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Abstract

It is known that filtered cathodic-arc-deposited ta-C films have outstanding properties even within the family of diamondlike materials. However, filtering of macroparticles is usually incomplete or accompanied by significant plasma losses. Ongoing research effort is directed towards the following goals: (1) complete elimination of macro- and nanoparticles from the vacuum arc plasma, (2) increase of plasma utilization in the cathodic-arc and macroparticle-filter system, (3) precise control and reproducibility of film deposition, and (4) synthesis of ultrathin films (< 5 nm) that meet requirements of the magnetic storage industry. The development of new filters, in particular the "Twist Filter", enables cathodic arc plasma deposition to synthesize ultrathin ta-C films of 3 nm on heads that pass corrosion and other relevant tests. We describe the Twist Filter system and report about recent ta-C tests results. In light of these results, even thinner films seem to be possible.

1. Introduction

Ultrathin (< 5 nm) hard carbon films are used as protective overcoats on hard disks and read-write heads. The tribological properties of the head-disk interface are not only mechanical but also chemical in nature: the overcoat is supposed to protect the magnetic layer against wear and corrosion [1, 2]. As the areal storage density increases at a breathtaking rate, the "magnetic spacing" between the magnetic layer of the disk and read/write sensor of the head must decrease. The magnetic spacing includes magnetically dead layers, carbon overcoats, lubrication, and the fly height. Thinner overcoats allow the head to be closer to the disk, and, hence, the size of individual bits to be smaller. As we work toward an areal storage density of 100 Gbits/in^2, the magnetic spacing approaches the sub-10-nm regime (pseudo-contact recording) and overcoat thickness must shrink to 1-2 nm. Overcoats currently used are sputtered or ion-beam deposited diamondlike carbon films, typically doped with hydrogen or nitrogen. They cease to provide acceptable levels of corrosion protection and wear resistance for films thinner than 5 nm.

Overcoats need to be tough (hard and elastic), chemically inert, pinhole-free, and compatible with the lubricant. The challenge of ultrathin film synthesis is to make the films as thin as possible and still continuous, as opposed to an assembly of islands.

Diamondlike films are characterized by an outstanding combination of advantageous properties: they can be very hard, tough, super-smooth, chemically inert, well adherent to the substrate, and compatible with lubricants. They can be deposited fast, efficiently, at low cost, and on room temperature substrates. The various deposition methods result in a variety of diamondlike films. Widely used is hydrogenated diamondlike carbon (DLC or a-C:H), nitrogen-doped amorphous carbon (a-C:N) or amorphous carbon nitride (CN_x) [3-5], hydrogenated carbon nitride (CH_xN_y), hydrogen-free amorphous carbon (a-C), silicon-doped amorphous carbon (a-C:Si) or silicon carbide (SiC) [6], and metal-doped amorphous carbon (a-C:Me) [7]. Amorphous carbon films (a-C) often have a very high percentage of tetrahedral (sp^3) bonding and therefore they are referred to as tetrahedral amorphous carbon (ta-C).

There are large variations even within each class of materials, depending on the method and parameters of deposition. For instance, ta-C can be synthesized by argon sputter deposition using a graphite target, by pulsed laser ablation of graphite, by mass-selected ion beam deposition of C^+, or by cathodic arc plasma deposition. Research in recent years has identified filtered cathodic arc plasma deposition as one of the most promising techniques [8]. The greatest challenge is the complete removal of "macroparticles" that are generated at the cathode spot.

2. Macroparticle-Free Carbon Plasma

2.1. Cathodic arc carbon plasma

Cathodic arc plasmas are characterized by a very high degree of ionization, with multiply charged ions present [9]. Carbon plasma is dominated by ions of charge state 1+. The plasma ion flows with supersonic velocity of Mach number 5, corresponding to kinetic carbon ion energy of about 54 eV [10]. This very recent result indicates that the carbon ion energy is significantly higher than the 20 eV that has been generally quoted. The ion energy is critical for the quality of ta-C films.

Solid cathode material is transformed to dense plasma that expands while moving away from the spot area. Not only plasma is produced but also debris particles, usually referred to as

"macroparticles." For carbon, large chunks of graphite (fractions of millimeters) are sometime ejected from the cathode – it is believed that shock-heated gas inclusions of the cathode contribute to these massive cathode losses. However, smaller macroparticles are much more frequent, and their distribution extends well down into the nanoparticle range [11]. The term "macroparticles" is used generically for all particles, including nanoparticles. The presence of macroparticles is detrimental to the quality of ta-C films, leading to pinholes and defects that cause ta-C films to fail in corrosion and other tests.

2.2. Macroparticle filters

The most successful approach to macroparticle removal is based on the vast mass-to-charge difference of macroparticles and plasma particles; see reviews [12-15]. The plasma can be guided out of the line-of-sight from the cathode using curved magnetic fields, employing combined magnetic and electric mechanisms for electrons and ions, respectively. The motion of macroparticles is almost independent of the presence of fields; thus they move along straight trajectories, thereby being separated from the plasma.

In the literature, two basic constructions and filter philosophies have been described. They can be described as the closed and open filter architectures. The "classic" 90° filter duct [16] employs a duct, a closed tube, surrounded by a set of magnetic field coils. The interior wall of the duct is equipped with baffles designed to catch or reflect macroparticles. Graphite particles may suffer multiple reflections, and fracture into several "sub-macroparticles." Consequently, there is a significant likelihood that carbon macroparticles, or fractions of them, arrive at the substrate via multiple reflections. Additionally, small nanoparticles may be transported through momentum exchanged with ions ("ion wind"). There are a numerous closed-architecture filters derived from the classic 90° filter, including 45° filters [17], S-filters [18], segmented filters [19], filters of rectangular cross section [20, 21], and out-of-plane double-bent filters [22, 23].

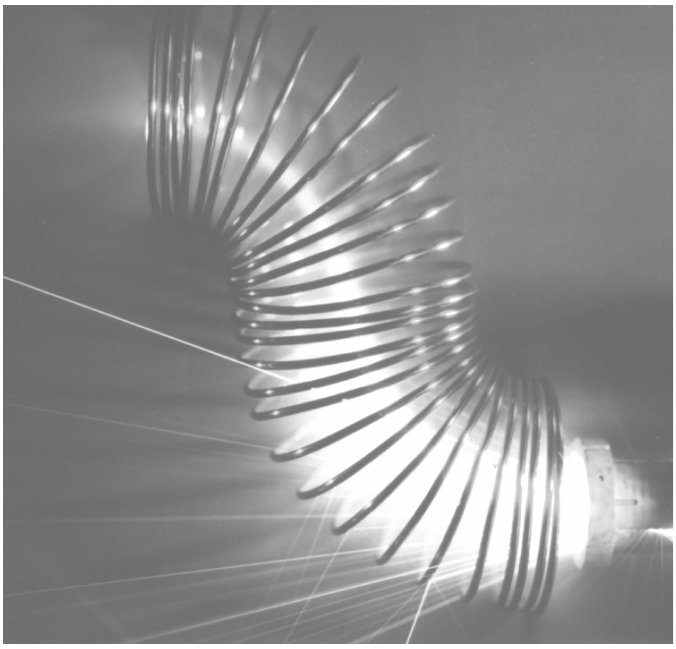

Figure 1 S-shaped open filter guiding carbon plasma from a cathodic arc plasma source (right) to the substrate region (left). Carbon macroparticles are hot and can therefore easily be identified by their bright traces. Open-shutter photograph, arc current 1000 A, pulse length 1 ms.

Filters of open architecture address the issue of particle reflection by removing the particle from the filter and plasma volume via openings [24-26]. Figure 1 shows an S-shaped open filter that consists of a bent solenoid. It is obvious that most macroparticles can leave the plasma volume, and some are removed even if they hit a turn of the magnetic field coil.

2.3. Twist Filter System

In recent work [27], a new open filter design was presented that is essentially a "twisted" S-shaped open solenoid, or "Twist Filter." Twisting refers to a rotation of one bend of the S-filter with respect the other bend. The Twist Filter system consists of several matching components: the cathodic arc plasma source and its power supply, the Twist Filter, the macroparticle "firewall," a plasma expansion zone, and a substrate positioning mechanism.

The design is based on several paradigms:

1. The filter must be short to minimize plasma losses [24].
2. The source (and thus the cathode) must be small to match the size of the filter.
3. If the cathode is small, it needs to be replenished via a cathode feed mechanism.
4. If the source and filter are small, heating constraints can be alleviated by operating in a pulsed mode.

5. Pulsed mode operation can be performed at relatively high arc currents, leading to reasonable average plasma production, with enhanced plasma properties such as enhanced average ion charge state.
6. In pulsed mode, the high arc current can be used to generate a strong magnetic filter field that is in perfect synchronization with the plasma production.
7. Pulsed operation requires simple and very reliable arc initiation such as the "triggerless" mechanism [28].
8. The system needs to have a macroparticle "firewall", i.e. a wall separating the source and filter area from the clean substrate area.
9. The system needs to have a designated region in which macroparticles are collected for removal on a regular maintenance schedule.
10. All parts of the system (source, filter, power supply, firewall, etc) need to be considered as a unit with well-matched parameters.

The Twist Filter system is a consequent realization of the design paradigms. The likelihood of macroparticle transport is greatly reduced while the plasma transport is impressive compared to other filter designs: the filtered ion current may reach up to 4% of the arc current. The coil turns have a flat cross-section promoting macroparticle reflection towards the outside of the filter volume, i.e., the coil represents a baffle structure. The Twist Filter has a strong magnetic field (0.25 mT/A) providing excellent plasma confinement. At high currents (> 1 kA), not only electrons but also carbon ions are magnetized. The fringe field at the entrance is well suited for source-filter coupling.

Figure 2 Photograph of the R&D prototype Twist Filter system, showing a Twist Filter (two 90° bends twisted by 90°, ID 30 mm, OD 48 mm) with its holding structure, the cathodic arc plasma source with advancing cathode, and the arc power supply. For scale: the arc supply can be mounted in a standard 19" (48.3 cm) rack.

3. Synthesis and Properties of ta-C Synthesized by Filtered Cathodic Plasma Deposition

It is known that the properties of a-C films can be easily tuned by carefully choosing the ion energy during deposition. Properties such as sp^3 content, hardness, Young's modulus, density, and stress have a maximum when ions have a kinetic energy of about 100 eV [29-32]. Here, ta-C depositions have been performed using the Twist Filter system as well as its predecessor system, a "minigun" cathodic arc source [33] that was coupled to an open-architecture S-filter [18].

3.1. Corrosion resistance

Corrosion resistance is the key property of ta-C films for 100 Gbit/in^2. When we approach film thicknesses that are better described by number of atoms than nanometers, some properties such as hardness become questionable, or ill-defined, or at least

very difficult to measure. However, the requirement of corrosion protection of the magnetic layers remains.

Early assessment of the closed versus open filter architectures were performed jointly by Lawrence Berkeley Laboratory and the Computer Mechanics Laboratory of the University of California at Berkeley. Results have been reported at meetings of the National Storage Industry Coalition (NSIC) and in the literature [34, 35]. This work can be summarized as follows. Films of various thickness down to 2 nm were deposited on low-resistivity Si <100> wafers that were coated with a magnetic layer of 100 nm permalloy (80% Ni / 20% Fe). The deposition methods included pulsed cathodic arcs with a closed-architecture S-duct filter and an open-architecture S-filter. 7-nm RF-sputter-deposited a-C:H films (30 at.% H_2) were used as reference. When the open S-filter was used, some but not all of the 2 nm ta-C films passed the pinhole count corrosion test (samples immersed for 24 h in a solution of 0.5 mol NaCl, 0.5 mol $(NH_4)H_2PO_4$, 1 g Liquinox, and 1 liter of deionized H_2O). An optical microscope at 100x magnification with image processing was used for the pinhole count. Deposition was not done in a cleanroom environment, and therefore these preliminary results were not conclusive about the thickness (or better, thinness) limits of ta-C corrosion protection capability.

Two major steps were done recently: one was the development and implementation of the Twist Filter system, and the other was its installation in a class 100 cleanroom. The Twist Filter was expected to give even better results because macroparticle removal should be close to complete. First corrosion tests indicate that this is indeed the case (Fig.3). For this experiment, 3 nm and 4 nm ta-C coatings were deposited on sets of read/write heads. Corrosion was tested using the standard "Battelle test" (48 h exposure at 30°C, 65% relative humidity, in the presence of 8 ppb hydrogen sulfide, 8 ppb chlorine gas, and 80 ppb nitrous oxide). The heads coated with the Twist Filter survived the corrosion test, indicating that the 3 nm overcoat is continuous.

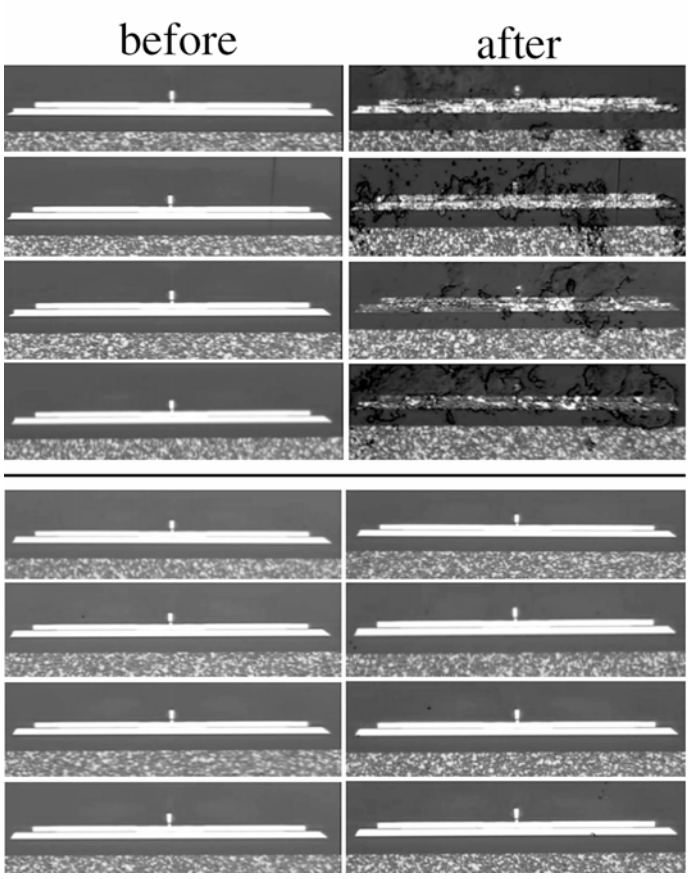

Figure 3 Battelle corrosion test of 3-nm ta-C coated read/write heads. Optical micrographs before (left column) and after (right column) the test. The top four rows show heads that were coated using a DC carbon vacuum arc with a closed filter; the bottom four rows show heads coated with the Twist Filter system.

3.2 Surface analysis by Atomic Force Microscopy (AFM)

Films of ta-C were deposited on <100> Si wafers using a closed-architecture filter and the Twist Filter system. Ultrathin films usually have too few particles to be quantified by AFM, therefore, relatively thick films of 40 nm were used for this study. Figure 4 shows clearly that macroparticle reflection is practically eliminated in the Twist Filter system.

3.3 Hardness and Elastic Modulus

Hardness and elastic modulus were measured using a force/displacement transducer coupled to an AFM. A corner of a cubic diamond tip with a nominal radius of 50 nm was used to indent the sample at loads of 1 μN to 10 mN. The measurement took indenter deformation [36] into account (otherwise hardness and elastic modulus are overestimated).

The results are shown in Fig. 5. Hardness and elastic modulus decrease with decreasing film thickness, in agreement with independent measurements [32]. Besides the substrate effect, the subplantation growth model suggests that the surface layer of ta-C films has much less sp^3 bonds than its bulk [37]. The ratio of the surface layer thickness to the total film thickness increases for thinner films and hence results in lower hardness values. While the modulus data shown in Figure 5 can be fitted linearly, there is an interesting feature in the hardness curve. If the data points for the thicker films (> 40 nm) are linearly fitted, one obtains the same slope as for the modulus data fit: this is the well-known proportionality *H~E* [38]. The hardness for thinner films is less and does not follow the *H~E* rule.

Although the hardness of the film in the sub-10-nm regime is relatively low, one must be careful in equating it to the film wear resistance. The wear rate of ultrathin films may be independent of hardness. Fracture toughness may be more relevant especially in the case of sliding wear [1]. Scratch tests of ultrathin, ion-beam-deposited carbon nitride films indicate film failure by brittle fracture followed by abrasive wear [5].

Nanoidentation of ultrathin films is pushing the limits because the effect of the substrate increases with decreasing thickness, and the indenter tip radius is much larger than the indentation depth. Other methods such as dispersion measurements of ultrasonic waves are being perfected for ultrathin films [39]. Young's modulus measurements performed this way often give greater values than nanoindentation, and the proportionality *H~E* must be questioned. This is an area of research and an example of the challenges of ultrathin film research.

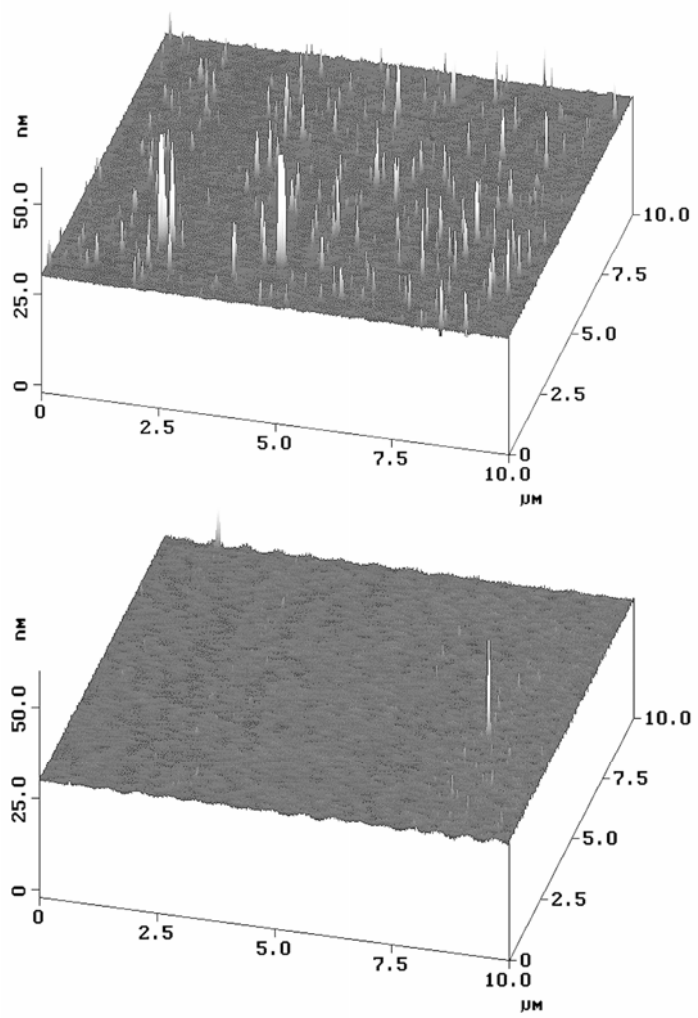

Figure 4. AFM pictures of the surfaces coated with ta-C (10 μm x 10 μm, height scale 25 nm). Top: Film of 41 nm deposited with DC arc and closed filter in a cleanroom environment. Bottom: Film of 40 nm deposited with a prototype Twist Filter system in a laboratory (non-cleanroom) environment.

3. 4. Deposition Rate, Reproducibility, and Uniformity

The Twist Filter System operates with typically 1500 A arc current in 1 ms pulses, giving about 30 A of filtered carbon ion current during the pulse. As usual for pulsed systems, one has to distinguish between instantaneous and average values. The duty cycle is defined by $\delta = t_{on} / (t_{on} + t_{off})$. For instance, when using 3 pulses per second, the duty cycle is 3 x 10^{-3} or 0.3%, thus the averaged ion current is 90 mA. This is comparable to the output of conventional filtered cathodic arcs that operate in continuous mode. This ion flux corresponds to about 150 pulses that are needed to deposit a 3 nm film on a 6 inch wafer. With 3 pulses per second, one would need a

deposition time of about 50 s. Faster deposition is possible and has been demonstrated, but cooling of the filter becomes an issue.

Film thickness and graphite cathode rod usage are very reproducible, and be predicted simply by counting arc pulses, provided that rod advancement is periodically performed and controlled (e.g. every 1000 pulses). The relationships scale linearly with the arc pulse number with a correlation of R=0.9998.

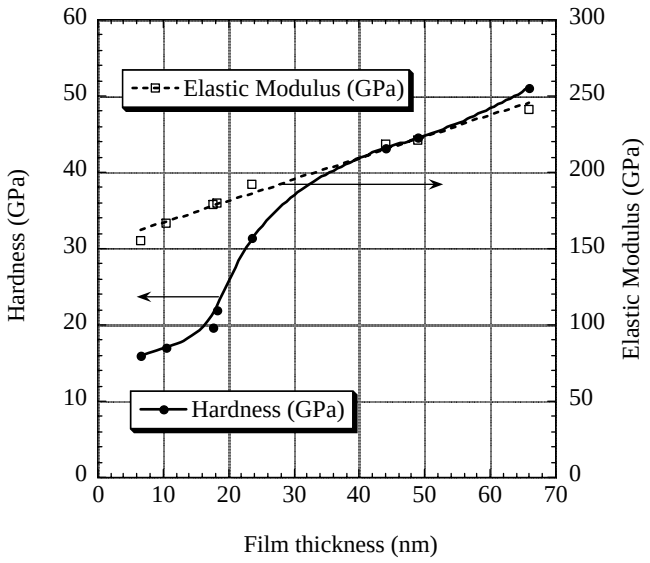

Figure 5 Hardness and elastic modulus of filtered-cathodic-arc ta-C films as a function of film thickness (data from [34]).

The output of the Twist Filter System is highly focussed at the filter exit and plasma expands on its way to the substrate. At the substrate location it has Gaussian profile. Film thickness was measured by ellipsometry and calibrated by AFM step height measurements. For a filter-substrate distance of 15 cm, the film thickness is peaked at the substrate center with $\sigma \approx 10\%$ of the film average. Improvements can be accomplished by increasing the source-substrate distance and by implementing magnetic multipole structures (as in "bucket" ion sources).

4. Conclusions

The development of improved macroparticle filters enables cathodic arc plasma deposition to produce thin and ultrathin films of excellent quality. Open-architecture filters address the reflection issue that have plagued traditional filter ducts (closed architecture). The most complete removal of carbon macroparticles is achieved by a "twisted" S-filter of open architecture ("Twist Filter"). Ultrathin ta-C films (< 5 nm) can meet the requirements of the magnetic storage industry. For storage densities approaching 100 Gbit/in^2, current research focuses on the synthesis and analysis of films thinner than 3 nm. Closed-architecture filtered-arc equipment is commercially available, and open-architecture equipment is entering the commercialization phase.

Acknowledgements

Robert A. MacGill deserves the credit for designing and manufacturing most of the Twist Filter, and Tom Miller figured out the details of Twist Filter mounting. The authors gratefully acknowledge the support by Thomas A. McVeigh, Othon R. Monteiro, and Ian G. Brown of LBNL, Berkeley; Marcela M.M. Bilek, University of Cambridge; David Baldwin and Frank Cumbo of CVC, Inc.; Niranjan Gopinathan, Ashok Kulkarni, Carolyn Robinson, and Bharti Patel of Read-Rite Inc, C. Singh Bhatia of IBM, and David B. Bogy and Walton Fong of UC Berkeley. This work was supported through the CRADA BG98-084(01) of the ER-LTR Program of the U.S. Department of Energy, under Contract No. DE-AC03-76SF00098, and by CVC-Veeco, the CRADA Industrial Partner.

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