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Nanoscratch properties of extremely thin diamond-like carbon films

Shojiro Miyake*, Shohei Yamazaki

Nippon Institute of Technology, 4-1 Gakuendai, Miyashiro-machi, Minamisaitama-gun, Saitama 345-8501, Japan

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ABSTRACT

Scratch properties of extremely thin (targeted at 0.03–5.0-nm-thick) diamond-like carbon (DLC) films deposited by the filtered cathodic vacuum arc (FCVA) and electron cyclotron resonance chemical vapor deposition (ECR-CVD) methods are investigated. The difference in profiles and friction coefficients of scratches in both the corn and edge directions are evaluated. The difference of scratch properties between deposition methods is clearly evaluated by the scratching in the corn direction. When scratching in the corn direction, the friction coefficient and wear depth of the FCVA-DLC film increased rapidly at critical load, whereas those of the ECR-CVD-DLC film increased gradually even beyond this critical load. These results are deduced to be caused by the differences in the hardness and brittleness of the films. The dependence of nanoscratch friction force and scratch profile on DLC film thickness can reveal differences in mechanical properties that correspond to atomic-scale thickness.

1. Introduction

Nanotribology is a key technology in the micro- and nanotechnology that includes micromachines and magnetic recording head–medium interfaces. In data storage devices, atomic-scale wear and minute friction fluctuations degrade equipment performance [1,2]. Therefore, improvements in nanotribology and magnetic head–medium mechanics are crucial for the future of the fast-growing magnetic recording industry. This is why methods for the modification of extremely thin surface layers play an important role in realizing higher-reliability magnetic storage devices [1–3].

A reduction in the effective magnetic spacing at the magnetic head–disk interface requires a reduction in the thickness of the protective film, which otherwise will lead to magnetic loss. Therefore, extremely thin protective layers have become important for ensuring the reliability of magnetic disk equipment [1,2]. Although diamond-like carbon (DLC) films are currently used as magnetic disk protective layers [4–7], mechanical endurance is difficult to maintain if a protective film is reduced to approximately 1.0 nm in thickness, which amounts to approximately several layers of atomicity [6,7]. When considering such thin films, the failure characteristics of the atomic bonding during friction and wear should be taken into account [8,9]. It is difficult to maintain tribological endurance when only several atomic layers of the protective film are involved. To this end, the mechanical properties of a few atomic protective layers should be evaluated.

Atomic force microscopy (AFM) is useful for tribological evaluation at the atomic scale, thereby enabling new understanding of the mechanisms of friction and wear. For example, AFM has been used to demonstrate nanoscale testing [10–15]. In particular, the nanoscratch techniques using a diamond tip can be employed to study mechanical properties at the nanometer scale on the surface of materials. The so-called microwear or nanowear properties are investigated by nanoscratch test using an atomic force microscope. The scratch parameters such as applied load, friction force, and scratched groove profile are experimentally evaluated. In general, in a scratch test, a diamond tip moves over a sample under a normal load that is increased until a failure is observed; the corresponding load, called the critical load, is obtained at this point.

Most research concerning mechanical properties of materials has dealt mainly with the friction, wear, and critical load of the surface of materials. In scratch tests [2], tip geometry effects on deformation behavior are investigated using a diamond tip. Some of this work has been done at the nanometer scale using a molecular dynamics (MD) simulation method [16–18]. However, the tips used were ideally sharp three-sided pyramids. Because the hemisphere at the top of the AFM pyramidal diamond tip affects the nanoscratch properties, the effects of tip geometry on the scratch properties at different scratch depths vary. Under atomic-scale evaluation, the effects of tip geometry cannot be ignored. However, a few experimental reports on this problem have been published, so the investigation of effects of tip geometry effects would be meaningful.

In this research, nanoscratch properties of extremely thin DLC films deposited by filtered cathodic vacuum arc (FCVA) and electron cyclotron resonance chemical vapor deposition (ECR-CVD) methods are evaluated by AFM. First, the effects of scratch direction on AFM nanoscratch tests are evaluated, and then, the differences between

* Corresponding author. Tel./fax: +81 480 33 7727.
E-mail addresses: saburo@nit.ac.jp, gosaburo@gmail.com (S. Miyake).

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the DLC deposition methods are evaluated. We establish the relationship between friction coefficient and scratched groove profiles of extremely thin DLC films, and then, analyze the dependence of these properties on film thickness.

2. Deposition and evaluation methods of DLC films

2.1. Deposition of extremely thin DLC films

Extremely thin DLC protective films were deposited on silicon wafers [Si (1 0 0) surfaces] by the FCVA and ECR-CVD methods. The targeted thicknesses of the thin protective DLC films deposited on Si (1 0 0) surfaces were 0.03, 0.4, 0.8, 1.0, 2.0, 5.0, and 100 nm, and the thickness was varied by changing the deposition time. Transmission electron microscopy (TEM) analysis was used to determine the thickness of each DLC thin film. The composition of the films was determined by Auger electron spectroscopy (AES) profile depth analysis [6].

2.2. Nanoscratch method

To evaluate the nanometer-scale mechanical properties of these DLC films, a nanoscratch test was performed using an atomic force microscope (Digital Instruments Nanoscope III with Hysitron indentation system). In the scratch tests, a diamond indenter slides on a specimen, generating a groove. The following three factors contributed to the friction force generated: the adhesion force occurring at the elastic contact areas, ploughing force, and removal of a wedge or chip. In the nanoscratch process, the size of the wedge in front of the diamond tip is comparable to the scratch depth, so the effect of the wedge on friction cannot be neglected. However, it is very difficult to assess this effect quantitatively in experiments.

We performed an increasing-load nanoscratch test and determined the differences in the scratch profile and friction coefficient for film failures on DLC films deposited by both deposition methods. The load increased in proportion to the position from the starting point of loading up to a length of 4 μm. A diagram of the nanoscratch test and its measurement model are shown in Fig. 1(a) and (b), respectively. Scratch tests of 4-μm length were captured using an atomic force microscope, and then, the scratched surface profiles were observed in the 5 μm × 5 μm test area. In addition, the friction force of the nanoscratched DLC films was evaluated.

First, maximum test load was set to 1000 μN, 500 μN, and 300 μN by changing the scratch direction. The directions of a scratch can be described as corn and edge of a Berkovich-type diamond indenter tip with 100-nm radius as shown in Fig. 1(a). Scratch tests were performed to investigate the effect of change in friction force on load. The adhesion friction force, ploughing friction force, and contribution of the wedge to friction force on the nanoscale are estimated and thoroughly studied with different friction forces and scratch depths for both DLC deposition methods. The dependence of the nanoscratch properties of the FCVA and ECR-CVD deposition methods on DLC film thickness is investigated. As a means of assessing reproducibility, the examination was performed more than three times, and the mean and typical results are discussed.

3. Results and discussions

3.1. Thickness, structure, density, and composition of deposited films

The properties such as real thickness, structure, density, and composition of these DLC films were evaluated by AES, TEM, Rutherford back-scattering spectroscopy (RBS), and Raman spectroscopy [6,7]. Raman spectroscopy showed that the FCVA-DLC and ECR-CVD-DLC films have tetrahedral hydrogen-free structures and amorphous hydrogen-containing carbon films, respectively. RBS showed the densities of these FCVA-DLC and ECR-CVD-DLC films to be 3.30 g/cm^3 and 1.87 g/cm^3, respectively, so the density of the FCVA film was 1.7 times larger than that of the ECR-CVD film.

The relationship between the actual thickness and target deposition thickness was evaluated by TEM and AES methods. After the thickness of each DLC thin film was confirmed by TEM, the deposition rate was calculated as shown in Fig. 2. The deposition rates showed that each deposition method produced extremely thin DLC films of similar target thickness, yet the DLC material had a thickness several times that of the atomic diameter of carbon. For example, for depth profiles of 2.0-nm-thick DLC films, in which carbon reaches the same value as Si, the carbon depth is 1.9 nm and 1.6 nm in the FCVA-DLC film and ECR-CVD-DLC film, respectively. These values are almost close to the 2.0 nm target thickness of the film. The surface roughness (Ra) of these films was evaluated by a non-contact mode atomic force microscope (SII SPA300HV) using a carbon nanotube tip. The FCVA-DLC films appeared to be free of microparticles and had low surface roughness, as did the ECR-CVD-DLC films. The roughness of these DLC films was as low as 0.09–0.2 nm Ra and was similar to that of the Si substrate.

3.2. Nanoscratch properties

3.2.1. Scratch properties dependence on scratch direction

Fig. 3(a) shows a cross-section AFM image of a scratch groove in a 1.0-nm-thick ECR-CVD-DLC film. The scratch depth in the corn direction is shallow in the beginning, and then, it increases. On the other hand, in the edge direction, scratch depth increases gradually. Fig. 3(b) shows a cross-section AFM image of a scratch in a 1.0-nm-thick FCVA-DLC film. Scratch depth in the corn direction shows the tendency to increase rapidly at a certain inflection depth, and a rough upheaval profile due to brittle fracture is observed in the vicinity of the scratch terminal. As for the corn direction, the wear debris formed as wedge could be observed near the end of the scratched groove. On the other hand, in the edge direction, the scratch depth increases gradually.

The dependence of cross-section profiles and the friction coefficients of 1.0-nm-thick ECR-CVD-DLC and the FCVA-DLC films on the corn and edge directions are shown in Fig. 4(a) and (b), respectively. The scratch profiles and friction coefficients during three test runs show good reproducibility. When scratching in the corn direction, there was large difference in the properties of FCVA-DLC and ECR-CVD-DLC films. In addition, the wear depth of FCVA-DLC film increased rapidly from a certain depth, and at this point, the friction coefficient also increased rapidly. In the edge direction, the wear depth and friction coefficients of both ECR-CVD-DLC and FCVA-DLC films show a similar tendency to increase gradually.

At the first stage of low-load contact between the diamond tip and film-coated specimen, the contact was elastic, so no groove was scratched and the wear depth was nearly zero. Under such a condition, only adhesion force acted. As the load was increased, ploughing began to occur, plastic deformation ensued, and friction increased. With further load increase, the scratch depth increased to near film thickness, and then, friction force and wear depth increased rapidly.

The friction coefficients at first were as low as 0.1. There were differences in frictional properties between the scratch directions and scratch depths. For scratching in the corn direction, the diamond tip contacted elastically at first, and then, ploughing occurred at a constant load. However, at a higher load, the friction coefficient of FCVA-DLC films was greater than that of ECR-CVD-DLC films. In the FCVA-DLC films, a wedge accumulated forward of the corn surface of the tip, and the wear depth increased rapidly. There was a clear difference in the scratch profiles of FCVA-DLC