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Materials Selection for Ultra-Thin Diamond-Like Carbon Film Metrology and Structural Characterization by TEM.

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Critical dimension metrology by transmission electron microscopy (TEM) plays a paramount role in ultra-thin 10-30Å diamond-like carbon (DLC) films used in hard-disk drive manufacturing, where performance is traded against reliability as the nominal thickness continues aggressive scaling downward [1]. TEM sample preparation requires that a protective coating is deposited prior to site-specific focused ion beam (FIB) cross-sectioning. This coating is also critical in providing a high contrast delineating marker of the DLC top surface for imaging contrast-based metrology. The criteria that such protective coating must be inert to the DLC, as well as free of coarse structure, narrows the materials selection significantly. Cr is one of the most common coating materials used due to its legacy from SEM coaters for high resolution applications as it forms a continuous, quasi-amorphous film. As the literature on protective layer materials selection for ultra-thin DLC film is non-existent, we disclose original research that has led to a breakthrough in the industry and expanded the current understanding.

DLC film was deposited on NiFe substrate using the conventional filtered cathodic arc (FCA) process [2]. TEM imaging and EELS were performed using a Schottky field emission gun TEM operating at 200 kV, and a post-column spectrometer. Samples, having the DLC film as outer surface layer, were initially coated prior to FIB with either Cr or Cr_2O_3 by ion beam sputtering. C and Cr quantified elemental profiles across a Cr-coated DLC film are shown in Fig.1(a). C is clearly skewed towards the Cr layer, indicating interaction. C-K edge extracted from the interfacial region reveals that carbon exists as (Cr-) carbide, with a characteristically intense π^* event [3] (Fig.1b). On the other hand, profile skewness is not present when the same DLC is coated with Cr_2O_3 (Fig.1a), and the C-K edge does not possess the intense π^* indicative of carbide (Fig.1b), retaining a more DLC-like shape instead. Thus the interaction between C and the protective coating material is central to the interpretation of spectroscopic data.

TEM-based film thickness metrology of the DLC layer is also affected by its interaction with Cr, as shown in Fig.2(a). Using identical film thickness metrology definitions, the Cr_2O_3-coated DLC film is ~ 10Å thicker than the Cr-coated counterpart. The excess film thickness comes from the fact that C formed carbide with Cr. To evaluate the degree of interaction between DLC and Cr_2O_3 (and Cr), evaporated Au film was deposited on the DLC. Evaporated Au is widely known to be the most gentle and thus the least interacting film deposition technique available. Results show that the Au-coated DLC film thickness is comparable to that of the Cr_2O_3-coated film, confirming that Cr_2O_3 deposited by the ion beam sputter deposition technique is likewise gentle and non-interacting to the DLC film. However, Au formed large grains (Fig.2b) which disturbed the continuity of its interface with the DLC. Fig.2(c) shows a cross-section of the same DLC sample without any protective coating applied, which was achieved by a very elaborate, manual sample preparation methodology on a Ta coupon substrate that cannot be reproduced for industrial-scale metrology. The uncoated DLC thickness is equivalent to that of evaporated Au-coated and Cr_2O_3-coated DLC samples, thus further validating the non-interacting nature

of Cr_2O_3 protective layer for ultra-thin DLC films. Other coating materials tested in this study, such as Ti, Ir, W, and Ta, all show some degree of interaction with C varying between Cr and Cr_2O_3, and therefore are not as ideal as Cr_2O_3 for use as protective marker coating materials for DLC.

In conclusion, ultra-thin DLC characterization by TEM is strongly dependent on the choice of protective material applied prior to cross-sectioning. In particular, the interplay between oxidation and carbidization leads to metrology inaccuracy and misinterpretation of structural information, as well as potential metrology control excursions given that Cr film stability is dependent on vacuum conditions and target material cleanliness. Cr_2O_3 makes a more reliable, stable and robust protective coating system in that regard, allowing for TEM/EELS characterization of the true, artifact-free DLC film at Å scale.

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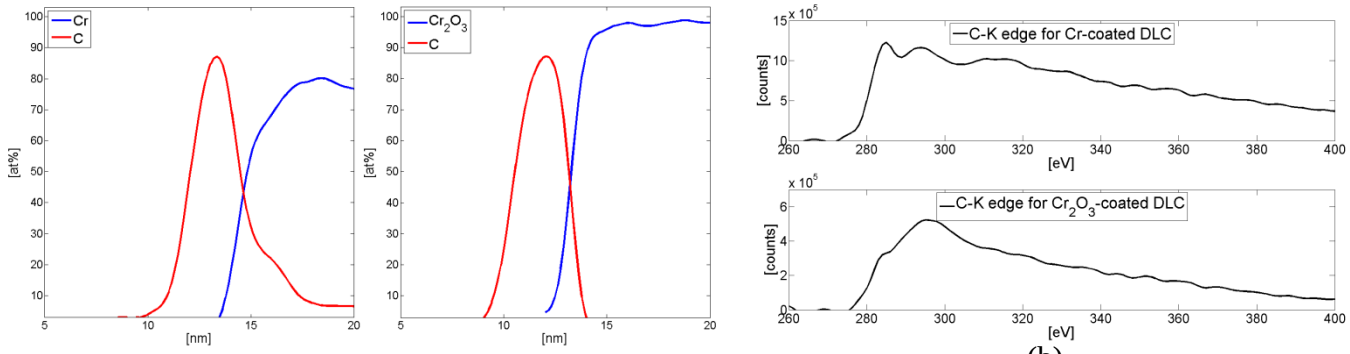
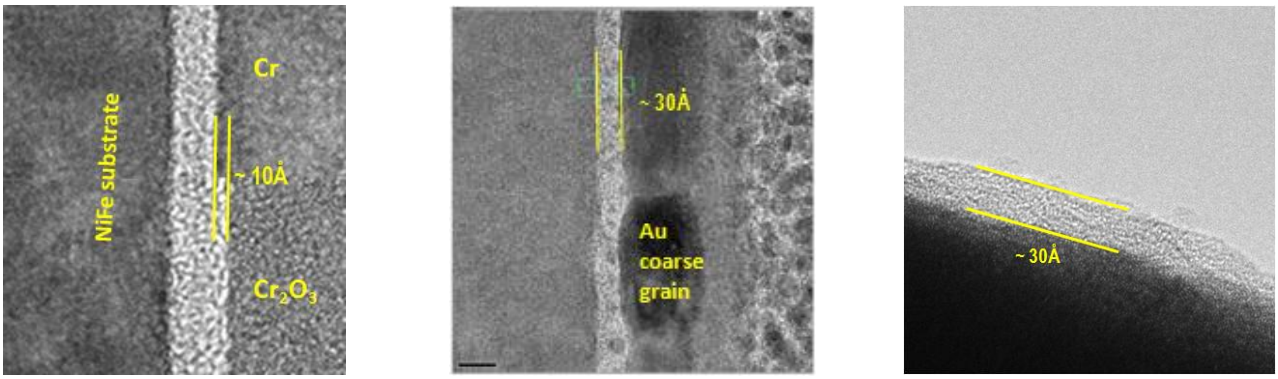


(a) (b)

Figure 1. (a) EELS profiles for C, Cr, and Cr_2O_3 across the DLC. Cr and C form a carbide, as shown by the pronounced C-K edge π* event (b) compared to the more DLC-like character retained in the case of the Cr_2O_3-coated sample. C-K edges were taken from the interface between DLC and protective layers.



(a) (b) (c)

Figure 2. (a) Comparison between Cr- and Cr_2O_3-coated DLC showing that the Cr-coated DLC is ~ 10Å thinner due to C/Cr interaction; not seen for the Cr_2O_3-coated DLC. Evaporated Au-coated (b) and uncoated (c) DLC films have the same thickness as compared to Cr_2O_3-coated DLC, confirming that Cr_2O_3 protective layer does not interact or change ultra-thin DLC films for TEM characterization.

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