

# Growth responses of ultrathin $\text{CN}_x$ overcoats to process parameters

D.J. Li <sup>a,b,\*</sup>, Yip-Wah Chung <sup>c</sup>

<sup>a</sup> College of Physics and Electronic Information Science, Tianjin Normal University, Tianjin 300074, P.R. China

<sup>b</sup> Department of Materials Science and Engineering, Northwestern University, Evanston, IL 60208, USA

<sup>c</sup> Department of Materials Science and Engineering, Northwestern University, Evanston, IL 60208, USA

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## Abstract

Ultrathin  $\text{CN}_x$  overcoats were grown using pulsed dc magnetron sputtering. Substrates were mounted on a holder that allowed  $45^\circ$  tilt angle and rotation. Effects of process parameters on film growth were reviewed. AFM scans over large sampling areas show that thin  $\text{CN}_x$  films obtained at  $-100$  V substrate bias with  $45^\circ$  substrate tilt and 20–25 rpm rotation have r.m.s. roughness about 0.2–0.3 nm when sampled over  $20 \times 20 \mu\text{m}^2$  areas, increasing to  $\sim 0.45$  nm when sampled over  $\sim 0.05 \times 3 \text{ cm}^2$  using X-ray reflectivity measurements. These 1–2 nm thick ultrasmooth coatings reduced corrosion damage compared with coatings of the same thickness grown without substrate tilt and rotation. This improved performance is likely a result of more efficient and uniform momentum transfer parallel to the surface during deposition in this configuration. In addition, detailed X-ray reflectivity measurements showed that the mass density of these  $\text{CN}_x$  films is  $\sim 2.0 \text{ g/cm}^3$ , independent of film thickness from  $\sim 1$  to 10 nm, consistent with ion beam analysis.

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## 1. Introduction

In the field of magnetic recording technology, strong attention is now being focused on increasing the areal storage density in computer hard disk drives. In order to achieve this goal, the magnetic spacing (distance between the pole piece of the read/write head and the top of the magnetic media layer) must be reduced. In recent years, hard disk capacity has been being increasing steadily with this spacing reduction (Fig. 1).

The current goal of the magnetic storage industry is to produce hard disk drives with  $1 \text{ Tb/in.}^2$  ( $155 \text{ Gb/cm}^2$ ) areal storage density, requiring reduction of the magnetic spacing to 5.0 nm. As shown in Fig. 1, this 5.0 nm spacing includes protective overcoat for the head, the air bearing, lubricant and protective overcoat for the disk. Tradeoffs between these

different components suggest that the protective overcoat for the head and disk has to be decreased from 4 to 5 nm in current drives to 1.0 nm thick each. At this small overcoat thickness, one has to worry about the susceptibility of the magnetic media layer to environmental attack. In this case, producing an atomically smooth, defect-free and dense protective overcoat over a large scale becomes crucial.

Nitrogenated carbon ( $\text{CN}_x$ ) is an excellent overcoat material due to its compatibility with existing lubricants as well as desirable tribological and corrosion performance [1–3]. In our previous studies [4–6], we used pulsed dc magnetron sputtering to synthesize  $\text{CN}_x$  overcoats down to 1.0 nm thickness with acceptable corrosion performance and smoothness by controlling process parameters. This article reviews our progress made by a proper combination of substrate tilt and rotation and optimum ion bombardment and discusses the significance of these process parameters and substrate mounting geometry in affecting surface roughness and corrosion performance of ultrathin  $\text{CN}_x$  overcoats. In addition, we also focus on detailed studies on several metrology issues

\* Corresponding author. College of Physics and Electronic Information Science, Tianjin Normal University, Tianjin 300074, P.R. China.

E-mail address: [dejunli@mail.tjnu.edu.cn](mailto:dejunli@mail.tjnu.edu.cn) (D.J. Li).

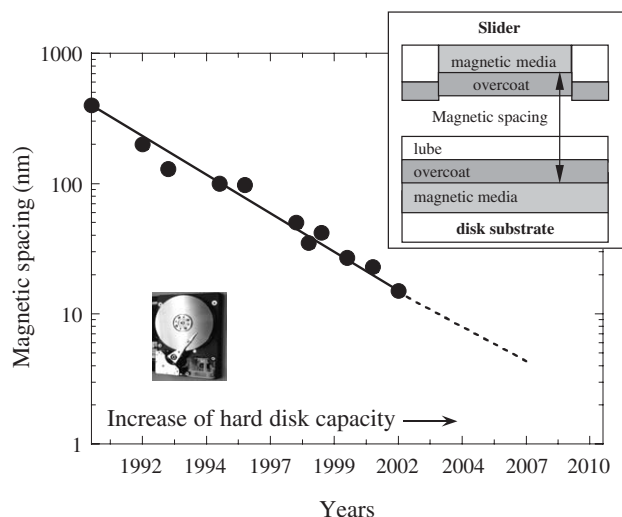


Fig. 1. Variation of magnetic spacing in hard disk drives with time.

associated with these 1–10 nm thick films using X-ray reflectivity (XRR).

## 2. Experimental detail

CN<sub>x</sub> overcoats were sputter-deposited to different thicknesses by varying the sputtering time in a single-cathode deposition system with a base pressure of  $4 \times 10^{-8}$  Torr. Graphite (purity 99.995%) was used as the target material, and an Ar–5% N<sub>2</sub> mixture was chosen as the process gas. During deposition, the total pressure was kept at 3 mTorr, and the target power was kept at 200 W. Silicon (001) wafers and supersmooth hard disks, the latter for corrosion tests, were used as substrates that can be positioned with no tilt or 45° and can be rotated at speeds up to 25 rpm. Substrates were cleaned ultrasonically first in acetone and methanol for 10 min, then transferred into the chamber and reverse-sputter etched in an argon plasma at 40 mTorr with –400 V bias for 3 min to remove surface contaminants. In order to enhance ion bombardment of the growing film, a negative bias was applied to the substrate during growth. This bias was varied between 25 and 250 V. The polarity of the target and the substrate was switched at a fixed frequency of 20 and 2 kHz, respectively, to minimize charging during deposition. In this case, the voltage applied in the positive cycle is set at 10% of that applied in the negative cycle.

The film growth rate was calibrated by measuring the thickness of coatings using a profilometer. The root-mean-square roughness was obtained over sampling areas ranging from  $1 \times 1$  to  $20 \times 20 \mu\text{m}^2$  by AFM. Corrosion tests were carried out to evaluate the defect density of these 1–10 nm ultrathin films according to our previous work [4]. XRR experiments were performed on an 18 kW Rigaku rotating copper anode diffractometer. The specularly reflected intensity was measured as function of incidence angle  $\theta$

(measured from the surface) and plotted as a function of  $k_{z,0} = 2\pi \sin \theta / \lambda$  ( $\lambda = 1.542 \text{ \AA}$ ). Since the CN<sub>x</sub> film has interfaces with air and the Si substrate, interference between X-rays reflected at these two interfaces leads to an oscillatory pattern, from which one deduces the film thickness [7–9]. The electron density, and hence mass density, can be calculated from the measured critical angle above which the reflected X-ray intensity drops abruptly. The rate at which the reflected X-ray intensity decreases with increasing  $\theta$  or  $k_{z,0}$  depends sensitively on surface roughness [9]. At these low angles of incidence, the X-ray beam spreads over distances  $\sim \text{cm}$  across the film. Therefore, this method gives surface roughness averaged on macroscopic dimensions.

## 3. Results and discussion

We deposited CN<sub>x</sub> coatings on Si with four combinations of substrate tilt and rotation at –100 V in order to compare the influence of substrate tilt and rotation on surface roughness. Fig. 2 shows its values as a function of scan size for these four combinations. This figure demonstrates that a combination of 45° substrate tilt and rotation leads to significant improvement in surface roughness. For example, at a scan size of  $10 \times 10 \mu\text{m}^2$ , the surface roughness is 0.16 nm with 45° substrate tilt and 20 rpm substrate rotation, increasing to 0.56 nm with no substrate tilt and rotation.

Fig. 3 shows the surface roughness of CN<sub>x</sub> films as a function of substrate rotation speeds at five scan sizes. The substrate was tilted at 45°. Surface roughness values from larger scan sizes clearly show that rotation speed at 20 rpm or greater give marked improvement. At a growth rate of about 2 nm/min, deposition of one atomic layer (about 0.25 nm) takes 7.5 s, during which 2.5 rotations occur at 20-rpm substrate rotation speed. The randomizing action of energetic neutrals arriving at different azimuthal positions is probably the cause of this roughness improvement. Similarly, the effect of

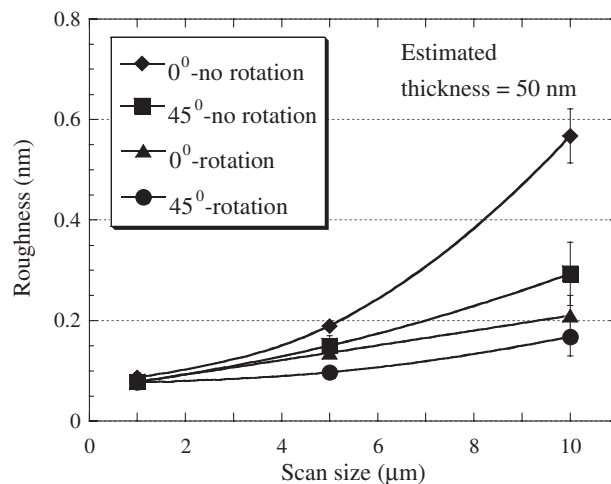


Fig. 2. Influence of substrate tilt and rotation on the r.m.s. roughness for CN<sub>x</sub> films deposited on Si [5].

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