, which is a slab laser that provides an average power of 100 W at a pulse repetition rate of 320 Hz [7]. The la-



Fig. 2. Illustration of the rotating cryogenic drum (top view).



Fig. 3. Faraday-cup signal of the ion time-of-flight (upper) and the corresponding energy spectrum (lower). Ion flux in the spectrum was calculated assuming the charge state was two.

ser pulse with a wavelength of 1064 nm and a pulse width of 20 ns was focused on the solid Xe target so that the plasma was produced continuously. The Xe plasma emits broad UV-X-ray radiation, and the average power in the 5-17 nm range was estimated to be 20 W. Plasma debris emitted from the LPX was also studied in detail; previous results indicated that the majority of Xe plasma debris comprised fast ions and neutrals could be disregarded [8]. Fig. 3 shows the TOF (Time-of-Flight) Xe ion signal detected with a Faraday-cup, and its energy spectrum. From Fig. 3, the ion energy was found to be less than 6 keV and the total flux of all energies ions was calculated to be $2\times 10^{14}/\text{sr/shot}.$ We concluded that the energies ions caused to sputter the films on the QCM sensor. After the LPX irradiation of the films for 2-10 min at 320 Hz, we monitored the total etching depth for the three films upon extended multi-shot plasma operation using the QCM, and calculated the etching depth per shot.

3. Results and discussion

Fig. 4 shows the measured (solid circle) and calculated (open circle) etching depth per shot of the DLC, Ru, and Au films at incident angles of 0° and 70°. The calculations were done using SRIM software [9]. The SRIM software can calculate the Xe ion sputtering yield, Y_{sputt} (*E*) (atoms/ion) at each ion energy *E*. Ion flux F_{ion} (*E*) (ions/cm²) at the films was obtained from the measured data of Fig. 3(b). Taking the summed product of Y_{sputt} (*E*) and F_{ion} (*E*) over all the ion energies (<6 keV), the etching depth on the substrate ΔT (cm/shot) was calculated as

$$\Delta T = \sum \alpha \cdot Y_{sputt}(E) \cdot F_{ion}(E) \cdot \frac{m}{\rho}$$
(1)

where *m* is the atomic mass (DLC: 1.99×10^{-23} , Ru: 1.68×10^{-22} , and Au: 3.27×10^{-22} g), ρ is the density (DLC: 2.8, Ru: 12.4, and

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