Durability Evaluation of Ultra-Thin Diamond-Like Carbon Films

Hard thin film coatings are widely used for many applications as mechanical, tribological, and corrosion protection layers. They can be as thin as 1 nm and as thick as 1 m. When the coating thickness is comparable to the smallest domain size of most materials, it is a very challenging task to evaluate thin film mechanical properties. Micro or nano hardness is somewhat misleading for such thin films, since the stress distribution may be deeper than the film thickness. This paper describes a newly developed testing procedure and counter surface using CETR Micro-Tribometer, designed for thin film evaluation.

The schematic for the Micro Tribometer model UMT is shown in Figure 1. It can provide rotational, translational, and reciprocating motions with speeds ranging from 0.1 m/s up to 10 m/s.

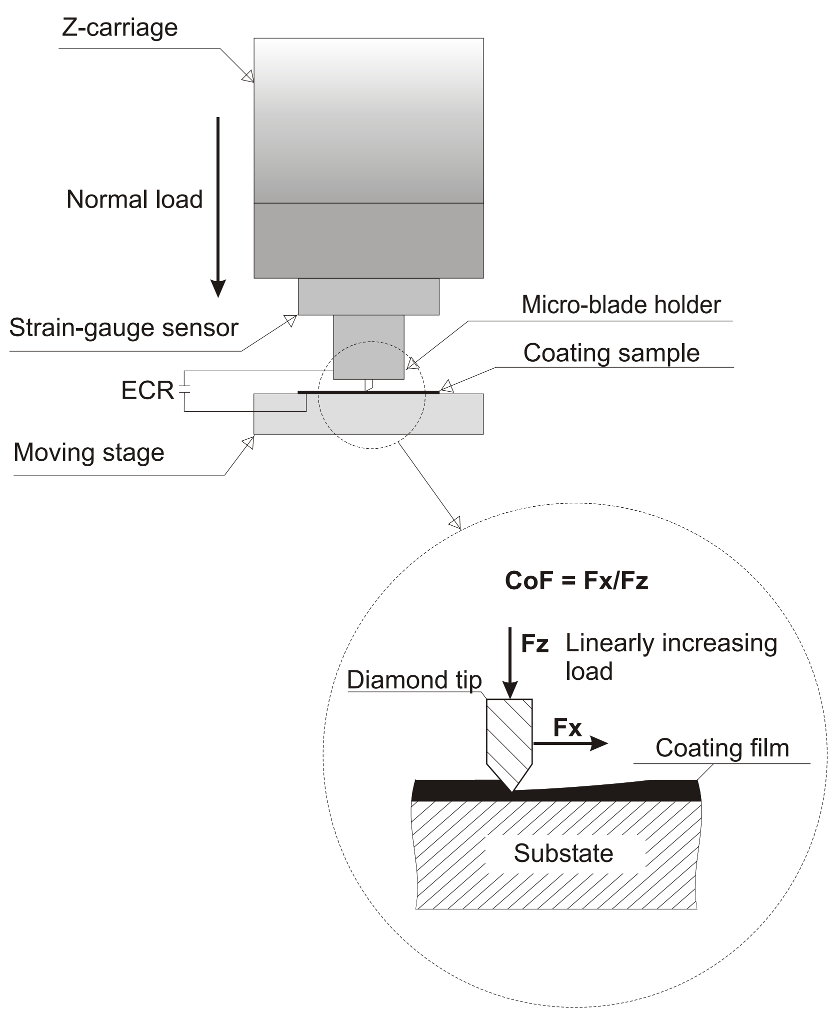

Figure 1. Schematic of scratch test for thin film coatings

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The load is applied using a close-loop servo mechanism for stability and accuracy, and can be kept constant or linearly increasing, ranging from as low as 0.5 mN to as high as 200 N. Temperature and environment/pressure can also be changed at the interface. Friction force (Fx), normal load (Fz), electric contact resistance (ECR) and acoustic emission (AE) are measured and recorded at a total sampling rate of 20 kHz. Wear depth, electric capacitance and digital camera are also readily available.

The samples used in this study were 95-mm magnetic disks consisting of a thin layer of diamond-like carbon (DLC) on a composite magnetic layer sputtered on a NiP substrate. The DLC carbon thickness ranged from 3 nm to 10 nm. Some disks were dip-coating lubed with a liquid PFPE of about 1.5 nm thick. The counter surface was a patented micro-blade with a 0.4 mm radius. The micro-blade was moving against the thin film at a constant speed of 10 mm/s under a linearly increasing load from 2 mN up to 2 N.

As the micro-blade sliced into the film coating, progressive material removal occurred, as demonstrated in Figure 2 for three tracks, post-test measured using the optical surface reflection for a carbon film of 3.5 nm. The DLC film was scratched through at the beginning of the dark tracks. As Fig. 2 shows, the scratch tests were very repeatable. For comparison, Figure 3 shows three repetitive scratch tracks for the same DLC coating, but with a liquid PFPE lubricant. One can see, the lubricant increased the carbon film durability remarkably; again, the scratch tracks were very repeatable.

The in-situ data for friction force Fx in red, normal load Fz in blue, coefficient of friction COF in light blue and electrical contact resistance ECR in green as a function of test time is shown in Fig. 4 for the 3.5-nm DLC film and in Fig.5 for a 6-nm DLC film.

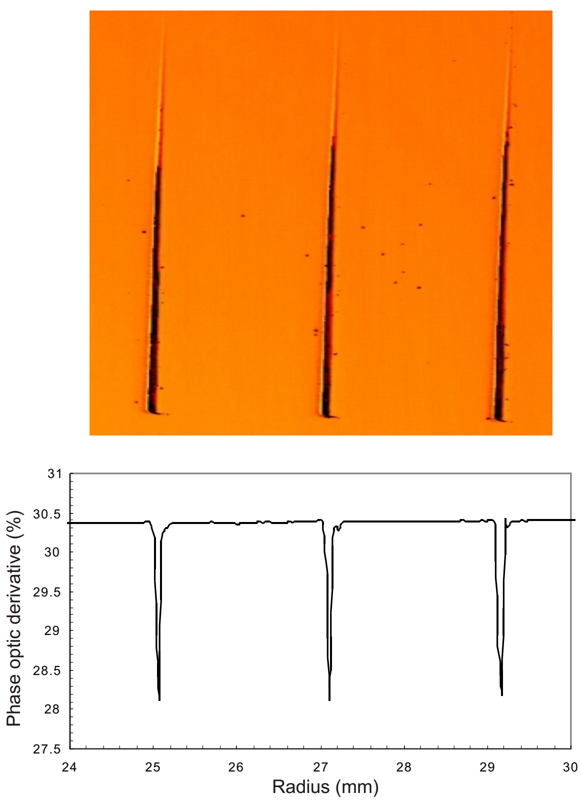

Figure 2. Apparent optical scratch depth on 3.5-nm carbon (unlubricated)

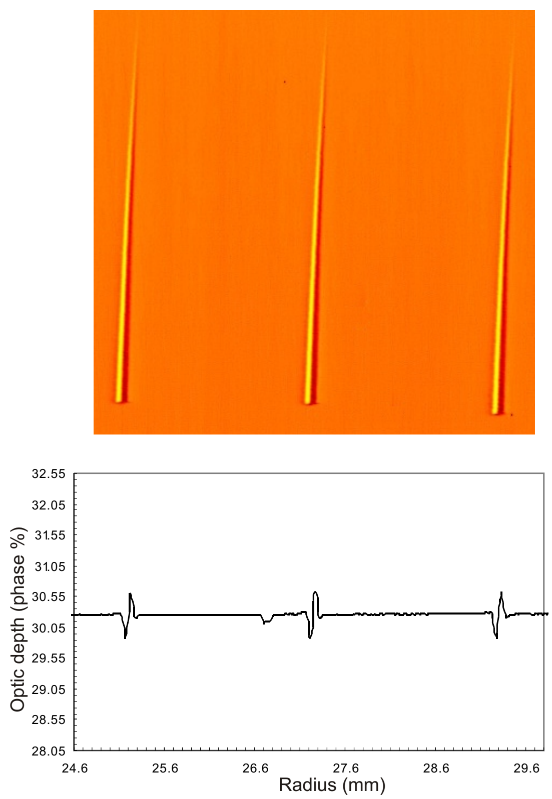

Figure 3. Apparent optical scratch depth on 3.5-nm carbon (lubricated)

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At the critical load, friction force shifted to a higher value and started to fluctuate violently. At exactly the same time, ECR dropped to practically zero, because the micro-blade made contact with a conductive magnetic layer after cutting through the DLC coating.

The critical load at which a carbon film was cut through by the micro-blade increased with increasing carbon thickness. Please note that Fz scale is from 0 to 250 grams in Figure 4, but from 0 to 120 grams in Figure 5. Also note that the ECR scale is from 0 to 2.5 kOhm in Figure 4, but from 0 to 120 kOhm in Figure 5 (carbon film was thicker). The critical load at which the 6-nm film was scratched through was 90 cN, whereas the critical load was 55 cN for the 3.5-nm DLC.

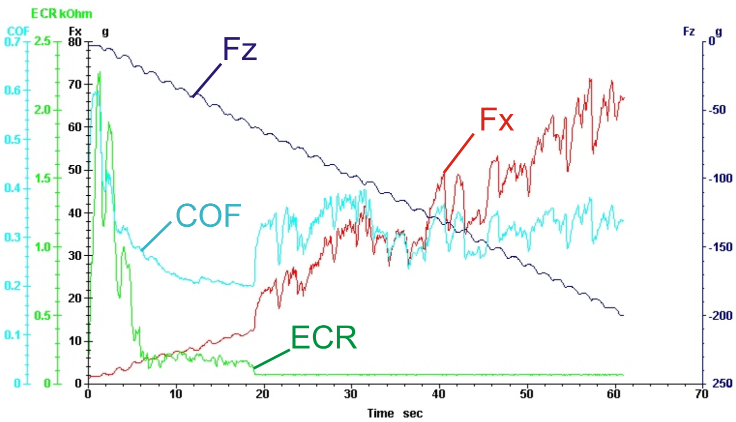

Figure 4. Scratch test for 3.5-nm carbon
The carbon film was scratched through at a critical load of 55 cN

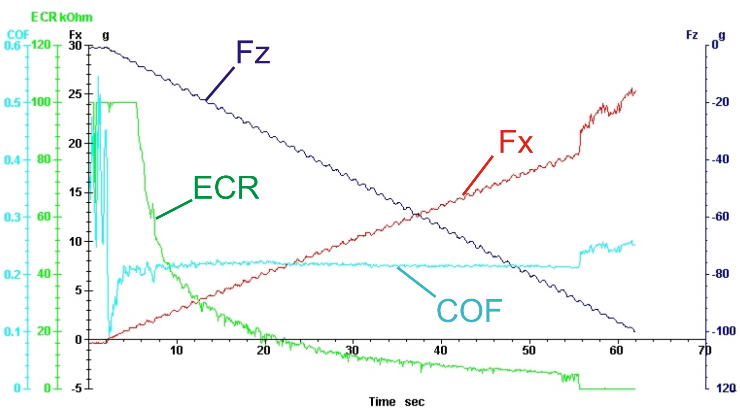

Figure 5. Scratch test for 6-nm carbon
The carbon film was scratched through at a critical load of 90 cN.

In conclusion, the newly developed CETR Micro-Tribometer and the above testing procedure is a very powerful and fast technique in evaluating durability of thin coatings with or without a lubricant for magnetic media and heads down to 1-nm thickness. When the coating was progressively scratched through under the patented micro-blade, friction force began fluctuating violently and the electric contact resistance dropped to practically zero. The fact of breaking through of DLC carbons at the critical loads was easily confirmed with optical reflection images and mechanical profiler traces. It is worthwhile to mention that a diamond stylus and stainless steel balls were found much less effective in the DLC durability evaluation. It is our understanding that ramp loading, precision motion and right choice of micro-blade geometry are the most important factors for repeatable and reproducible evaluation of thin film durability.