



Study on structure, mechanical and tribological properties of iron incorporated diamond-like carbon films



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ABSTRACT

In current paper, iron incorporated diamond-like carbon films were deposited successfully via direct current reactive magnetron sputtering process at different methane flow rates. The morphologies, structures and phases of the Fe-DLC films were studied by field-emission scanning electron microscopy, X-ray photoelectron spectra, X-ray diffraction and Raman spectroscopy, and their mechanical properties were investigated using nanoindentation tester and scratch tester. In particular, the friction and wear behaviors of as-deposited films were evaluated both in the ambient air and deionized water conditions via reciprocating sliding tribometer. It revealed that Fe₃C nanocrystallines were generated, and appropriate content of Fe₃C nanocrystallines in the amorphous carbon matrix could improve the hardness and adhesion strength of the films. As a result, the iron incorporated diamond-like carbon film deposited at methane flow rate of 18 sccm could possess relatively higher hardness of 14.4 GPa, elastic modulus of 134.7 GPa and best adhesion strength with critical load of 38 N. Especially, it presented superior friction and wear performances both in ambient air and deionized water conditions. In comparison with as-deposited films tested in ambient air condition, each sample exhibited more excellent tribological performances with lower friction coefficient and wear rate in deionized water condition.

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1. Introduction

Diamond-like carbon (DLC) films have attracted a great deal of attention in industries as protective film, which cover a wide range of industrial applications to provide low wear and low friction for moving mechanical components [1–4]. However, the main obstacle is a high internal stress to practical applications for DLC films [5]. Because high internal stress makes it difficult for DLC films to comply with substrate deformation, which can lead to the films peeling off from substrates and massive failure of the films during the deposition or in service. Addition of metal atoms in the carbon matrix as an effective strategy has been proved to reduce internal stress and increase adhesion of DLC films on substrates. Recently, incorporation of transition metal elements which exist in the form of solid solution atoms, small nanocrystallines of pure metal or metal carbide, such as Ti, Cr, W, Fe and Al, in DLC films have been extensively researched. The metal atoms embedded into the amorphous carbon network can significantly influence the mechanical and tribological properties [6–8]. Fe as the incorporated metal element exhibited a strong bond with the carbon matrix which could form hard carbide phases with functional characteristics to improve the tribological properties of films. According to the reports, the iron incorporated DLC (Fe-DLC) films were deposited by different techniques,

such as magnetron sputter deposition, pulsed laser deposition (PLD) technique, electrochemical technique, etc. [9–11]. Reactive magnetron sputtering is one of the most often used deposition technique for metal incorporated DLC (Me-DLC) films, and the reactive magnetron sputtering technique can offer a lot of advantages such as mass production, low temperature deposition, and convenient metal incorporation. In general, Me-DLC films prepared by reactive magnetron sputtering technique had superior mechanical and tribological properties. Also, previous studies of the Fe-DLC films mainly focused on the field emission, electronic and magnetic functional properties [12–15]. However, some investigations on structure and tribological properties of the Fe-DLC films were seldom reported.

In present work, the Fe-DLC films were successfully prepared on Si(100) wafers and stainless steel at different methane (CH₄) flow rates by direct current (DC) reactive magnetron sputtering. The morphology, structure, mechanical properties of the Fe-DLC films were systematically investigated, especially, their friction and wear behaviors were evaluated both in ambient air and deionized water conditions.

2. Experimental details

2.1. Deposition process

The Fe-DLC films were deposited by DC reactive magnetron sputtering of two pure Fe target in a gas mixture of Ar and CH₄. The

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doped Fe concentration was controlled by varying the CH₄ flow rate. Prior to be fixed onto the substrate holder in the chamber, all the Si(100) wafers and mirror polished stainless steel substrates were cleaned ultrasonically in acetone and ethanol successively, and then dried in nitrogen blow. Then, the chamber was pumped to pressure below 2×10^{-3} Pa. A pure Fe interlayer was deposited for 30 min in Ar atmosphere before depositing the Fe-DLC films in order to enhance the adhesion between the Fe-DLC films and the substrates. The main deposition parameters of the Fe-DLC films were summarized below: two pure Fe target ($\Phi 60 \times 5$ mm) power of 160 W, Ar flow rate of 50 sccm, the bias potential of -300 V and no intentional substrate heating applied to the substrate, the CH₄ flow rate varied from 12 to 20 sccm while a constant working pressure of 2 Pa. The deposition time was kept at about 90 min for all the samples.

2.2. Characterization techniques

The chemical information of the deposited films was determined by X-ray photoelectron spectroscopy (XPS) (AXIS-ULTRA, Kratos) with monochromatic Al K α radiation. Before the testing, the surface of the samples was cleaned by Ar⁺ ion beam to remove adventitious contaminants. The cross-section structure of films was analyzed by a field emission scanning electron microscope (FESEM) (Hitachi S4800). The crystalline structure of films was determined by X-ray diffraction (XRD) (Bruker D8 X-ray facility). Raman spectroscopy system (HR800) with a wavelength of 532 nm was used to investigate the carbon atomic bond structures of films. The hardness and elastic modulus were measured using a nanoindentation tester (MTS Nano-Indenter G200) with a Berkovich indenter in a continuous stiffness measurement mode. Before performing nanoindentation tests on the Fe-DLC films, area function calibration and standard tests have been done on fused silica to avoid systematic errors. The maximum indentation depth was 1000 nm and the characteristic hardness of the films was chosen to be that which was measured at a depth of around 1/10 of the film thickness in order to minimize the substrate contribution. Each presented hardness value was obtained from four times measurement results. The residual stress was measured by the stress-induced bending on an interferometric surface profiler. The curvature radii of the substrate before and after the film deposition were measured by the observation of Newton's rings using an optical interferometry system, and the internal stress was calculated by the Stoney equation. The adhesion of the samples was analyzed by a scratch tester (Revetest CSM) with a conical diamond tip of a 0.2 mm radius and 120° taper angle as the scratching stylus. The measurement parameters were as follows: normal loading scale of 0 to 60 N, fixed loading rate of 40 N/min, scratching speed of 2 mm/min and scratch length of 3 mm under ambient air condition. The acoustic emission signal was collected when the films were broken and the load at the point of breaking was called the adhesive critical load (L_c). The microstructure of the scratch track was examined by an optical microscope. The friction coefficients and wear rates of the samples were evaluated with a Rtec tribometer (USA) reciprocating sliding tribometer in ambient air of a relative humidity $55 \pm 5\%$ and deionized water at 25 °C. Si₃N₄ ball with diameter of 3 mm was used as the mating material. A sliding speed of 20 mm/s, a constant normal load of 5 N and a sliding stroke of 5 mm were used in the experiments. The friction coefficient was continuously recorded during testing. The morphology of the wear traces and the worn surfaces of the tested Si₃N₄ balls were examined using a scanning electron microscope (SEM) FEI-Quanta FEG250. Based on the wear trace depth profiles measured by an Alpha-Step IQ profilometer, the wear volumes can be obtained after the sliding tests were completed. The wear rate could be calculated in accordance to the equation: $K = V / FS$. V is the wear volumes of the films, S is the sliding length on the films and F is the applied load.

3. Results

3.1. Microstructural characterization

The chemical composition of Fe-DLC films prepared at different CH₄ flow rate determined by XPS. As the CH₄ flow rate increased from 12 to 20 sccm, the Fe content in the as-deposited films decreased from 13.8 to 1.7 at.% and the C content increased from 69.8% to 95.9%. Besides, oxygen is also demonstrated in the as-deposited Fe-DLC films. The presence of oxygen might be attributed to the unavoidable oxygen contamination during the film deposited in the limited vacuum and the surface oxidation when exposed to air. Fig. 1 shows the high-resolution XPS C1s spectra of as-prepared Fe-DLC films, it can be seen that there is a major peak around 284.7 eV. In general, the C1s core peak can be deconvoluted into three Gaussian peaks around 283.3 eV, 284.4 eV and 285.2 eV corresponding to Fe—C bonds, sp² and sp³ bonded carbon, respectively [16–17]. With the increase of CH₄ flow rate, the C1s peak shifted towards higher binding energy implying increased sp³ bonds content in the films. When the film deposited at CH₄ flow rate of 20 sccm, the peak around 283.3 eV distinguished unclearly, indicating almost no formation of Fe—C. As the CH₄ flow rate decreased, the peak around 283.3 eV appeared a little bit, indicating some carbide was formed in the films. XPS Fe 2p spectra of Fe-DLC films appeared Fe—O bonds around 709.5 eV. According to the XPS results, the Fe could exist in the form of some carbide and unavoidable oxide in as-deposited films.

Fig. 2 presents the cross-sectional morphology of the Fe-DLC films deposited at different CH₄ flow rates. The bottom Fe interlayers were typical columnar structure. With increasing CH₄ flow rate, the structure of the top Fe-DLC films changed from less dense and rough to more dense homogenous and smooth. Furthermore, there is no localized delamination in all Fe-DLC films after undergoing mechanical breaking. The thicknesses of the films were 2133 nm, 1935 nm, 1390 nm, 1092 nm, 942 nm, for the films deposited at CH₄ flow rate from 12 sccm to 20 sccm, respectively. The highest growth rate of Fe-DLC film was 23.7 nm/min when the CH₄ flow rate was 12 sccm, while the growth rate decreased as the CH₄ flow rate increased.

The XRD pattern of the Fe-DLC films was depicted in Fig. 3(a). Diffraction peaks at 33.0° and 62.0° 2 θ correspond to reflections from (102) and (222) planes for the phase of Fe₃C, respectively. Compared to the positions of the diffraction peaks against that of the International Committee for Diffraction Data (ICDD) cards, all peaks positions of Fe₃C were lower due to internal compressive stress existed in the films. As can be seen from Fig. 3(a), the intensity of the peak of iron carbide decrease continuously with the CH₄ flow rate increased from 12 to 20 sccm. When the CH₄ flow rate was 20 sccm, no diffraction peaks were observed. Moreover, no carbon diffraction peaks were detected

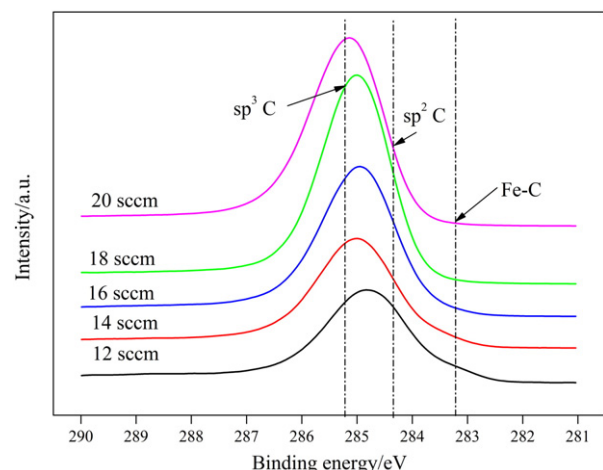


Fig. 1. XPS C1s spectra of Fe-DLC films deposited at different CH₄ flow rate.

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