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## Carbon films deposited by MCECR plasma sputtering

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#### Abstract

The mirror-confinement-type electron cyclotron resonance (MCECR) plasma source has high plasma density and high electron temperature, and it is quite useful in many plasma processing, and has been used for etching and thin-film deposition. In this paper, the carbon films about 50 nm thickness were deposited on Si (100) by MCECR plasma sputtering the sintered carbon target with the argon plasma, and its properties were studied. The bonding structure of the film was analyzed by using the X-ray photoelectron spectropscopy (XPS) and the nanostructure was evaluated with the high-resolution transmission electron microscopy (HRTEM). The tribological properties (friction coefficient, wear rate, and wear life) of the film was investigated by using the pin-on-disk tribometer under the conditions that the normal load is 1 N and the sliding velocity is  $0.05\,\text{m/s}$ . The nanohardness of the films was measured by using the nanoindenter under conditions that the maximum displacement is 30 nm and the maximum load is  $500\,\mu\text{N}$ . The optical properties were measured by using the ellipsometer. The residual stress was measured with a surface profilometer. The surface morphology was studied by using the atomic force microscope (AFM).

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Keywords: MCECR plasma sputtering; Carbon films; Films properties

#### 0. Introduction

Carbon films have received much attention recently due to their interesting properties such as extreme hardness, low coefficient of friction, superior mechanical properties, chemical inertness, biocompatibility, high transparency and chemical inertness, which are close to those of diamond. Carbon films have received a great deal of attention recently for a variety of applications [1-4]. Carbon films have been prepared by a variety of methods, for example, RF plasma deposition from hydrocarbon gases, low-energy ion beam deposition, variations of sputtering processes, ECR chemical vapor deposition, and so on. The ECR plasma has high plasma density and high electron temperature. A feature of ECR plasma is that a high-density ion of  $10^{10}$ – $10^{13}$  cm<sup>-3</sup>, and low iontemperature plasma can be generated under low pressure of  $10^{-1}$ – $10^{-2}$  Pa, and it is quite useful in many plasma processing, and has been used for etching and thin-film deposition. For example, Suzuki et al. have prepared the DLC films using RF bias ECR microwave plasma deposition from ethylene gas (C<sub>2</sub>H<sub>2</sub>), Kamata et al. have deposited the amorphous hydrogenated carbon films on glass using ECR-CVD of CH<sub>4</sub> and H<sub>2</sub>, Muramatsu et al. have deposited the amorphous carbon films using ECR sputtering, Kamijo et al. have deposited the DLC films using ECR sputtering method, and Yajima et al. have deposited the highly wear-durable carbon films using ECR sputtering method [5-8], and so on. Among ECR plasma sources, the ion density and ionization ratio of the mirrorconfinement-type electron cyclotron resonance (MCECR) is higher, and its application is larger, Miyake et al. have deposited the BaSrTiO<sub>4</sub> and SrTiO<sub>3</sub> films using MCECR plasma sputtering [9,10]. However, there are no reports on the preparation of carbon films using MCECR plasma sputtering.

In this paper, the carbon films were deposited by MCECR plasma sputtering on Si, and the properties of carbon films was studied. The nanostructure was evaluated

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an H-9000 HRTEM with point-to-point resolution of 0.19 nm, the bonding structure of the film was analyzed by XPS, the refractive index were measured by the ellipsometer, the nanohardness of the films was measured by the nanoindenter, the tribological properties were measured by the POD, the residual stress were measured with a surface profilometer, and the surface morphology was studied by using the atomic force microscope (AFM).

#### 1. Experimental method

The MCECR plasma sputtering apparatus is schematically shown in Fig. 1. Typically, the power of 2.45 GHz microwave was varied up to 500 W for obtaining MCECR plasmas. The sintered carbon target of 46 mm in length and 110 mm in inner diameter was located near the center of the mirror field. The target material was sputtered by a DC power supply. The DC power was varied up to 1000 V. The substrate was not water-cooled and heated. The substrate was kept at floating potential, or a bias voltage between -120 and +120 V. Carbon films were deposited on p-type Si (100) substrate at a base pressure of  $2 \times 10^{-4}$  Pa and a deposited pressure of  $2 \times 10^{-2}$  Pa. The discharge gas used is argon. The thickness of films was about 40 nm. For the sputtering, the substrate temperature was room temperature.

#### 2. Results and discussion

#### 2.1. Nanostructure of carbon films

Fig. 2 shows bright-field cross-sectional HRTEM images of two films prepared using a bias voltage of +30 (sample A) and +80 V (sample B). Fig. 3 shows the C1s XPS spectra of samples A and B. Diaz et al. reported that a C1s spectrum analysis is effective in determining the relative concentration of sp<sup>2</sup> and sp<sup>3</sup> hybrids. Both spectra mainly consist of two components, one at 284.14 eV and the other at 284.73 eV, which are identified as sp<sup>2</sup> and sp<sup>3</sup> hybrids. The small peaks at 285.38 eV in Fig. 3 are due to C-O

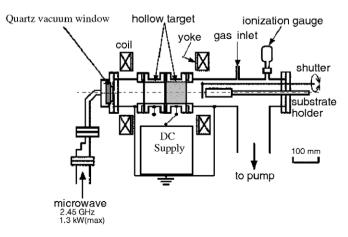


Fig. 1. MCECR plasma sputtering apparatus.

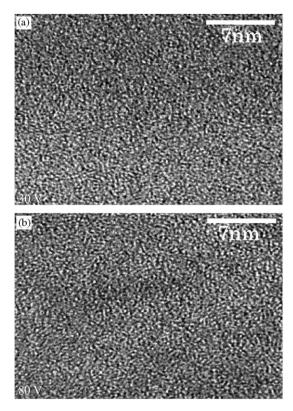


Fig. 2. Bright-field cross-sectional HRTEM images of carbon films: (a)  $+30\,\mathrm{V}$  bias, (b)  $+80\,\mathrm{V}$  bias.

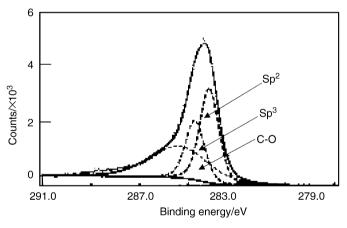


Fig. 3. C1s XPS spectra of MCECR-sputtered carbon films.

bonding. The XPS spectra revealed that the bond structure of the films is a mixture of  $\rm sp^2$  and  $\rm sp^3$ , and the main bond structure is  $\rm sp^2$ . The  $\rm sp^2/\rm sp^3$  ratio, which is the ratio between the areas of  $\rm sp^2$  and  $\rm sp^3$  spectra, is 2.05. Fig. 4 is the relationship between  $\rm sp^2/\rm sp^3$  and bias, from it, the bias has an important effect on the structure and composition of films. At  $+30\,\rm V$  of bias,  $\rm sp^2/\rm sp^3$  of carbon films is least, and its value is 1.61. The reason of this result is that, before  $+30\,\rm V$  of bias, the energy of ion is too high, making the temperature of carbon films high, and making the  $\rm sp^2$  come into being, after  $+30\,\rm V$  of bias, the energy of ion decreases,

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