

Relationship between mechanical properties and chemical groups in a-C:F films prepared by RF unbalanced magnetron sputter deposition

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Abstract

a-C:F films were prepared by RF unbalanced magnetron sputter deposition on Si substrates. The modulus and hardness of the films and their relationship with chemical groups in the films were investigated. The results show that the modulus and hardness of the deposited films are not only determined by the nature of cross-link C–C network, but also affected by the fluorocarbon groups. The C–C network of the films is composed of sp^2 cluster, thus the modulus and hardness of films are close to those of polycrystalline graphite. Compared with other fluorocarbon groups existing in the films, the effect of $-(CF-CF)_n-$ group on the modulus and hardness of the films is much higher. With increasing of $-(CF-CF)_n-$ group proportion, modulus and hardness of the films linearly decrease.

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Keywords: a-C:F film; Hardness; Modulus; Fluorocarbon groups

1. Introduction

In recent years, fluorination amorphous carbon (a-C:F) film is of interest for its special properties, such as low friction coefficients, low dielectric constants and low surface energy. It has potential application in many fields, such as self-lubrication layer [1,2], low-k materials of ultra-large scale integration [3,4], anti-adhesion layer for micro-machined device [5,6] and so on. Modulus and hardness are important properties of a-C:F films. They are determined by the characters of the crossing-link C–C group and the fluorine content in the films. According to the results of different research groups, in general, the modulus and hardness of the a-C:F film decrease with the increasing of fluorine content in the films [7–11]. That is to say, the high hardness and high fluorine content are incompatible. However, people desire high fluorine content in the film to improve the hydrophobic property and the high hardness to improve the wear resistance. The fluorocarbon groups in a-C:F films are variety, which kind of fluorocarbon group has the main effect on the decreasing of modulus and hardness is worth of investiga-

tion. If we have a good insight to this problem, a-C:F film with high hardness and fluorine content can be deposited by design of the type of fluorocarbon groups in the films.

2. Experiments

Fig. 1 is a schematic diagram of RF unbalanced magnetron sputter deposition equipment. A PTFE target was set on the side wall of the vacuum chamber for film deposition. RF power of

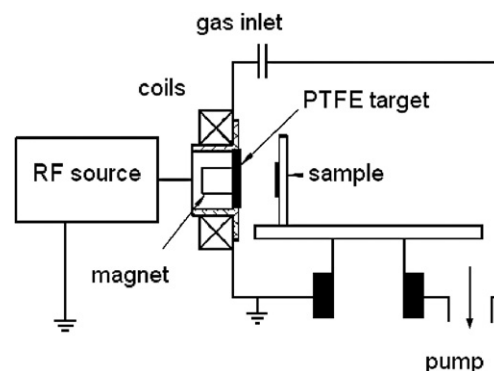


Fig. 1. Schematic diagram of RF sputtering equipment.

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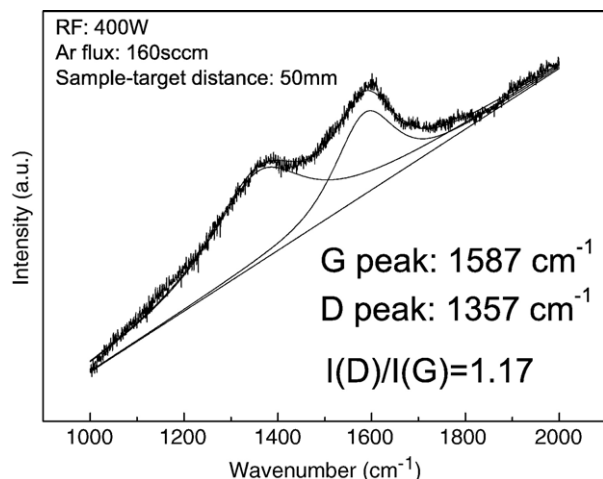


Fig. 2. Typical Raman spectrum of a-C:F films.

200 W, 300 W and 400 W were applied to the target during deposition process, respectively. The working gas was Ar with flux of 80 sccm, 160 sccm and 200 sccm, respectively. Silicon<100> substrates were floated and located in front of the target at a distance of 30 mm, 50 mm and 70 mm, respectively. The processing time was 3 h.

Composition and chemical groups of a-C:F films were measured by PHI 5700 X-ray photoelectron spectroscopy (XPS) with take-off angle of 45°. In order to get rid of the effect of organic impurity on the sample surface, the deposited films were sputtered by Ar ions gun for 40 s with energy of 1 keV. The structure of the deposited films was measured by using a JYT6400 Raman spectroscopy with laser wavelength of 532 nm. Modulus and hardness were measured by Nano-Indenter XP (MTS Systems Corp.). For each sample, six indentation tests were performed with repeatability within 5% errors. In indentation test, three-side pyramidal diamond (Berkovich) indenter with radius of about 40 nm was used.

3. Results and discussion

Fig. 2 shows a Raman spectrum from a typical a-C:F film deposited in this experiment. It can be seen that the Raman spectrum includes two peaks, which are D peak and G peak. By deconvolving the spectrum, the peak positions and their

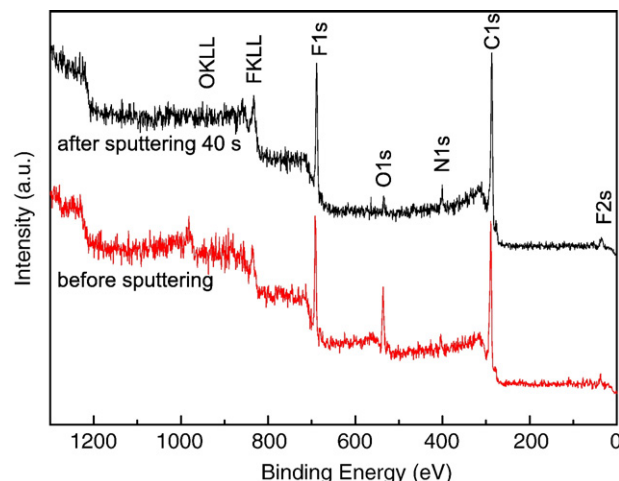


Fig. 3. Typical XPS survey spectrum of a-C:F films before and after sputtering 40 s.

intensities can be obtained. From the G peak position and intensity ratio of D peak to G peak ($I(D)/I(G)$), the sp^2 cluster content and its size can be calculated [12]. For all samples in our experiment, the characters of Raman spectrum are similar: the G peak position ranges between 1583 and 1590 cm^{-1} , and the value of $I(D)/I(G)$ ranges between 1.0 and 1.17. According to literature [12], the results indicate that the size and content of sp^2 cluster in a-C:F films are similar, and the sp^3 content in the film is low. It is believed that amorphous graphitic sp^2 cluster is the main component of C–C network in the deposited a-C:F films. Table 1 lists the deposition parameters, modulus and hardness from nano-indentation measurement, proportion of fluorocarbon groups from XPS results. Since the modulus and hardness of a-C:F films are mainly determined by the character of C–C network, the values of them are close to those of polycrystalline graphite, whose modulus and hardness are 0.5 GPa and 20 GPa measured by indentation test, respectively.

In order to remove the impurity elements absorbed from the air during transporting from deposition vacuum chamber to XPS analyzing chamber, a short time Ar ion sputtering cleaning was carried out before XPS analysis. Fig. 3 shows a typical XPS spectrum from a-C:F film before and after 40 s sputtering. The elements of C, O, N, F are detected by XPS before sputtering; and after 40 s sputtering the peaks intensity of O1 s and N1 s

Table 1
Hardness, modulus, and the proportion of C–F unit and $-(CF-CF)_n-$ unit in C1s spectroscopy of films prepared under different deposition parameters

RF power W	Target-sample distance mm	Ar flux sccm	Modulus GPa	Hardness GPa	Proportion of C–C group %	Proportion of C–CF group %	Proportion of C–F group %	Proportion of $-(CF-CF)_n-$ group %
200	50	80	21	0.8	60.7	21.6	13.1	4.6
		160	22	0.9	56.0	23.8	15.9	4.3
		200	14	0.6	59.8	21.1	13.0	6.1
300	50	60	6	0.4	55.7	19.1	14.0	9.5
		60	11	0.6	61.1	18.2	13.1	7.6
		160	14	0.6	64.5	17.1	13.3	5.1
400	50	200	9	0.5	51.6	23.1	14.4	8.1
		60	14	0.7	55.3	20.7	14.8	6.8
		80	14	0.6	55.0	21.0	16.5	7.5
		160	8	0.5	41.3	27.9	17.2	10.2

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