



Friction properties of amorphous carbon ultrathin films deposited by filtered cathodic vacuum arc and radio-frequency sputtering



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ABSTRACT

The friction properties of ultrathin films of amorphous carbon (*a*-C) deposited on Si(100) substrates by filtered cathodic vacuum arc and radio-frequency sputtering were investigated by surface force microscopy. Deposition parameters yielding *a*-C films with high sp^3 content were used to deposit films of thickness between 5 and 35 nm. The coefficient of friction of both types of *a*-C films was measured with a 1- μ m-radius conical diamond tip and normal loads in the range of 20–640 μ N. The results show a strong dependence of the friction properties on the surface roughness, thickness, and structure of the *a*-C films, which are influenced by the intricacies of the deposition method. The dependence of the coefficient of friction on normal load and the dominance of adhesion and plowing friction mechanisms are interpreted in terms of the through-thickness variation of carbon atom hybridization of the *a*-C films.

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1. Introduction

Amorphous carbon (*a*-C) films are of particular interest because their unique properties (i.e., high hardness, low friction, high wear resistance, chemical inertness, low magnetic susceptibility, and large optical band gap) are critical to many industrial and medical applications, including magnetic storage devices, microelectromechanical systems, and biomedical/implantable components [1–4]. Several deposition methods have been used to synthesize carbon films, such as radio-frequency (RF) sputtering, ion-beam deposition, laser ablation, and filtered cathodic vacuum arc (FCVA). Depending on the deposition method and corresponding process parameters, the film quality is a strong function of the film roughness, uniformity, and structure (carbon atom bonding). Considering the industrial relevance of thin *a*-C films, characterization of the friction properties of these films is of high significance.

Among various cutting-edge technologies, thin *a*-C films are extensively used in magnetic recording where maintaining the integrity of stored data is critical. In hard-disk drives, information is stored in the magnetic medium (e.g., CoCrPt or FeNi) of a hard disk by the magnetic field of a read/write transducer embedded at the trailing edge of the head. To preserve the reliability of magnetic storage devices, thin *a*-C films are used as overcoats to protect the hard disk and magnetic head against mechanical wear and corrosion [5]. In addition, a lubricant monolayer adsorbed onto the carbon overcoat reduces adhesion at the head-disk interface, whenever intermittent asperity–asperity contact

is encountered during operation. Because of the exponential increase in data storage density with decreasing magnetic spacing, the trend is to reduce the physical spacing between the read/write transducer and the magnetic medium by thinning the carbon overcoat to a few nanometers. With such thin overcoats, the storage density may be increased from several hundreds of Gbit/in² to several Tbit/in² [6–8]. Decreasing the carbon overcoat thickness to a few nanometers, while preserving the mechanical and tribological properties and preventing corrosion of the magnetic medium, presents a challenging task of high technological importance.

The structure of *a*-C films is characterized by the short-range order of carbon atoms in linear (sp^1), trigonal (sp^2), and tetrahedral (sp^3) configurations [9]. While sp^2 hybridization (graphite-like) has both σ and π bonds, sp^3 hybridization (diamond-like) has only σ bonds. The mechanical properties depend on the stronger σ bonds, while the electrical and optical properties are mostly affected by the weaker π bonds [10]. Hard *a*-C films with high sp^3 contents form three-dimensional metastable amorphous networks exhibiting various levels of intermediate-range order [11]. Correlating the sp^3 content with the friction characteristics of thin *a*-C films requires the use of surface-sensitive probe-based mechanical testing and knowledge of the through-thickness film structure.

Several deposition methods can be used to synthesize thin *a*-C films including FCVA, pulsed laser deposition, plasma-assisted chemical vapor deposition, and RF sputtering [12–16]. The film structure and mechanical properties depend on the deposition method and corresponding process parameters, such as plasma power, substrate bias voltage (ion kinetic energy), ion fluence, and ion incidence angle. The mechanical and tribological properties of *a*-C films can be influenced by the film structure (sp^3/sp^2 ratio), hydrogen content, and strength of adhesion to

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the substrate, all of which depend on the particular deposition method. In general, high sp^3 content correlates with high hardness [17]. The sp^3/sp^2 ratio decreases in the following ranking order of deposition method: FCVA, pulsed laser vaporization, direct ion-beam deposition, plasma-enhanced chemical vapor deposition, ion-beam sputtering, and RF sputtering [18].

Despite significant advances in thin-film deposition methods and characterization techniques, basic knowledge of the effects of film roughness and structure, which depend on the process conditions of the particular deposition method, on the friction characteristics of thin a -C films is inadequate. Consequently, the main objective of this study is to elucidate the dependence of the friction properties of thin (5–35 nm thick) a -C films deposited by FCVA and RF sputtering on corresponding surface roughness, thickness, and structure. The key deposition parameters of each process were configured to maximize the sp^3 content of both FCVA and sputtered a -C films. The friction behavior is interpreted in terms of adhesion and plowing mechanisms affected by the surface roughness and through-thickness structure of the deposited films.

2. Experimental procedure

Substrates for both FCVA and RF sputtering deposition experiments were obtained by sectioning p-type Si(100) wafers into $10 \times 10 \text{ mm}^2$ pieces. The substrates were then cleaned by rinsing with isopropanol for 10 min and with acetone also for 10 min and, finally, blow drying with nitrogen gas.

2.1. Filtered cathodic vacuum arc

FCVA is ideal for low-temperature deposition of continuous, thin, and smooth films on various substrates without the need of an adhesion underlayer. Technical improvements (especially plasma stabilization and particle filtering) have offset early problems with plasma instabilities, difficulties with particle filtering, and poor film adhesion to electrically insulating substrate materials [19]. The FCVA system used in the present study utilizes a vacuum arc plasma source to deposit the film onto the substrate. The process relies on the ignition of a vacuum electric arc between an anode and a cathode consisting of a high purity material (99.99% pure graphite in this study). During arc discharging, electrons flowing from the cathode generate both pressure and electrical potential gradients, refocusing the arc on fluctuating small spots on the cathode surface. The resulting pressure gradients close to the cathode surface result in the ejection of the cathode material in the form of plasma, which is then guided by upstream, auxiliary, and downstream coils toward a rotating substrate holder to ensure uniform film deposition. Four orthogonally mounted raster coils positioned outside the downstream coil are used to raster the plasma beam. To stabilize the arc discharges, a “cusp” configuration magnetic field is generated by superposing the magnetic fields generated by the cathode and upstream anode coils [19,21]. The out-of-plane S-shaped configuration of the magnetic filter prevents macroparticles and/or droplets that may be ejected from the cathode from depositing onto the film surface. Water cooling removes any excess heat from the substrate holder. The film quality strongly correlates with the C^+ ion energy and substrate bias voltage. The increase of the ion energy causes intense collisions of the C^+ ions with substrate atoms, promoting sp^3 hybridization. Too high of an ion energy, however, can also result in thermal relaxation, which is conducive to $sp^3 \rightarrow sp^2$ rehybridization [22]. Therefore, an optimum C^+ ion energy of $\sim 120 \text{ eV}$ is used to balance these opposing processes and to maximize the sp^3 fraction and hardness of the FCVA films [21].

After mounting the samples onto the substrate holder, the chamber was pumped down to a base pressure of $<5 \times 10^{-7} \text{ Torr}$ by a cryopump. Before initiating film deposition, the native SiO_2 layer and any adsorbents were removed by bombarding the silicon substrate with 500-eV

Table 1
Deposition parameters and thickness of FCVA a -C films.^a

Film thickness (nm)	Incidence angle (deg.)	Substrate bias voltage (V)	Deposition time (s)
6–7	20	–100	30
10	10	–100	12
20	45	–100	30
35	90	–100	30

^a C^+ ion energy $\approx 120 \text{ eV}$.

Ar^+ ions generated by a 64-mm Kaufman ion source. This cleaning step was performed for 2 min at a pressure of $2 \times 10^{-4} \text{ Torr}$ and Ar^+ ion incidence angle equal to 60° relative to the substrate normal. In all film depositions, the base pressure was less than $5 \times 10^{-7} \text{ Torr}$ and a pulsed bias voltage of -100 V and frequency of 25 kHz was applied to the substrate. The deposition time was varied to obtain films of thickness in the range of 6–35 nm. More details about the FCVA system used in this study can be found elsewhere [19]. The thickness of the FCVA a -C films examined in this study versus deposition parameters is given in Table 1.

2.2. Radio-frequency sputtering

RF sputtering enables fast deposition of carbon films at lower working pressures than those of FCVA, resulting in less gas molecule collisions and enhanced line-of-sight deposition. In addition, reversing the polarity prevents charging and reduces arcing. Unlike FCVA, RF sputtering does not produce continuous films with thickness less than 5 nm, because initial film growth is characterized by the formation of small islands, which coalesce to form a continuous film when an effective film thickness of $\sim 5 \text{ nm}$ is reached. Because the sp^3 content of sputtered films is usually less than that of FCVA films, they are usually softer than a -C films deposited by the FCVA method.

In RF sputtering, a glow discharge is produced by an RF power source in a vacuum chamber under pressure-control flow of an inert gas (usually Ar). Sufficient ion momentum transfer to the graphite target surface causes the ejection of carbon atoms or clusters of carbon atoms, which travel through the plasma space and deposit onto the substrate surface to form a carbon film. Because individual atoms are chemically active, an inert gas is used to prevent the formation of undesirable compounds. The build-up of a positive electric charge on the target surface due to impinging plasma ions is avoided by applying an RF voltage to the target assembly, which reverses the polarity by attracting enough electrons from the discharge.

Film deposition by RF sputtering comprised first pumping down the chamber to a base pressure of $<5 \times 10^{-6} \text{ Torr}$ by a cryopump, introducing Ar gas at a flow rate of 20 sccm, and raising the chamber pressure to 3 mTorr by adjusting the throttle valve. Before each film deposition, the graphite target was sputter-cleaned for 10 min and the Si(100) substrate for 3 min to remove the native SiO_2 layer; both processes were performed at 250 W power and 3 mTorr working pressure in pure Ar plasma. The self-biased target voltage ranged from -750 to -850 V during substrate cleaning and between -980 and -1000 V during target cleaning. The film quality strongly correlates with the RF power and substrate bias voltage. An optimum forward RF power of 750 W and a substrate bias voltage of -200 V were used to maximize the film hardness and elastic modulus, while minimizing the surface roughness [22]. Under these sputtering conditions, the bombarding Ar^+ ions effectively eliminate the weakly bonded carbon atoms without damaging the film structure. Therefore, during film deposition the substrate bias voltage and forward RF power were set equal to -200 V and 750 W, respectively. The deposition time was varied to produce films of thickness in the range of 5–35 nm. The thickness of the RF sputtered a -C films examined in this study versus deposition parameters is given in Table 2.

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