In-situ Quantitative Integrated Tribo-SPM Nano-Micro-Metrology

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ABSTRACT

A novel quantitative nano+micro-tribometer with integrated SPM and optical microscope imaging has been developed to characterize numerous physical and mechanical properties of liquid and solid thin films and coatings, with in-situ monitoring their changes during micro and nano indentation, scratching, reciprocating, rotating and other tribology tests. Both the materials properties and surface topography can be assessed periodically during the tests. The integrated multi-sensing tribo-metrology is illustrated with two examples, of nano-indentation characterization of silicon wafer based coatings and micro-scratch characterization of diamond-like carbon coatings on magnetic media on the same instrument

Keywords: nano-tribology, tribometer, nano-indentation

1 INTRODUCTION

Quantitative nanometer resolution metrology tools have become a standard in semiconductor, data storage and other hi-tech industries, where products have to be tested for thin-film properties. Though it is critical to characterize advanced thin films and coatings, today's off-line nano-scale metrology tools can capture only a limited number of parameters. There is an immediate need for process control instruments capable of in-situ nanometer scale quantitative characterization at different stages of manufacturing process. Latest advances in nano and micro tribology forced the development of integrated instrumentation. A new generation of innovative optical/SPM instruments for chemical and mechanical characterization of surfaces is being developed for tribological testing applications. A combinatorial approach in micro-scale tribology tests [1] indicated the need for in-situ characterization instruments integrated into a tribometer. A recent nano-scale tribometer combination with AFM instrument has been reported [2]. Therefore, the number of novel tribometer applications is growing fast and covering fast changing industries, such as biomedical [3].

Performance of a quantitative nano+micro-tribometer [4], integrated with AFM and high resolution optical microscope, is demonstrated in two examples, on silicon wafer coatings, where quantitative materials properties were derived at several intermediate characterization steps with nano-indentation, and on diamond-like carbon coatings on magnetic disks, where disk durability was characterized by micro-scratching with a patented variable-angle blade micro-scratcher

2 NANOINDENTATION

2.1 Instrumentation

Photo of the newly developed instrument is shown in Fig. 1. The Universal Nano+Micro Materials Tester (UNMT) consists of a fully automated precision electro-mechanical tester [4], capable of performing numerous multi-axial linear and rotary tribological tests, 3-µm resolution optical microscope, closed-loop interchangeable SPM scanner and a nano-indentation instrument Nanohead.

Figure 1: Photo of UNMT with integrated AFM and optical microscope.

The UNMT has easily interchangeable rotary and linear, including fast oscillations, lower and upper drives, that provide a speed range from 0.001 (0.1 µ/s) to 10,000 rpm (50 m/s). It can apply a servo-controlled load that can be programmed to be constant or variable, in several ranges from 0.1 µN to 0.1 kN. The AFM and Nanohead can be installed onto the motorized Z-stage using a quick-release connector. All the UNMT motions and signals are controlled with a dedicated PC and sophisticated control software package. Both the Nanohead and the AFM have sub-nanometer resolution in terms of displacement noise

floor. The Nanohead has a sub-micro-Newton force noise-floor, maximum ranges of Z–displacement and force of 300 µm and 500 mN, respectively. Instrument calibration and mechanical properties extraction for the Nanohead are performed automatically according to the ISO 14577 standard for instrumented nano-indentation [5].

2.2 Methodology

The Nanohead–1 shown in Fig. 2 is a sub-nanometer resolution nano-mechanical instrument integrated into the Universal Nano+Micro Tester and capable of performing nano-indentation tests, where the applied load F_z and penetration depth h are continuously monitored. The load versus depth plots are generated and processed from the collected data. The sample hardness H and the reduced elastic modulus E_r are calculated from the unloading segment of the curve as below. The reduced modulus is defined as follows:

$$E_r = S\frac{\sqrt{\pi}}{2\sqrt{A}} \ , \qquad (1)$$

where S is the unloading stiffness and A is the projected contact area. The stiffness S is calculated by fitting the unloading curve to the power law curve.

The nano-indentation hardness is defined by the ratio of the maximum load to the projected contact area:

$$H = \frac{F_{Z\max}}{A} \ . \qquad (2)$$

Figure 2: Photo of the UNMT nano-indentation module.

2.3 Experimental

Eight silica nitride and titanium nitride coated samples were blindly tested and characterized using the methodology and the instrument described above. Over 120 indentation tests were performed on all the samples. The samples were individually mounted on AFM specimen stubs and tested using a Berkovich tip, a 3-sided tip with an included angle of 142º, with a tip radius of approximately 80 nm.

The samples were tested using a trapezoidal loading profile that loaded in 5 s, held the maximum load for 5 s to allow for creep, and unloaded in 5 s. Several tests were conducted at various loading rates to ensure that the sample was not strain-rate dependant and so was not causing hardening at higher loading rates. The majority of the tests were performed at 3 N, but additional tests were performed at 1.5 and 4.5 N loads to ensure that resulting indentation depth was in the range of 150 nm, that is less than 5% of the thickness of coatings (4 mm). For the silica based coatings, substrate effects are observed on the data when indentation depth is approaching 30 – 40 % of the total coating thickness. Overall, different types of samples produced different reduced elastic modulus and nano-hardness values, indicating that both the nano-indentation technique and the instrument can distinguish different samples.

The results are summarized in the Table 1, yielding the following observations:

1. Similarly patterned TiN coated samples #5 and #6 have Er and H differences of 10%. Indentation into the silicon substrate of the sample #5 gave Er=72 GPa and H=10.5 GPa values, that are in the range of the typical values for silicon.
2. Mechanical properties of the pair of similarly patterned SiN coated samples #7 and #8 were found to be almost the same, suggesting that the same type of coating process was used.
3. TiN coated sample #1 had the highest hardness, though its Er was in the same range as for #7 and #8.
4. The softest was Cu coating #2, which had Er and H values of the order of magnitude lower than the rest of the samples.
5. Sample #4 had Er and H similar to the bare silicon.

A typical loading-unloading curve for the SiN coated wafer indent is shown in Fig 3.

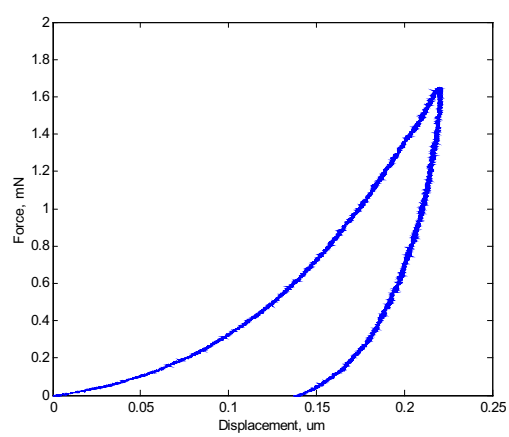

Figure 3: Loading-unloading curve on Si wafer.

Samples	#1 (SiN)	#2 (Cu)	#3 (SiN)	#4 (Si)	#5 (TiN)	#6 (TiN)	#7 (SiN)	#8 (SiN)
REM, Er, [GPa]	105.4±5.1	14.3±0.7	89.1±2.28	74.2±1.78	110.26±4.8	128.9±6.9	95.58±5.1	94.59±2.4
Hardness, H, [GPa]	16.64±0.43	0.87±0.04	13.71±0.36	12.5±0.55	15.21±0.78	16.01±0.4	14.33±0.72	13.83±0.3

Table 1: Mean values of elastic modulus and hardness with corresponding data scattering

3 MICROSCRATCH

3.1 Instrumentation

Micro and nano scratch testing is frequently used to investigate the behavior of thin films under various loading conditions. In particular, scratch testing can be used to investigate coating adhesion and failure modes. Many types of scratch probes are utilized for different types of coatings such as the DLC (diamond-like carbon) coating on magnetic disks. A tungsten carbide micro-blade with adjustable-angle holder has been used to evaluate durability of both freshly deposited bare overcoats and finished (lubricated and burnished) disk surfaces. The 0.8-mm micro-blade was chosen based on the contact stress analysis. For spherical or cylindrical contact geometry, the contact stresses are distributed deeper than a few nanometers of film thickness. For evaluation of thin films, however, the contact stresses should be concentrated within or near the films, which is achieved with the special micro-blade geometry. The schematic of the UNMT used in this study is shown in Fig. 4. The test procedure involved servo-controlled loading, multiple sensors, and precision motion.

3.2 Experimental

The fast and quantitative micro-scratch method was applied for ultra-thin coatings of 4.5 nm, covered with a mono-layer 1.5-nm lubricant. Three types of lubricated disks, two samples of each, were tested, named as Bias Carbon at 300V (samples 300_1 and 300_2), Bias Carbon at 150V (samples 150_1 and 150_2), and Bias Carbon at 50V (samples 50_1 and 50_2). Here, bias means film deposition voltage. The tests were performed at both A and B sides of the disk, three tests on each side at three different locations, OD (disk radius 47.5 mm), MD (disk radius 32 mm), and ID (disk radius 16 mm). During the test, while the micro-blade moved slowly against the film coating, progressive materials removal occurred. As shown in Figure 5, the measured coefficient of friction COF, normal force Fz, and electric contact resistance ECR were monitored with time. The normal force was gradually increased from 20 mN to 1000 mN. The coating film was cut through in 36 s, which corresponded to a critical load of about 300 mN. At that critical load, COF shifted to a higher value with a different slope, while ECR dropped to practically zero, because the micro-blade made contact with the conductive magnetic film after cutting through the coating.

The fact of the ultra-thin coatings break-through at the observed critical loads was confirmed by the integrated AFM images.

The UNMT allows for mapping of the tested surfaces, with 3-dimensional maps of coating durability, with a statistical analysis data for both the radial and the circumferential directions.

Wear tracks were examined after the test, using the Optical Surface Analyzer (made by Candela Instruments). They confirmed that wear tracks were very repeatable in both track pattern and track depth.

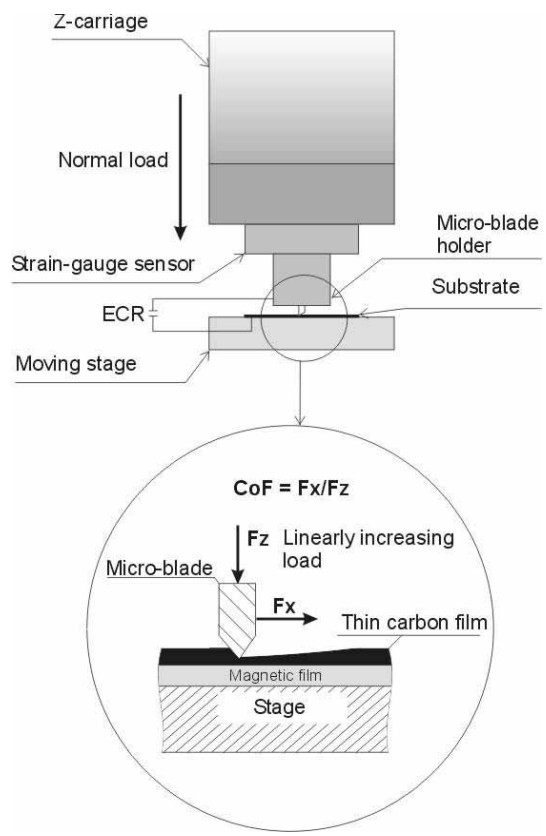

Figure 4: Schematics of UNMT micro-scratch module

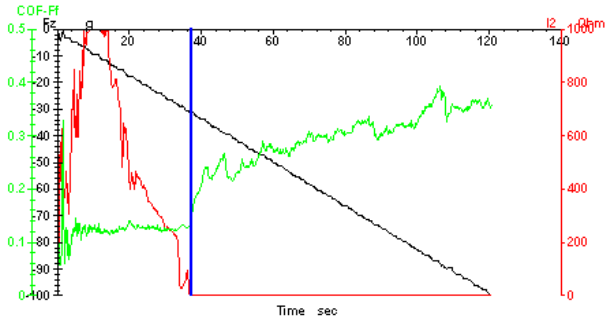

Figure 5: Coefficient of friction, contact electrical resistance and ramping vertical force versus time.

Table 2 summarizes the critical loads for the disk overcoats at different disk radii. The scratch resistance was the highest for the disks with the 300V-bias carbon, slightly lower for the disks with 150V-bias, and the lowest for the disks with 50V bias.

The Table 2 shows some radial non-uniformity of disk durability, with the scratch-resistance being the highest at the MD of the disks, lower at the ID and the lowest at the OD; this was then related to a sputtering chamber fixture, which was re-designed based on this durability data.

4 CONCLUSION

The Universal Nano+Micro Tester with integrated nano-indenter, micro-scratcher, optical microscope, AFM and many other tribology modules is a powerful tool for nano and micro mechanical characterization of thin films and other nano-technology specimens.

In the example with nano-indentation, both hardness and reduced elastic modulus indicated differences in mechanical properties between different SiN and TiN coated blindly selected samples. In terms of the data scattering, the reduced elastic modulus measurement results were more pronounced than the nano-hardness ones.

Micro-scratch experiments resulted in finding critical loads to breakthrough the ultra-thin films. Simultaneous coefficient of friction and contact electrical resistance measurements helped to "fingerprint" the samples.

5 REFERENCES

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Sample	Critical Load (g), at Disk Radius						
	16 mm		32 mm		47.5 mm		Average
	Side A	Side B	Side A	Side B	Side A	Side B	
300_1	32	35	44	45	28	31	37.3
300_2	35	36	46	40	34	41	
150_1	37	39	41	49	25	35	34.8
150_2	28	29	36	44	25	30	
50_1	30	20	25	30	18	20	
50_2	19	24	25	18	26	30	
Average	30.3		36.9		28.6		

Table 2. Micro-Scratch Test Results For Ultra-Thin DLC Coatings