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# Dependence of the friction durability of extremely thin diamond-like carbon films on film thickness

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ABSTRACT

Tribological durability properties of extremely thin diamond-like carbon (DLC) films (thickness 0.03–5 nm) deposited using filtered cathodic vacuum arc (FCVA) and plasma-chemical vapor deposition (P-CVD) methods were evaluated using load-increase-and-decrease, ball-on-disk, and low-load reciprocating friction tests. Friction durability notably increased at a certain film thickness for FCVA-DLC and P-CVD-DLC films. These thicknesses were nearly equal to surface roughness and corresponded to film thickness at which the nanowear profiles changed from protuberance to grooves and at which nano-scratch resistance increased. Excellent friction durability of FCVA-DLC films (thickness ≥ 0.4 nm), evaluated by rapid increase in the friction coefficient, was observed. In contrast, the friction durability of P-CVD-DLC films gradually increased when the film thickness was 0.6 nm or greater. When the thickness of the DLC films was 2.0 nm or greater, the films did not exhibit a rapid increase in their friction coefficient within the total number of testing cycles. The tribological properties of extremely thin DLC films depend on film thickness; extremely thin FCVA-DLC films exhibit excellent wear resistance. The film thickness at which FCVA-DLC films endured the total number of test cycles was approximately one-fifth the corresponding thickness of CVD-DLC films, evaluated by the three different friction tests.



## 1. Introduction

The development of high-density recording magnetic disk systems has advanced in recent years. In magnetic disk devices, a diamond-like carbon (DLC) film is usually applied as a protective layer to the magnetic recording head and the disk [1–7].

Reducing magnetic losses associated with an increase of the memory density requires that the magnetic space at the magnetic head–disk interface be reduced, which, in turn, requires a reduction in the thickness of the DLC protective film [5–9]. Improvement in the tribological durability of the magnetic head–disk interface is important for the future of the magnetic recording industry. The tribological properties of the protective film, such as its durability, are very important in preventing fracture by contact sliding, and the importance of these properties increases as the thickness of the protective film decreases. Therefore, methods of depositing extremely thin protective films such as DLC films onto magnetic heads and disks play an important role in realizing higher-reliability magnetic storage devices.

Typically, a protective film with a thickness of approximately 1 nm is required to achieve higher-density magnetic storage. However, maintaining mechanical durability with such extremely thin protective films wherein a thickness of 1 nm corresponds to approximately seven atomic layers of carbon atoms is difficult. That is, in the case of 1 nm-thick protective films, the durability of the films when subjected to friction and wear should be considered. The plasma chemical vapor deposition (P-CVD) method [6,7] is used to deposit the thin films onto magnetic disks. In addition, tetrahedral DLC (ta-C) [8–10] thin films deposited by filtered cathodic vacuum (FCVA-DLC) are expected to be applied to magnetic disks because of their higher hardness and density [10–13].

In a previous study, we evaluated the nanotribological properties of extremely thin DLC films using atomic force microscopy (AFM) [14,15]. We successfully evaluated the difference between the nanotribological properties of approximately 1 nm-thick FCVA-DLC and P-CVD-DLC films and clarified that FCVA-DLC films are hard and exhibit superior nanowear resistance. The FCVA-DLC films were observed to exhibit greater resistance to plastic wear than the P-CVD-DLC films.

In this study, the film-thickness dependence of the tribological properties—especially the durability—of extremely thin DLC films deposited by FCVA and P-CVD methods dependence are evaluated

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using load-increase-and-decrease, ball-on-disk, and low-load reciprocating friction tests. We then discuss the mechanism responsible for the difference in durability between these films.

## 2. Experimental method

### *2.1. Samples*

DLC films with thicknesses of 0.03, 0.1, 0.2, 0.3, 0.4, 0.6, 0.8, 1.0, 1.5, 2.0, 5.0, and 100 nm were deposited onto Si(100) wafers using the FCVA and P-CVD methods [14–18]. In this research, in order to clarify the film thickness at which friction durability is achieved, we evaluated the dependence of the durability on film thickness. For depositing the extremely thin DLC films, we deposited DLC films of various thicknesses, including the insufficient initial layer, by changing the deposition time, e.g., the 0.03- and 0.1 nm-thick layers are thinner than the C–C covalent bond length (0.154 nm); thus, a uniform film could not be formed. However, the friction durability of the 0.03- and 0.1 nm-thick initial layer of DLC film is important for the purpose of this study in order to clarify the critical thickness at which durability can be achieved. Details of the films deposited in this work are shown in Table 1. The C–C bond length is approximately 0.154 nm; therefore, a 0.3 nm-thick layer of carbon atoms corresponds to a thickness of two atoms. At thicknesses less than 0.1 nm, less than a monolayer carbon, on average, is deposited on the Si substrate. The model of a 1 nm-thick DLC film coated onto a flat Si surface is approximately seven carbon atoms thick, as shown in Table 1. Films of different thicknesses were obtained by changing the deposition time, and transmission electron microscopy (TEM) analysis was used to determine the thickness of each DLC thin film. Evaluation of the deposition rate confirmed that DLC films with thickness approaching the target thicknesses were deposited. The composition of the films varied with the depth and was determined by Auger electron spectroscopy (AES) profile depth analysis [14]. Surface analysis evaluation did not clearly reveal the structure and profile of the initially deposited layer of DLC film. We attempted to observe the 0.03- and 0.1 nm-thick DLC films using TEM; however, neither clear layers nor patched profiles could be observed. Subsequently, we attempted measurement of the profile, friction distribution, and electric current distribution using scanning probe microscopy; however, no clear difference was observed between these extremely thin 0.03- and 0.1 nm-thick films. Therefore, we investigated the dependence of the friction durability and nanomechanical properties on target film thickness. The surface profiles were observed by scanning the diamond tip with a radius of approximately 150 nm over an area of 1 μm × 1 μm. Surface three-dimensional (3D) parameters such as Sp (maximum height of peaks), Sz (maximum height of the surface) and Sa (arithmetical mean height of the surface) were evaluated by atomic force microscopy (AFM).

### *2.2. Friction and oscillating scratch tests*

The three friction tests—load-increase-and-decrease, ball-on-disk, and low-load reciprocating friction tests—were performed as shown in Fig. 1(a)–(c). The dependence of the durability of the films on their thickness was mainly evaluated. The test conditions used in the three friction tests are shown in Table 2. The contact pressures used and total cycles performed during the three friction tests were varied to allow the dependence of the films' durability on their thickness to be evaluated. The real Hertzian contact stress of the DLC-coated Si wafers was difficult to evaluate because the DLC films deposited onto the Si substrates were extremely thin. Therefore, the contact pressures on the Si substrates were evaluated using Young's moduli and Poisson's ratio of Si and each opposite tip material; these contact stresses are presented in Table 2.

#### *2.2.1. Load-increase-and-decrease friction tests*

Load-increase-and-decrease friction tests (Shinto Scientific Co., Ltd., HHS2000) were performed as shown in Fig. 1(a) to evaluate the dependence of the friction coefficient of extremely thin DLC films on the number of sliding cycles, the applied load, and the number of durability cycles. A sample was fixed on the stage, which reciprocates. The load was increased and decreased when the stage reciprocated by the load increased or decreased gradually on the opposite ball specimens, and the friction force was measured. The test conditions were a reciprocating speed of 5 mm/s and a reciprocating slide length of 10 mm. The load was increased and decreased between 0.05 and 0.5 N during each cycle. The total number of reciprocating cycles was 100. The opposite specimen was $Al_2O_3$–TiC ball with a diameter of $\Phi$2 mm. The contact pressure of the load during the load-increase-and-decrease friction tests varied from 0.30 to 0.65 GPa.

#### *2.2.2. Ball-on-disk friction tests*

A ball-on-disk friction tester (Rhesca Co., Ltd., FPR-2000) was used to evaluate the dependence of the friction properties on film thickness by one-way directional sliding, as shown in Fig. 1(b). The test conditions were a load 0.5 N, 2250 total sliding cycle, a turning radius of 4.0 mm, and a friction speed of 31.4 mm/s. SUS440C ($\Phi$6 mm) was used as the opposite specimen. The Hertzian contact stress was 0.27 GPa. The DLC coated sample was mounted onto a stage, which was rotated while a load was applied to opposite specimens.

**Table 1**
Thickness of FCVA-DLC and P-CVD-DLC films.

| Film thickness (nm) | 0 | 0.03 | 0.1 | 0.2 | 0.3 | 0.4 | 0.6 | 0.8 | 1.0 | 1.5 | 2.0 | 5.0 | 100 |
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