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Evaluation of Mechanical Properties of Hard Coatings

Comprehensive mechanical testing of two coated metal samples was performed on the UNMT-1. The tests clearly distinguished brittle and ductile samples, with excellent correlation of the micro-wear, nano-hardness, scratch-hardness and scratch-adhesion data.

Description of Test Equipment

A number of tests were carried out on the Universal Nano+Micro Tester mod. UNMT-1. The general appearance of the tester is shown in Figure 1. It is a highly precision instrument for nano and micro mechanical and tribological testing of practically all types of thin films and coatings, including metals, ceramics, composites, polymers, etc. It provides a combination of rotary and linear, including reciprocating, motions in both vertical and horizontal directions, and measures numerous parameters with high precision, such as normal and lateral forces and torques in two to six axes in ranges from micrograms to kilograms, wear and deformation, contact acoustic emission, contact electrical resistance, temperature, etc. A normal-load sensor provides feedback to the vertical motion controller, actively adjusting the sample position to ensure the user-programmed either constant or changing load during testing. The tester has fully computerized motor-control and data-acquisition system, with test data acquired, calculated and displayed in real time, as well as stored for future retrieval. An optional nano-indentation head measures nano-hardness and elastic modulus of thin films. The unique modular design provides the capability of fast switching between multiple testing modes (such as friction, wear, micro-scratch, nano-indentation, AFM imaging, etc.) by simply swapping the easily-replaceable drive stages and sensors. A high-frequency multi-channel data-acquisition system, with data sampling at thousands times per second, allows for detection of almost instantaneous tiny sub-micro-contact and sub-micro-failure events in sophisticated test sequences. Integrated optical

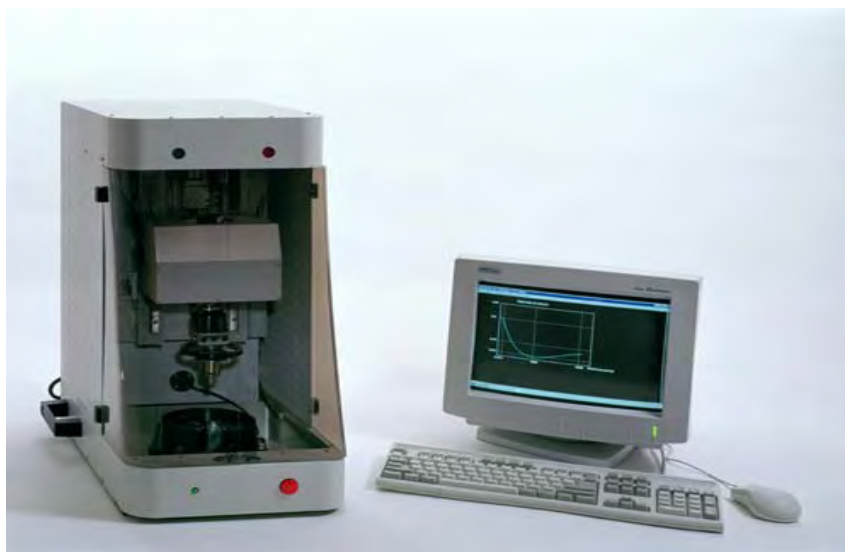


Fig. 1. Photo of the UMT tester



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microscopy allows for precision sample micro-positioning, digital video of the in-situ dynamics of surface failure, and micro-images of wear tracks, indents and scratches. Integrated atomic force microscope provides nano-imaging of test surfaces, wear tracks, indents and scratches, both periodically during testing and post-test.

Description of Four Test Procedures

The following test procedures were used for the most comprehensive evaluation of the sample coatings properties:

1. Reciprocating wear tests for evaluation of coating friction and durability.
2. Scratch-hardness tests under constant load for scratch resistance and micro-hardness measurement.
3. Scratch-adhesion tests under progressively increasing load for evaluation of the coating adhesion and scratch toughness properties.
4. Nano-indentation tests for coating nano-hardness and elastic modulus evaluation.

All four types of tests were performed on the same tester, re-configured for each type of test by installing corresponding replaceable attachments.

1. Friction and Wear Test

The friction and wear tests were carried out using a 1.6-mm sapphire ball and the ASTM G-133 standard test method for linearly reciprocating ball-on-flat sliding wear. The test sample was mounted on a table of the lower drive and was reciprocating in contact with a stationary ball, as shown in Figure 2. The ball in the ball holder was mounted via suspension on a dual-axis force sensor, attached to the carriage, which is a part of the electro-mechanical loading mechanism. A lateral slider on the carriage was used for positioning of the ball on the coated sample. An acoustic emission sensor was attached to the ball holder for monitoring the intensity of the high-frequency vibrations generated in the ball-to-sample interface.

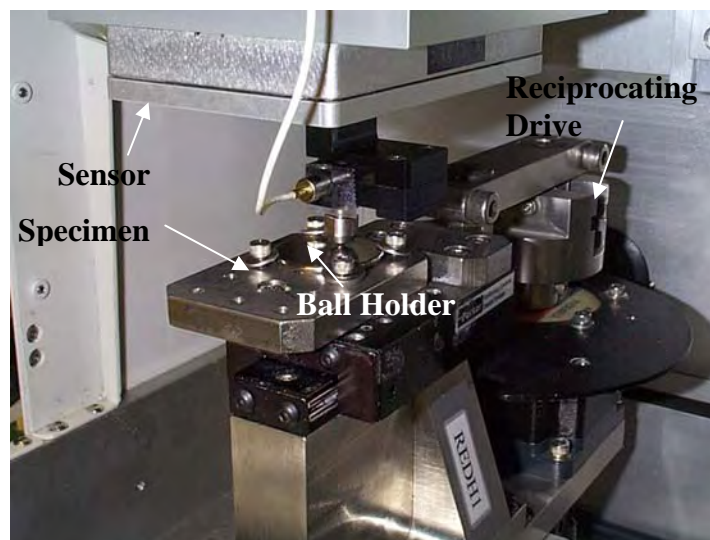


Fig. 2. UMT with upper stationary ball on lower reciprocating plate



The tests were conducted at a constant normal load of 2 N over a distance of 5 mm on the specimens, with a 3 Hz frequency of oscillation for 1 hour. The ball surface facing the specimen was changed before the start of each test by rotating the ball and inspecting under an optical microscope to make sure the new ball surface is fresh and smooth. The friction force F_x and normal load F_z were simultaneously recorded during the tests. The width W and depth H of the wear tracks were measured after the test at five different locations on the wear scar, and the average values were reported. Then a mean cross sectional area A (in mm^2) was calculated, and then specific wear rate WR was calculated:

$$WR = \frac{A * 5}{F_z * 3(2 * 0.005) * 3600} \text{ mm}^3/\text{N} * \text{m}.$$

COF and AE data as a function of time are plotted in Figure 3 for both samples.

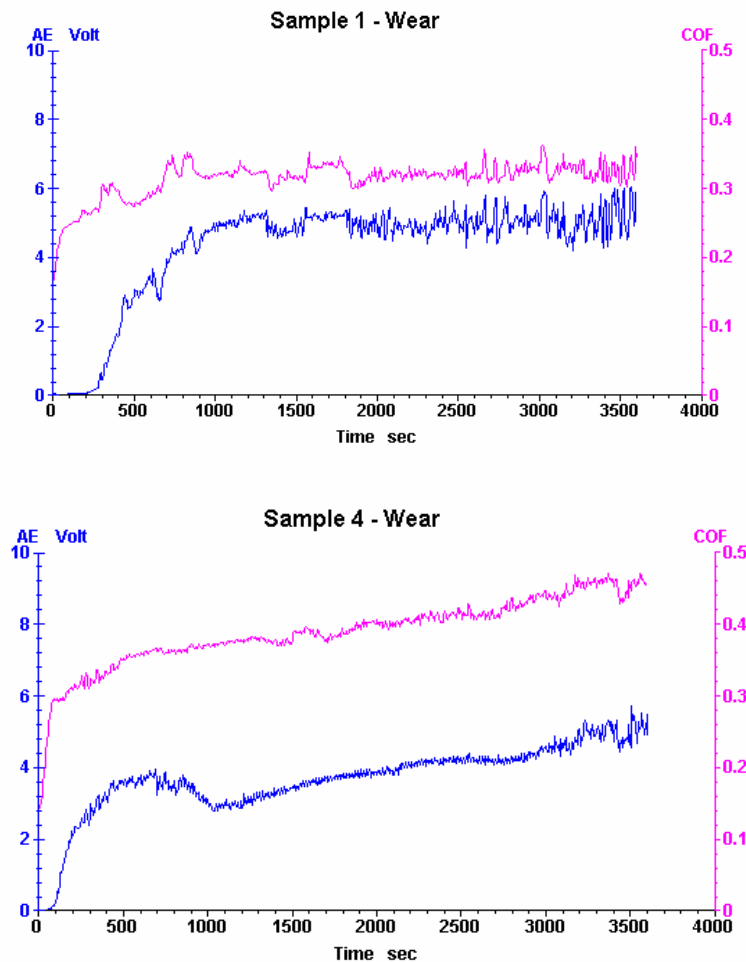


Fig. 3. Friction coefficient (pink) and AE (blue) signals during reciprocating wear tests



Table 1 shows the mean values of the coefficient of friction (COF) and AE signal after break-in period for both specimens, as well as wear scar width W, depth H, cross-sectional area A, and specific wear rate WR.

Table 1. Friction and Wear Data

Sample #	COF	AE	W, μm	H, μm	A, mm ³ *10 ⁶	WR*10 ⁶
1	0.32	5.1	105	0.49	25.4	1.18
4	0.41	4.0	87	0.46	20.1	0.93

In the reciprocating wear test, Sample 1 had lower friction, while Sample 4 demonstrated lower wear and acoustic emission, indicating less surface damage.

2. Scratch-Hardness Tests Under Constant Load

Micro-scratch-hardness tests were performed on both specimens using a diamond stylus with the tip radius of 5 μm. The test was conducted with the reference to the ASTM G171-03 standard method for determination of scratch hardness of materials, modified for thin coatings.

The scratch hardness of a material can be determined by producing a scratch on the sample surface by a sharp hard (diamond) tool with known tip geometry, under the constant, controllable load. Measuring the scratch width, one can define the sample scratch hardness as:

$$HS_p = k * F_z / W^2$$

where HS_p - scratch hardness number; k - constant; F_z - applied load; W - scratch width.

When the constant k is unknown, the scratch hardness can be determined by comparison of the scratch width on the sample and on a reference material with known hardness:

$$HS_{sample} = (HS_{ref} * W_{ref}^2 / F_{z,ref}) * F_{z,sample} / W_{sample}^2$$

where the indexes “sample” and “ref” refer to the test sample and reference material, correspondingly.

The diamond stylus in a stylus holder was mounted on the force sensor with a suspension (instead of and similar to the ball in the wear test per Figure 2). To provide automated programmable sample shifting for multiple scratches, the tested sample was mounted on a table of the lower linear drive. AE sensor was attached to the stylus holder to monitor the high-frequency signal, generated during scratching and indicating the intensity of material fracture. The normal load of 0.4 N was applied to the stylus and maintained constant with an active feedback from the force sensor. The scratch was produced by dragging the stylus along the sample surface with the upper lateral slider. The scratch length was 5 mm, the dragging speed – 0.5 mm/s. The test was repeated 3 times on each sample to verify the data consistency and repeatability. A polished fused quartz (with the hardness of 9.5 GPa) was used as the reference material for the scratch-hardness calculations.

COF and AE data as a function of time are plotted in Figure 4.

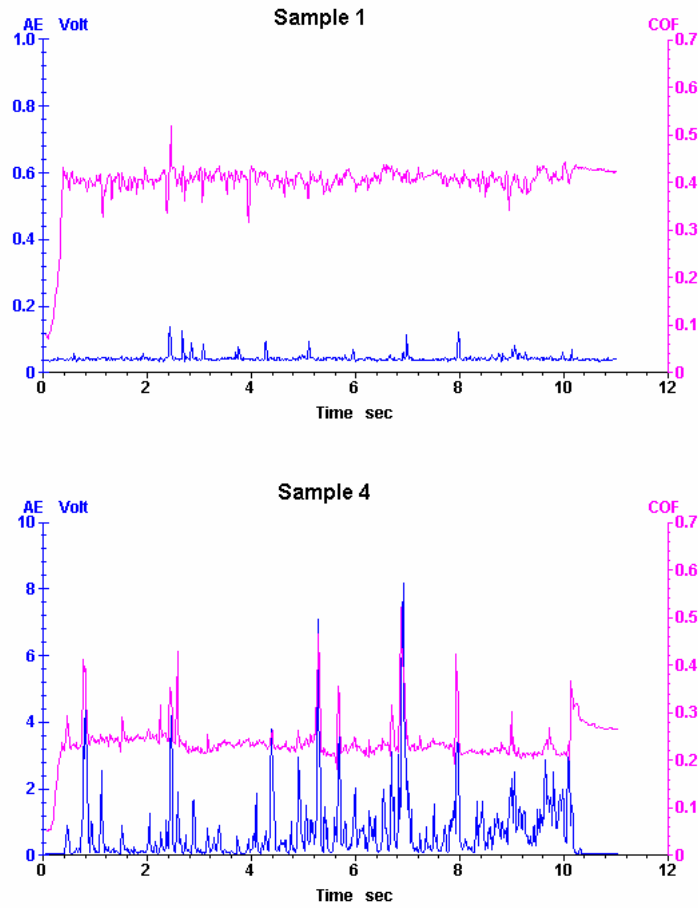


Fig. 4. Friction coefficient (pink) and AE (blue) signals during micro-scratch tests on Samples 1 and 4

The scratch width was measured on the same UNMT tester, by replacing the force sensor with an AFM head and taking the AFM images of the scratches. Both the force sensor with stylus and the AFM head were mounted on a fast-exchange fixture, which allows for their fast and easy swap (Figure 5).

Table 2 shows the mean values of the coefficient of friction (COF) and acoustic emission (AE) for both specimens, along with the scratch width W and scratch micro-hardness HS . The raw data for the scratch width measurements are shown in the Appendix.

Table 2. Micro-Scratch Test Data with Diamond Stylus

Sample #	Mean COF	Mean AE	W , μm	HS , GPa
1	0.49	0.04	5.87	23.4
4	0.27	0.67	5.0	32.2



Fig. 5. UNMT with the AFM head and optical digital microscope attachments

Figure 6 represents 3-D AFM images of the scratches on both samples. Screenshots with the

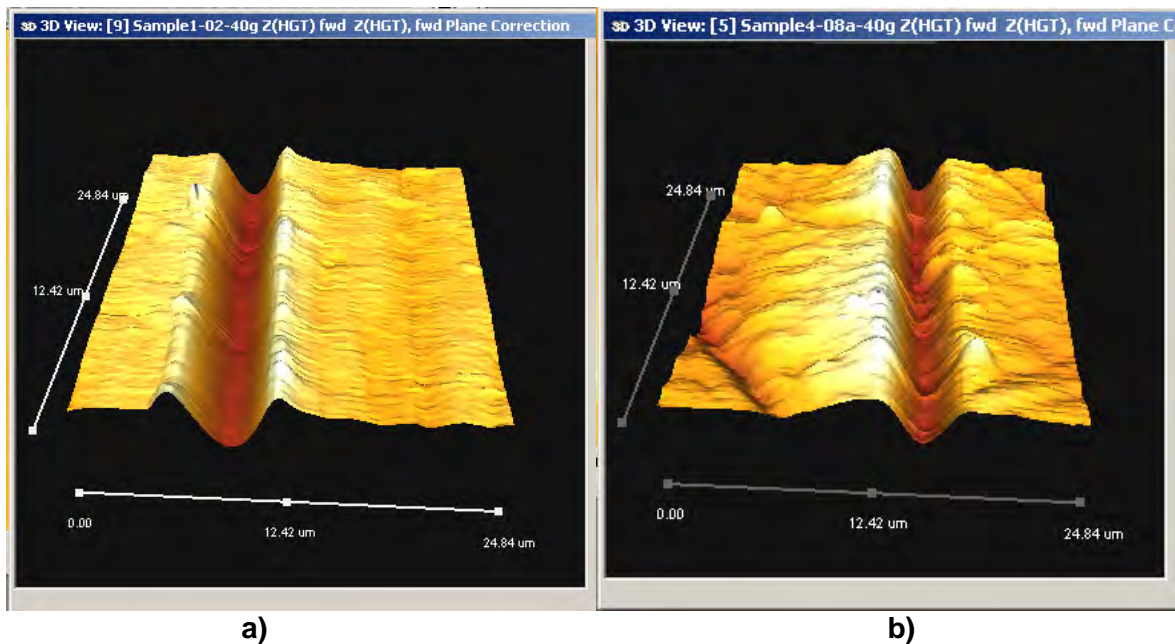


Fig. 6. 3-D AFM images of scratches on Sample 1 (a) and Sample 4 (b)
(scan size 25 x 25 μm)

scratch width measurements are shown in Figures 7 and 8 for samples 1 and 4, respectively.

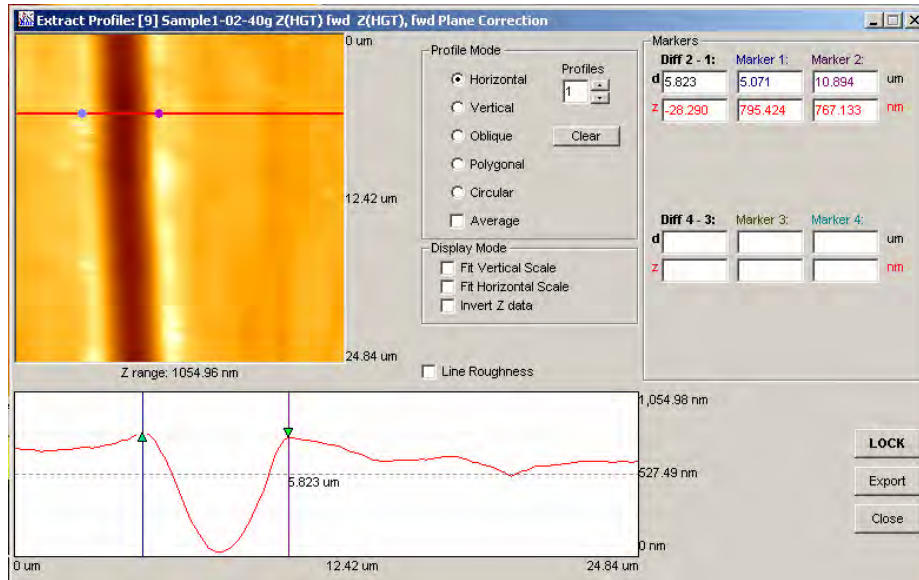


Fig. 7. Example of scratch width measurement for Sample 1

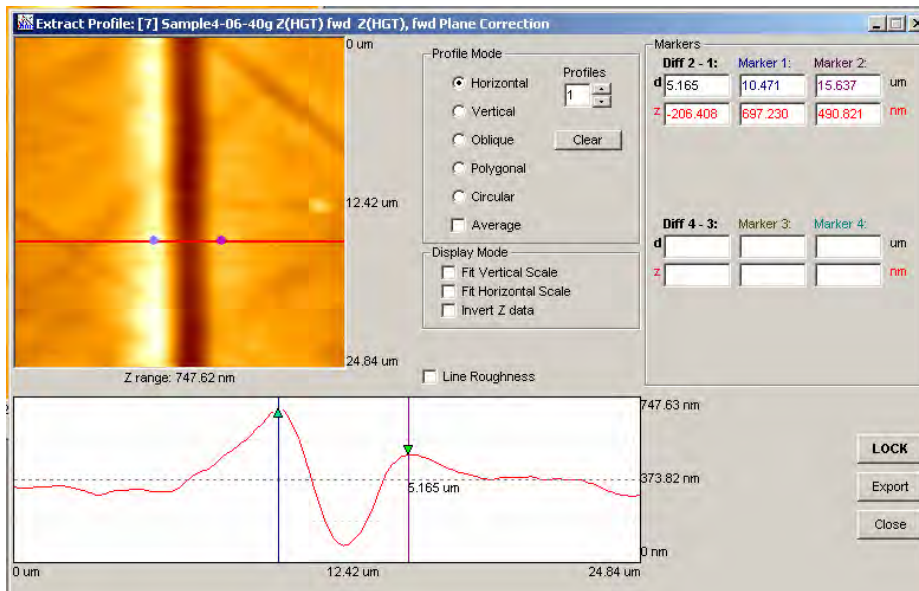


Fig. 8. Example of scratch width measurement for Sample 4

The AFM image analysis software allows for the sample surface roughness measurement on either the entire scanned image or a selected area. The example screenshot of the roughness measurement shown in Figure 9 below.

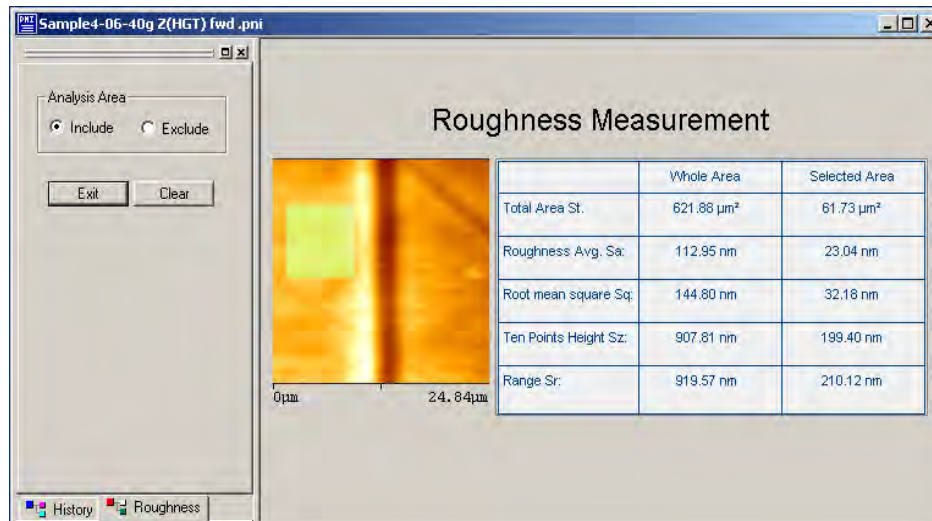


Fig. 9. Screenshot for surface roughness measurement (Sample 4)

In the micro-scratch-hardness test, Sample 1 exhibited lower hardness and acoustic emission, and higher dragging force than Sample 4. It indicates that the coating 1 is more ductile and is easier to deform plastically (deeper penetration of the stylus corresponds to higher dragging force), while the coating 4 is harder and more brittle (higher AE level corresponds to higher degree of material fracture).

3. Scratch-Adhesion Tests Under Increasing Load

The macro-scratch tests with progressively increasing load were conducted for evaluation of the coatings adhesion and scratch toughness using a diamond Rockwell indenter with the tip radius of 200 microns. As in the previous test, the indenter in its holder was mounted on the force sensor (of higher load range) with a suspension. The tested sample was mounted on the table of the lower linear drive to provide automated programmable sample shifting for multiple scratches. An AE sensor was attached to the indenter holder to monitor the high-frequency emission during scratching, indicating the intensity of material fracture.

The initial load of 1 N was applied to the indenter and stabilized for a few seconds. The scratch of the length of 10 mm was produced by dragging the indenter along the sample by the upper lateral slider with a speed of 1 mm/s. During the indenter dragging, the normal load was increased linearly from 1 to 60 N, according to the pre-programmed test script, while the load F_z , dragging force F_x and AE level were continuously monitored and saved. The tests were repeated several times on each sample to verify the data consistency and repeatability.

In the beginning of the scratch, at low loads, the indenter tip was mostly sliding on the sample surface, not causing coating damage. When the load reached a certain critical level, the coating started fracturing and/or delaminating from the substrate, since at higher load on the indenter having large tip radius, the depth of the mechanical stress distribution in the sample exceeded the coating thickness. This process reflected in sharp increases of both F_x and AE signals. The value of the critical load is used for the coating scratch toughness and adhesion evaluation.



An example of Fz, COF and AE plot, illustrating the critical load detection, is shown in Fig. 10.

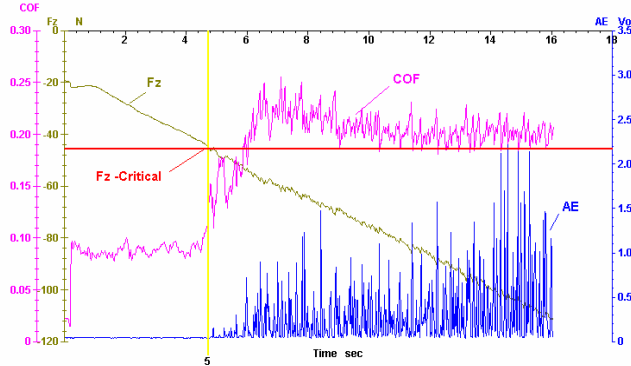


Fig. 10. Definition of the critical load (intersection of yellow and red lines) in scratch-adhesion test at progressively increasing load Fz.

Table 3 summarizes the COF and AE before and after coatings failure, and the critical loads.

Table 3. Scratch-Adhesion Test Data with Rockwell Indenter

Sample #	COF before Failure	COF after Failure	AE before Failure	AE after Failure	Critical Load, N
Sample 1	0.09	0.12	0.04	0.24	15.3
	0.08	0.14	0.04	0.92	20.6
	0.08	0.15	0.05	0.56	20.2
	0.08	0.17	0.04	0.81	18.6
	0.08	0.15	0.04	1.08	20.2
	0.08	0.12	0.05	0.89	18.8
Mean	0.08	0.14	0.04	0.75	19.0
Sample 4	0.09	0.18	0.05	0.46	28.9
	0.09	0.14	0.05	0.22	41.9
	0.08	0.15	0.05	0.29	27.6
	0.08	0.15	0.05	0.47	31.9
	0.08	0.14	0.04	0.46	29.7
	0.08	0.14	0.05	0.51	33
Mean	0.08	0.15	0.05	0.40	32.2

Typical graphs of COF and AE signals as functions of time are shown in Figure 11 for both samples 1 and 4. In the macro-scratch-adhesion test, Sample 1 exhibited lower critical load and higher AE level after the coating failure than Sample 4, which indicates that the coating 1 has lower scratch fracture toughness at higher loads, or lower adhesion to the substrate. The dragging (friction) coefficient values were almost identical for both samples, which confirms that AE is more sensitive to the coating failures.



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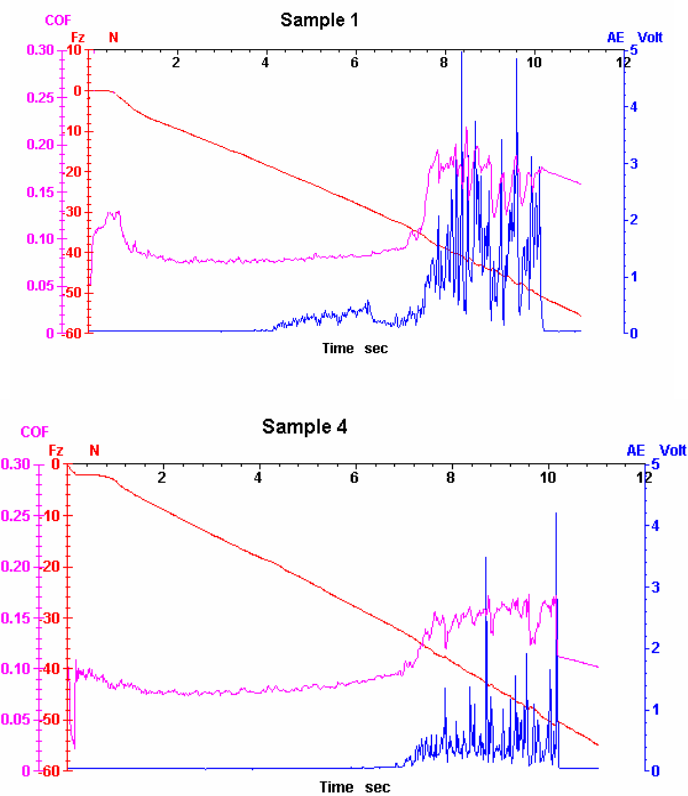


Fig. 11. Load Fz (red), dragging (friction) coefficient (pink), and AE (blue) signals during macro-scratch-adhesion tests

4. Nano-indentation Test

The tests were performed on the UNMT-1 with the nano-indentation head NH-1 (see photo in Figure 12), attached to the same fast-exchange fixture as described above for an AFM head.



Fig. 12. Photo of Nano-head NH-1



Both samples were tested using a standard nano-indentation technique at the maximum loads of 70 mN. Nano-hardness (H) and Reduced Elastic Modulus (Er) measurement results are summarized in Table 4 for six measured points on each sample.

Table 4. Nano-indentation test data

Tests	Sample # 1		Sample # 4	
	H, GPa	Er, GPa	H, GPa	Er, GPa
#1	19.53	169.60	24.42	191.89
#2	20.81	174.55	26.61	179.80
#3	20.75	154.30	26.34	178.97
#4	22.55	193.90	26.87	193.04
#5	20.22	171.53	26.35	198.08
#6	20.91	176.81	26.72	184.54
Average	20.79	173.45	26.21	187.72
Deviation(+/-)	1.51	19.8	1.22	6.77

The typical loading-unloading curves are shown in Figures 13 and 14.

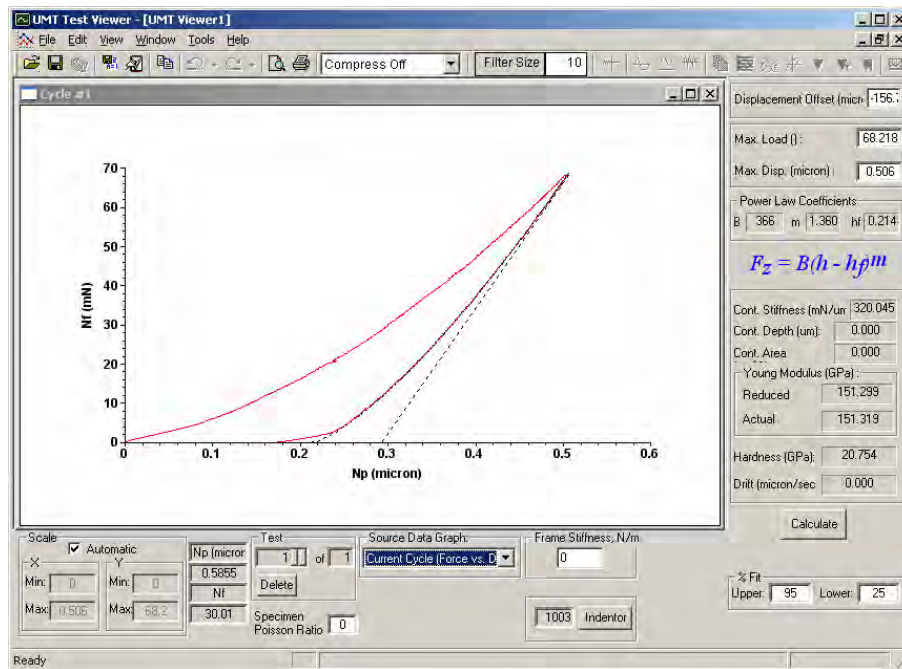


Fig. 13. Loading-unloading curve on Sample 1



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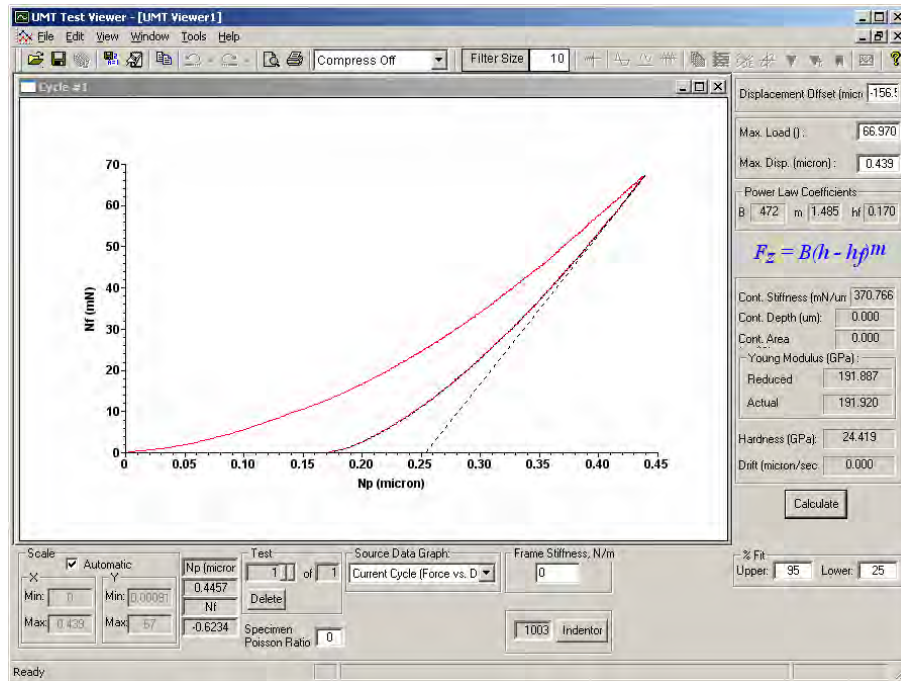


Fig. 14. Loading-unloading curve on Sample 4

In the nano-indentation test, Sample 4 showed higher values of both H and Er, while Sample 1 showed lower values and higher data scattering of Er. The scattering can be attributed to the surface roughness induced effects.

CONCLUSIONS

1. The Universal Nano+Micro Tester mod. UNMT-1 is uniquely capable of performing multiple tests for comprehensive precision evaluation of mechanical properties of hard coatings, including wear resistance, micro- and macro- scratch resistance, adhesion, micro- and nano-hardness, and elastic modulus. This capability is based on the modular design of the tester, allowing for inter-changeable attachments, precision servo-control of normal load, advanced sensors with simultaneous monitoring of normal and lateral forces, micro and nano wear and deformation, high-frequency acoustic signal and electrical resistance, as well as integrated digital optical microscope and AFM.
2. The test procedures on the UNMT-1 are proven to be effective for accurate quantitative characterization of durability and other mechanical properties of hard coatings.