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Advanced Methods of Coating Adhesion Testing

ABSTRACT: Adhesion of coatings from 1 nm to 0.1 mm was successfully evaluated with multi-sensing technology, based on simultaneous high-resolution force, electrical and acoustic measurements. Sharp changes in the signals were indicative of the thresholds of adhesion and delamination for various thin and thick coatings during scratching and indenting with several test tools, both sharp (diamond tips) and dull (balls).

KEYWORDS: Adhesion, coatings, thin films, testing.

Introduction

There are numerous techniques known for adhesion and delamination testing, some of the most common being a tape test, stud-pull test, scratch test and an indentation test.¹ In the tape test, a tape is pulled off the surface containing a scratch, which provides the failure initiation. In the stud pull test, a stud held with thermosetting epoxy is pulled off the film surface. The indentation test, wherein a ball is pressed into the surface, is used for hard coatings, and the failure pattern indicates acceptable behavior. In the scratch test, where an indenter moves in both vertical (loading) and horizontal (sliding) directions, and an acoustic emission sensor allows for detection of the initiation of fracture, while the scratch pattern indicates the type of failure.

The Universal Nano+Micro Materials Tester (UNMT) has been developed to perform all the variety of the common adhesion tests.² During any of them, it can simultaneously measure contact or surface electrical resistance, displacement or deformation or depth of penetration, contact acoustic emission, temperature, forces in all three directions and digital video of the contact area. This report covers evaluation of the adhesion and delamination properties of coatings by the scratch test.

Instrumentation

The fully computerized tester UNMT provides a combination of precision linear and rotational, including reciprocating, motions to the specimens, with programmable speeds ranging from 0.1 $\mu\text{m/s}$ to 10 m/s. A load is applied via a closed-loop servo-mechanism and is programmed to be kept constant or linearly increasing, ranging from 10 nN to 1 kN. The environmental temperature and pressure can also be controlled. Friction force, normal load,

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electrical contact resistance (ECR), and contact acoustic emission (AE) are all measured at a total sampling rate of 20 kHz, displayed in real time and recorded for further analysis.

Specimens in a wide variety of shapes and dimensions (up to 200 mm) can be accommodated. For scratch-adhesion tests various tool geometries and materials can be used, metal or ceramic ball or needle, diamond stylus or patented micro-blade.

Experimental

Two types of coatings were tested in this work, 50-micron “thick” soft elastomer coatings used on ink-jet cartridges and few-nanometer “thin” hard diamond-like coatings used on hard magnetic disks. These two types have been chosen as being very different in both hardness and thickness, thus covering a wide spectrum of practical applications.

The effects of scratching tools were studied on three types of tools, 1.6-mm balls from stainless steel and tungsten carbide, 10-micron sharp diamond stylus and a novel tungsten carbide micro-blade, which is as sharp as the stylus (10 micron), but very wide (0.8 mm).³

To achieve effective results, the multi-sensor technology was utilized.

Results on Thick Soft Coatings

Neither steel nor tungsten carbide balls produced useful delamination data. The sharp diamond stylus scratched the coating, but did not delaminate it. The micro-blade produced repeatable both scratch-resistance and delamination/adhesion results.

The micro-blade test consisted of linearly increasing the applied load from 10 to 1,000 mN, while slowly sliding the micro-blade at 1 mm/s, with continuous multi-sensor process monitoring.

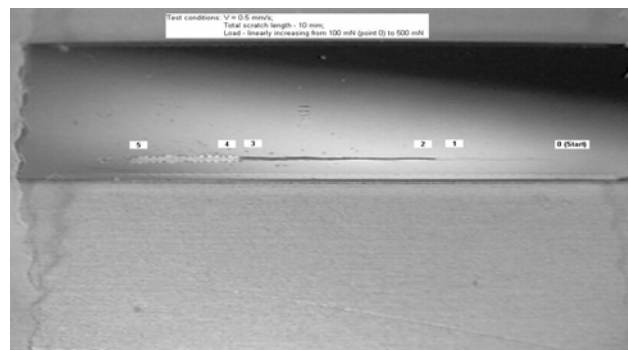


FIG. 1— *Coated Surface Tested With Micro-Blade*

The optical photo of the tested surface is shown in Figure 1 above; the corresponding friction force plot is presented in Figure 2 below. One can clearly see three zones, namely: deformation with no debris formation at very low loads (right part of Fig. 1, left part of Fig. 2), micro-scratching with production of a lot of micro-debris (middle parts of Fig-s 1 and 2), followed by delamination with chunks of debris formed at higher loads (left part of Fig. 1, right part of Fig. 2). The critical loads (or times) of scratching (on the borderline of the deformation and micro-

scratching zones) and delamination (on the borderline of the scratching and delamination zones) can be found easily from these tests, and tend to be repeatable.

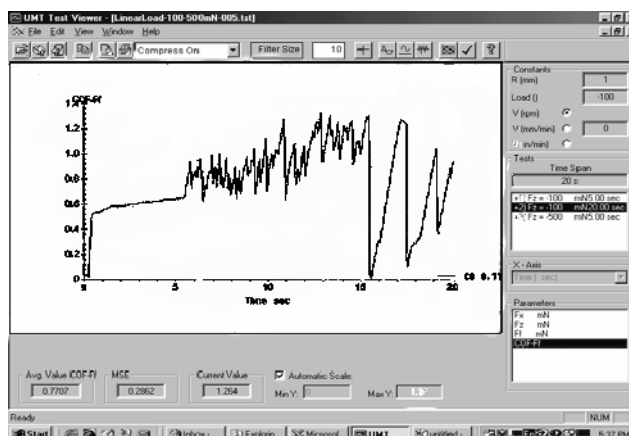


FIG. 2— *Friction Force in Test with Micro-Blade*

Though both the optical and force plots show all the three zones, the borderlines between them, defining the critical loads (times) or micro-scratching and delamination, can be determined with higher accuracy by utilizing additional electrical and acoustic measurements.

If a coating is non-conductive and a substrate is conductive, then electrical contact resistance is measured. If a coating is conductive, the electrical surface resistance is monitored. In either case, the electrical measurements typically show both critical thresholds of the onset of scratching (when electrical resistance begins to change) and breaking through the coating (when electrical resistance reaches the level of the substrate resistance). An example of a sharp threshold is given in Figure 3.

The high-frequency contact acoustic emission reflects the scratching and delamination processes of solid coatings when their structure is relatively ordered and they are relatively hard. For instance, most metal and ceramic coatings emit substantial acoustic waves during such tests, while most soft polymers do not emit measurable acoustics. The acoustic emission resolution in terms of surface defects is defined by its frequency. For example, to detect a 10-nm scratch or delamination defect in the coating at a test speed of 1 cm/s (taking into account that 1 cm = 10,000 nm and a signal has to be several times higher than the process), one needs the signal frequency of at least 5 kHz. In fact, we use the range of up to 5 MHz, which allows for detection of the very beginning of the tiniest micro-scratches and micro-delamination.

The effective use of the contact acoustic emission signal is illustrated in Figure 3 below. In this example, the critical threshold of breaking through the coating can be determined by the sharp drop in electrical resistance, and is fully supported by the sharp increases in friction and acoustic signals. It is interesting that both friction and acoustic show a substantial pre-failure rise at the same lower load, corresponding to the beginning of coating damage. Moreover, the ultra-sensitive acoustic emission started even earlier, which reflected the onset of tiny coating damage, undetectable yet by the electrical and force signals.

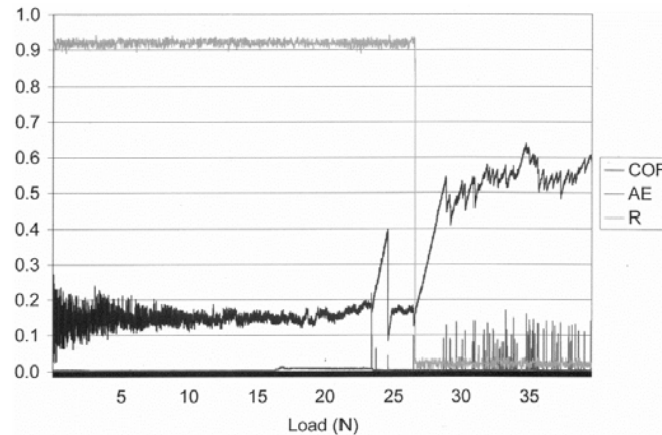


FIG. 3— *Multi-Sensor Determination of Coating Failure.*

Results on Thin DLC Coatings

DLC, or diamond-like carbon coatings, are used in various industries. Their deposition technologies and therefore properties vary from application to application⁴. For example, we have observed in numerous tests that the durability of very thin DLC coatings on hard magnetic disks and heads is the highest, while the durability of the DLC coatings on window glass and razor blades is much lower.⁴ Correspondingly, the scratch adhesion test procedure should be different.

Soft copper or gold-plated 4-mm or 6-mm balls have produced the most repeatable test data on the softest DLC coatings. Hard 1.6-mm tungsten carbide balls produced quite repeatable results on mid-range DLC coatings. Sharp diamond styluses scratched the coating, but did not delaminate it. Also, the sharp stylus data had limited repeatability due to the surface morphology differences from one scratch to another. The tungsten carbide micro-blade, averaging the scratch data over their substantial width, produced the most repeatable results for the hardest and thinnest coatings. Again, in all these tests the critical thresholds were determined by simultaneous measurements of forces, electrical and acoustic signals (see Fig. 3), though the latter ones were not informative for very thin coating films. The electrical resistance of DLC coatings was always measurable, though the harder and more diamond-like was a coating, the less conductive it was.

The thinnest coating tested was a 1.5-nm DLC film on a magnetic head wafer, the durability of which was found to be dependent on the rate of deposition. The Figure 4 below includes some of the results for thin hard DLC coatings on magnetic disks. For each DLC thickness four disks were tested, and the high data repeatability between them is obvious from this figure. Reduction in film thickness by a factor of 4, from 10 nm to 2.5 nm, decreased the critical load by a factor of 5, from 125 to 25 cN. Lubricating the disk with a thin 2-nm layer of a topical Fomblin lubricant, common for the magnetic disks, increased the critical load by a factor of 4.5 for the thin 2.5-nm coating. The micro-blade test consisted of linearly increasing the applied load from 1 cN to 100 cN, while slowly sliding the micro-blade at 1 mm/s, with continuous multi-sensor process monitoring.

Conclusion

1. The multi-sensing technology, based on simultaneous high-resolution force, electrical and acoustic measurements, allows for very accurate determination of both delamination/adhesion and scratch resistance of both thin and thick coatings. Using the test tools with higher contact area (or length), like micro-blade or ball, may allow for more repeatable results than those obtained with sharp styluses.

2. The nano+micro tester UNMT provides a useful platform for all common types of adhesion and delamination tests, with the complete utilization of all the advantages of the multi-sensing technology.

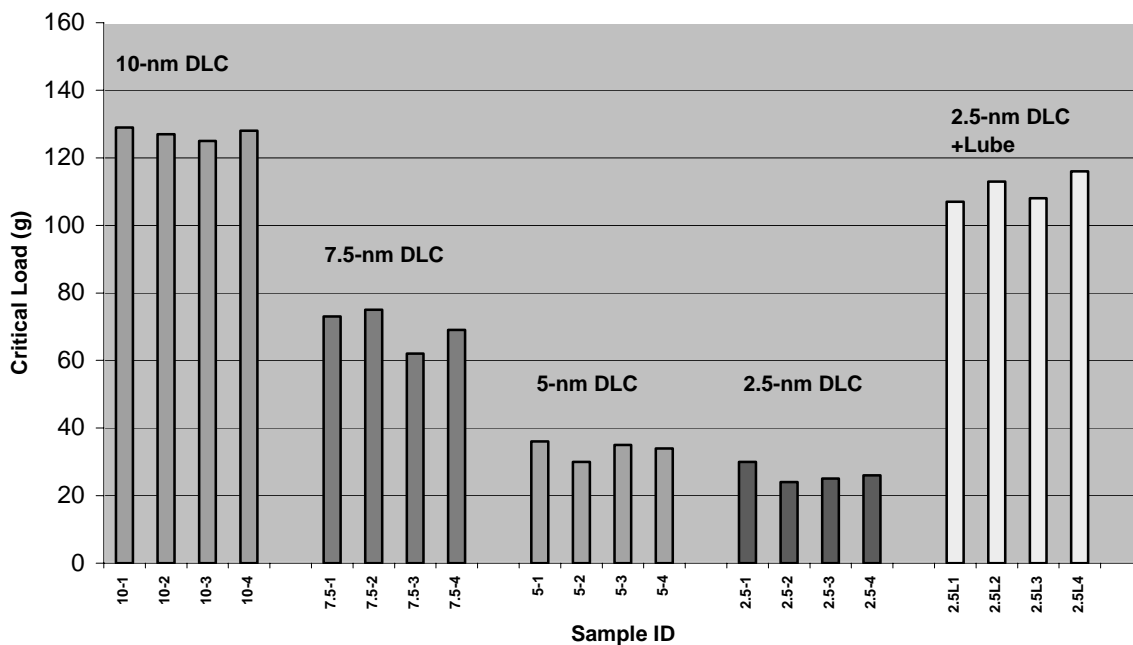


FIG. 4— *Micro-Scratch Test Data for Thin Hard Coatings.*

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