AUTOMATED MULTI SAMPLE SCRATCH TESTING

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INTRODUCTION

Coatings are extensively used in a variety of industries for their functional properties, including hardness and erosion resistance enhancement of mechanical parts, low friction and high wear resistance for tribology, inertness and barrier for corrosion prevention, and countless other optical, electrical and magnetic applications. Scratch test is a widely used comparative measurement to evaluate the relative cohesive or adhesive properties of coatings.

Based on comparison of the critical loads at which failures appear, as well as of the cohesive or adhesive failure modes of the coating, the intrinsic quality of the coating and the interfacial integrity of the coating/substrate system can be evaluated.

IMPORTANCE OF AUTOMATED MULTI SCRATCH TESTING FOR QUALITY CONTROL

During the scratch test, a stylus slides over the sample surface at a linearly increasing load, leading to certain type of coating failures occurring at different load thresholds, called critical loads. The critical load is a function of many factors, including the stylus shape and radius, sliding speed, loading rate, coating adhesion to the substrate, residual stress at the interface, coating thickness, defects and others. Identical test parameters should be used in the same batch of test samples in order to rule out the influence of different test parameters and use the critical loads for comparative evaluation of coatings. Therefore, it becomes desirable to develop a way of making scratch measurement on multiple samples using the same test parameters, in order to avoid possible errors occurring during test setup and user operation. This is also an indispensable feature for efficient and reliable quality control of large quantity of test specimens in the mass production industries.

MEASUREMENT OBJECTIVE

In this application, the Nanovea Mechanical Tester is used to evaluate the adhesion of six coated samples using the multi sample scratch test. We would like to showcase the capacity of Nanovea Mechanical Tester in performing automated scratch tests on multiple samples with high efficiency and reproducibility.

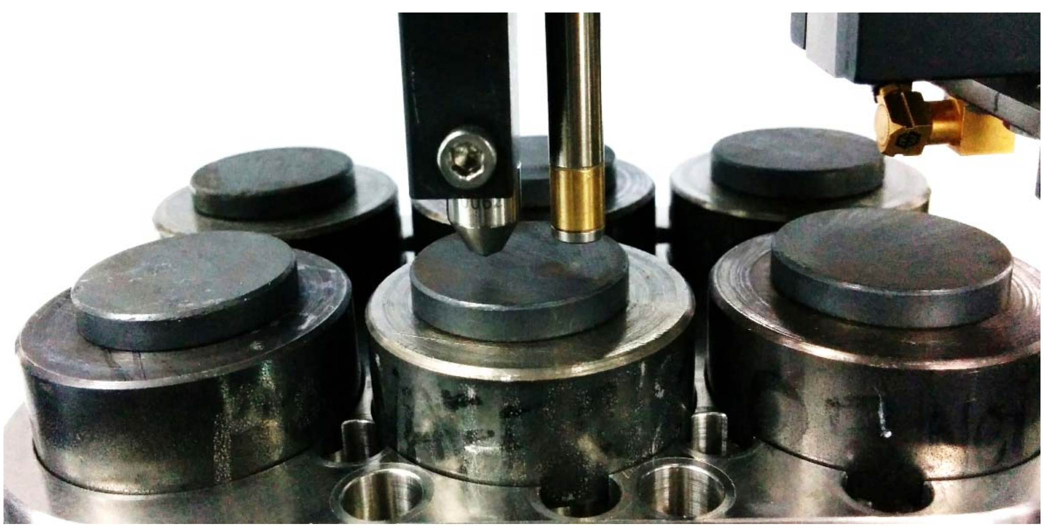


Fig. 1: Scratch tip and depth sensor on the test sample matrix.

TEST CONDITIONS

The Nanovea Mechanical Tester was used to perform a series of scratch tests on six coated samples sitting in the puck holder using the same test parameters as summarized in Table 1. As shown in Fig. 3, three scratches were planned on each of the six samples in the scratch map during test setup to ensure reproducibility of the results.

Test parameters	Value
Load type	Progressive
Initial load	5 N
Final load	30 N
Sliding speed	5 mm/min
Sliding distance	10 mm
Indenter geometry	Rockwell (120° cone)
Indenter material (tip)	Diamond
Indenter tip radius	100 μm
Atmosphere	Air
Temperature	24°C (room)

Table 1: Test parameters of the scratch tests.

Fig. 2: Scratch map on the sample matrix.

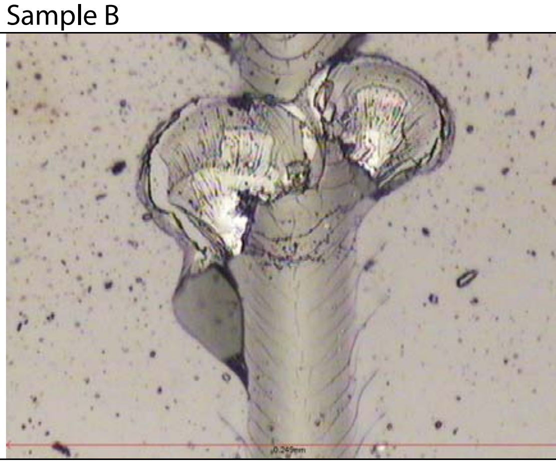
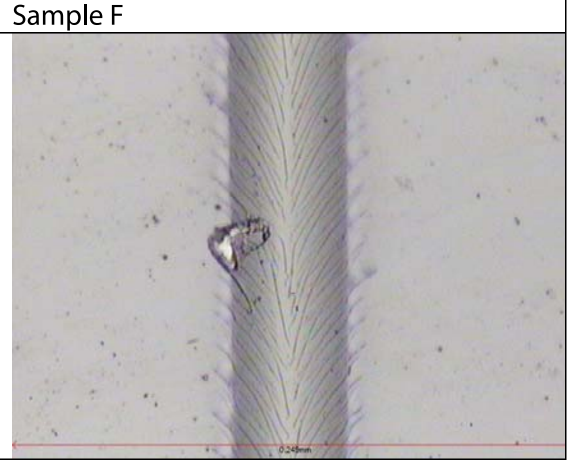
RESULTS AND DISCUSSION

The high-precision position control of the sample stage allows automated multi sample scratch test at the target area of the six test samples. Table 2 summarizes the scratch test results of all six coatings, where critical load, L_{c1} is defined as the load at which the first sign of adhesive failure occurs in the scratch track, and L_{c2} is the load after which repetitive adhesive failures take place. It is evident that Sample B exhibits the lowest L_{c1} and L_{c2} values at 9.4 and 10.7 N, respectively, compared to 13.0 and 17.9 N for Sample F, which shows the best performance.

Sample	**First delamination (L_{c1}) [N]**			**Repetitive delamination (L_{c2}) [N]**		
A	10.7	±	0.6	15.0	±	0.9
B	9.4	±	0.1	10.7	±	0.3
C	12.2	±	0.7	15.4	±	0.3
D	12.4	±	1.0	15.6	±	0.3
E	11.6	±	1.0	15.8	±	0.6
F	13.0	±	1.3	17.9	±	0.1

Table 2: Summary of critical load of the tested samples.

The scratch track of Sample B and Sample F were compared side-by-side to get more insight in the adhesion failure mechanism as shown in Fig. 3. It can be observed that before adhesive failure occurs, Chevron cracking takes place along the scratch track, featuring as small cracks orientated 45° with the scratch direction. When L_{c1} of 9.4 N is reached for Sample B, catastrophical wedging failure occurs in the form of annular-circular cracks that extends beyond the edge of the scratch track. This is attributed to high compressive stress the moving stylus creates in front and poor adhesion of the coating to the substrate. Repetitive wedging failures take place soon after the normal load increases above L_{c2} of 10.7 N. In comparison, Sample F exhibits a small buckling failure at a high L_{c1} of 13 N and such repetitive buckling failures take place after 17.9 N.

	Sample B	Sample F
L_{c1}		

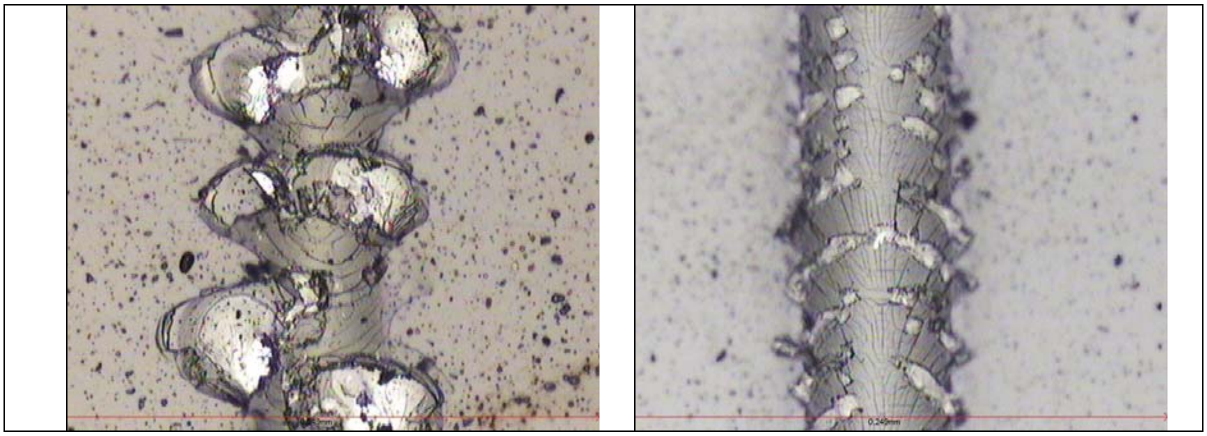
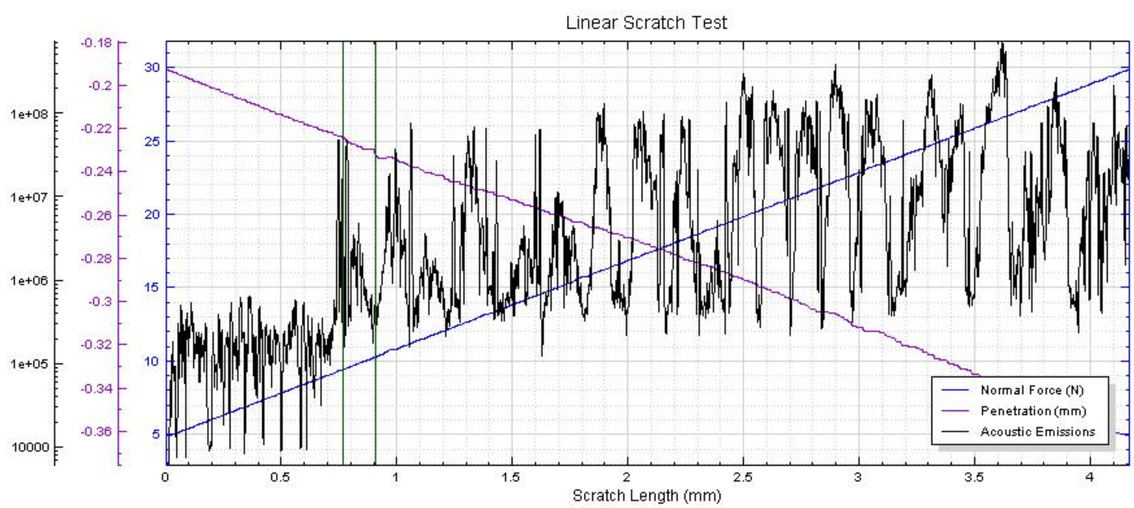


Fig. 3: Comparison of Sample B and Sample F at different critical loads.

The different adhesive failure modes and critical loads of Sample B and Sample F are also reflected in the evolution of the acoustic emission recorded in situ as displayed in Fig. 4. The first high spike of acoustic emissions (AE) for Sample B was detected at an early stage of the scratch test (~0.75 mm). This is quickly followed by repetitive AE peaks at around the same height, indicating the occurrence of continual similar failures thereafter. Sample F shows an AE spike at ~1.1 mm, corresponding to the first sign of chipping failure. As the load linearly increases, higher AE peaks start to occur after ~2.15 mm, which correlates to the failures in the next stage.

(a) Sample B:



(b) Sample F:

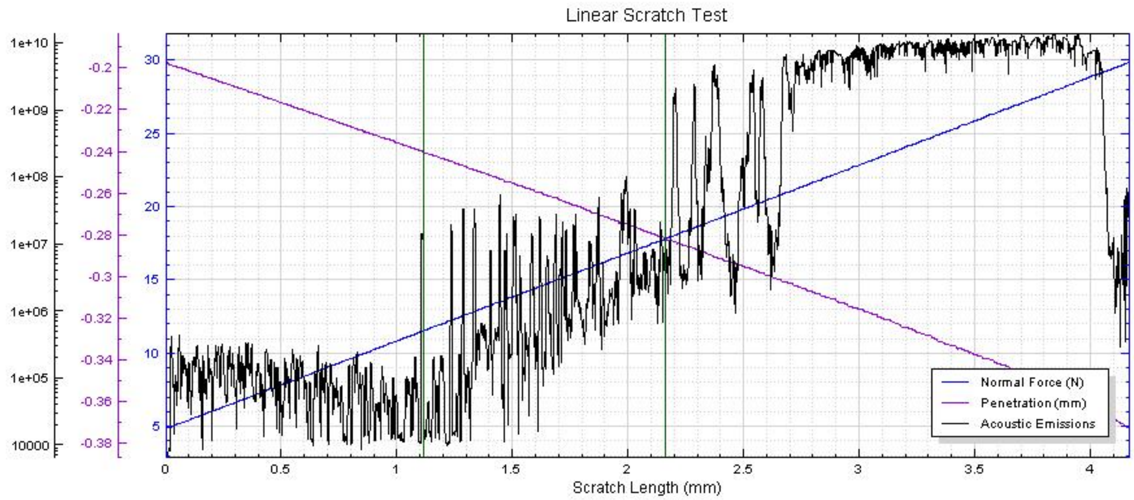


Fig. 4: Evolution of normal force, penetration and acoustic emission of (a) Sample B and (b) Sample F.

The scratch test matrix was finished within minutes with minimum operator handling and superior efficiency and repeatability, thanks to the high precision sample stage and user-friendly software interface of the Nanovea Mechanical Tester. Compared to conventional scratch test procedure, the multi sample scratch test in this study can substantially cut the time and possible errors in sample handling and test setup.

CONCLUSION

In this study, we showcased the capacity of Nanovea Mechanical Tester in performing multi sample scratch testing in a reliable and repeatable manner. It demonstrates the easy setup and precise position control on multiple samples. This is particularly important for quality control of a large quantity of test samples, it substantially eases the test setup and improves the test efficiency.

The Nano, Micro or Macro modules of the Nanovea Mechanical Tester all include ISO and ASTM compliant indentation, scratch and wear tester modes, providing the widest and most user friendly range of testing available in a single system. Nanovea's unmatched range is an ideal solution for determining the full range of mechanical properties of thin or thick, soft or hard coatings, films and substrates, including hardness, Young's modulus, fracture toughness, adhesion, wear resistance and many others.

In addition, optional 3D non-contact profiler and AFM Module are available for high resolution 3D imaging of indentation, scratch and wear track in addition to other surface measurements such as roughness.

To learn more about Nanovea Mechanical Tester or Lab Services.

MEASUREMENT PRINCIPLE

Principle of scratch test

The scratch testing method is a very reproducible quantitative technique. Critical loads at which failures appear are used to compare the cohesive or adhesive properties of coatings or bulk materials. During the test, scratches are made on the sample with a sphero-conical stylus (tip radius ranging from 1 to 200 μm) which is drawn at a constant speed across the sample, under a constant load, or, more commonly, a progressive load with a fixed loading rate. Sphero-conical stylus is available with different radii (which describes the "sharpness" of the stylus). Common radii are from 20 to 200 μm for micro/macro scratch tests, and from 1 to 20 μm for nano scratch tests.

When performing a progressive load test, the critical load is defined as the smallest load at which a recognizable failure occurs. In the case of a constant load test, the critical load corresponds to the load at which a regular occurrence of such failure along the track is observed.

In the case of bulk materials, the critical loads observed are cohesive failures, such as cracking or plastic deformation of the material. In the case of coated samples, the lower load regime results in conformal or tensile cracking of the coating which still remains fully adherent (which usually defines the first critical load). In the higher load regime, further damage usually comes from coating detachment from the substrate by spalling, buckling or chipping. Fig. 5 illustrates the principle of scratch testing.

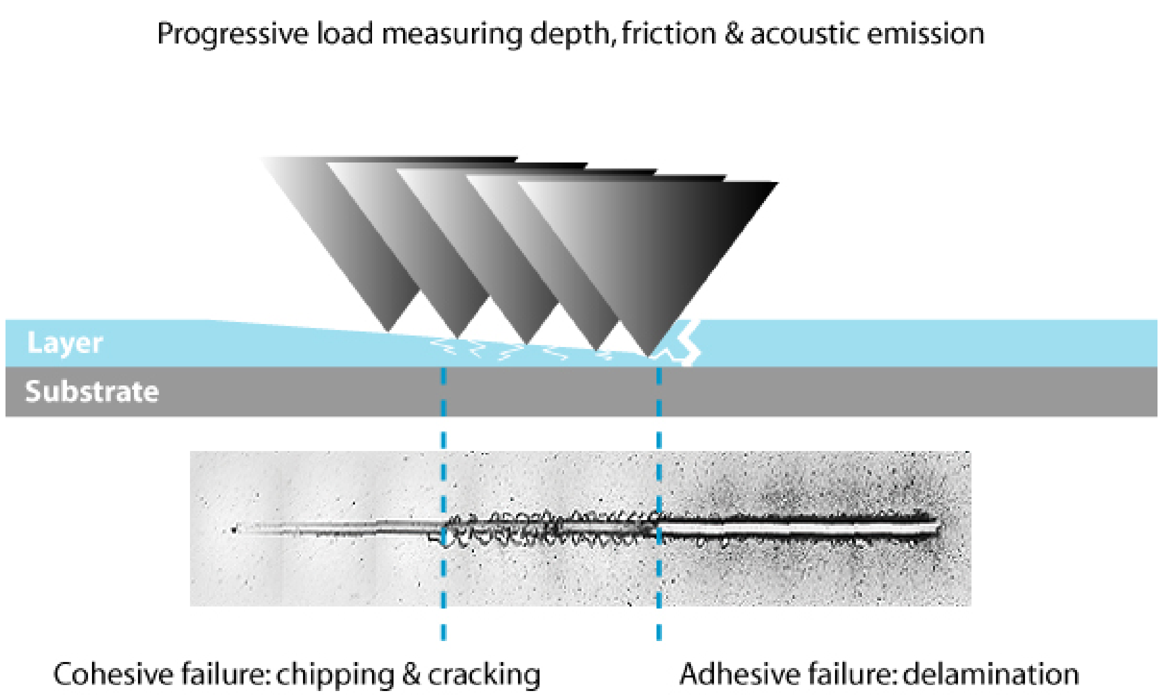


Fig. 5: Principle of scratch testing.

Comments on the critical load

The scratch test gives reproducible quantitative data that can be used to compare the behavior of various coatings. The critical loads depend on the mechanical strength (adhesion, cohesion) of a coating-substrate composite but also on several other parameters: some of them are directly related to the test itself, while others are related to the coating-substrate system. The parameters that determine the critical loads are summarized in Table 3.

Test specific parameters	Sample specific parameters
Loading rate	Friction coefficient between surface and indenter
Scratching speed	Internal stresses in the material for bulk materials
Indenter tip radius	Material hardness & roughness for coating-substrate systems
Indenter material	Substrate hardness and roughness
	Coating hardness and roughness
	Coating thickness

Table 3: List of parameters that determine the critical loads.

Means for critical load determination

Microscopic observation
This is the most reliable method to detect surface damage. This technique is able to differentiate between cohesive failure within the coating and adhesive failure at the interface of the coating-substrate system.

Tangential (frictional) force recording
This enables the force fluctuations along the scratch to be studied and correlated to the failures observed under the microscope. Typically, a failure in the sample will result in a change (a step, or a change in slope) in coefficient of friction. Frictional responses to failures are very specific to the coating-substrate system in study.

Acoustic emission (AE) detection
Detection of elastic waves generated as a result of the formation and propagation of microcracks. The AE sensor is insensitive to mechanical vibration frequencies of the instrument. This method of critical load determination is mostly adequate for hard coatings that crack with more energy.

Depth Sensing
Sudden change in the depth data can indicate delimitation. Depth information pre and post scratch can also give information on plastic versus elastic deformation during the test. 3D Non-Contact imaging such as white light axial chromatism technique and AFMs can be useful to measure exact depth of scratch after the test.

Other possible measurements by Nanovea Mechanical Tester:

Hardness and Young's Modulus, Stress-Strain & Yield Stress, Fracture Toughness, Compression strength, Fatigue testing and many others.