UNDERSTANDING COATING FAILURES USING SCRATCH TESTING

Prepared by

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INTRO:

Surface engineering of materials plays a significant role in a variety of functional applications, ranging from decorative appearance to protecting the substrates from wear, corrosion and other forms of attacks. An important and overriding factor that determines the quality and service lifetime of the coatings is their cohesive and adhesive strength.

Heaven [1] developed the scratch test for adhesion evaluation for the first time in 1950. Benjamin and Weaver [2] proposed critical load, L_c, as a quantitative value to evaluate the coating adhesion. The scratch test has become one of the most widely applied methods to evaluate coating adhesion at present. Over the past two decades, this technique is extensively used to assess the adhesion of PVD/CVD deposited superhard nanocomposite coatings, which usually exhibit a high residual stress at the coating/substrate interface. Moreover, the scratch test is an essential tool for quality control of materials used in wear/abrasive environment, allowing us to detect premature adhesive/cohesive failure of coatings in real-life applications.

MEASUREMENT OBJECTIVE

The process of scratching is carried out in a controlled and monitored manner to observe adhesive or cohesive failures. In this study, different coatings were tested and compared using Nanovea Mechanical Tester (see Fig. 1) to showcase the capacity of the instrument and investigate the behaviors of different coatings during the scratch tests.

Fig. 1: Image of the indenter tip over the scratches of the tested sample.

MEASUREMENT PRINCIPLE:

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 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 or 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. 2 illustrates the principle of scratch testing.

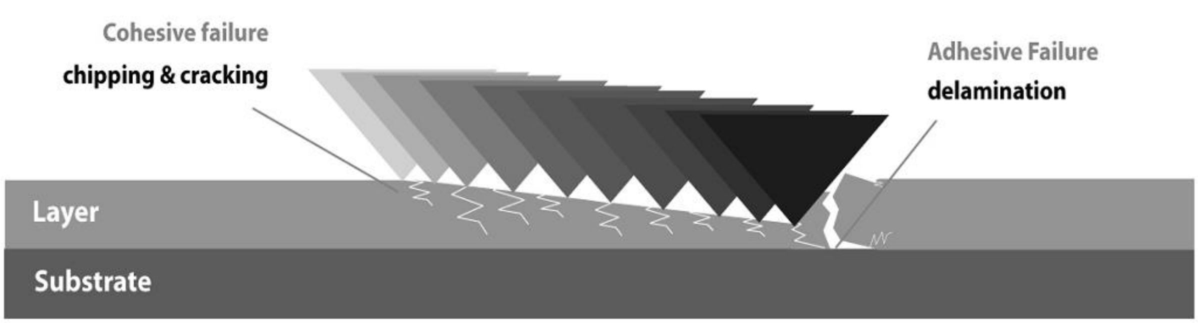

Fig. 2: Principle of scratch testing.

Comments on the critical load

The scratch test gives very 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 1.

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 1: 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.

TEST PROCEDURE:

Nanovea Mechanical Tester equipped with a sphero-conical diamond stylus (tip radius ranging from 20 to 200 μm) was used to perform progressive load scratch tests on a variety of different coated samples using Macro, Micro or Nano Scratch Tester Mode. Three tests were repeated at the same testing conditions on each sample to ensure reproducibility of the results. The test parameters of each material are listed in Table 2, and the tip geometry is illustrated in Fig. 3.

Coating	Anodized Al	DLC	Brittle Ceramic	CD coating
Load type	Progressive	Progressive	Progressive	Progressive
Initial Load	0.03 N	0.01 mN	0.01 mN	0.1 N
Final Load	100 N	15 N	40 N	25 N
Loading rate	200 N/min	30 N/min	80 N/min	25 N/min
Scratch Length	10 mm	3 mm	3 mm	20 mm
Scratching speed	20 mm/min	6.0 mm/min	6.0 mm/min	20 mm/min
Indenter geometry	120° cone	90° cone	120° cone	120° cone
Indenter tip material	Diamond	Diamond	Diamond	Diamond
Indenter tip radius	200 μm	20 μm	50 μm	200 μm

Table 2: Test parameters.

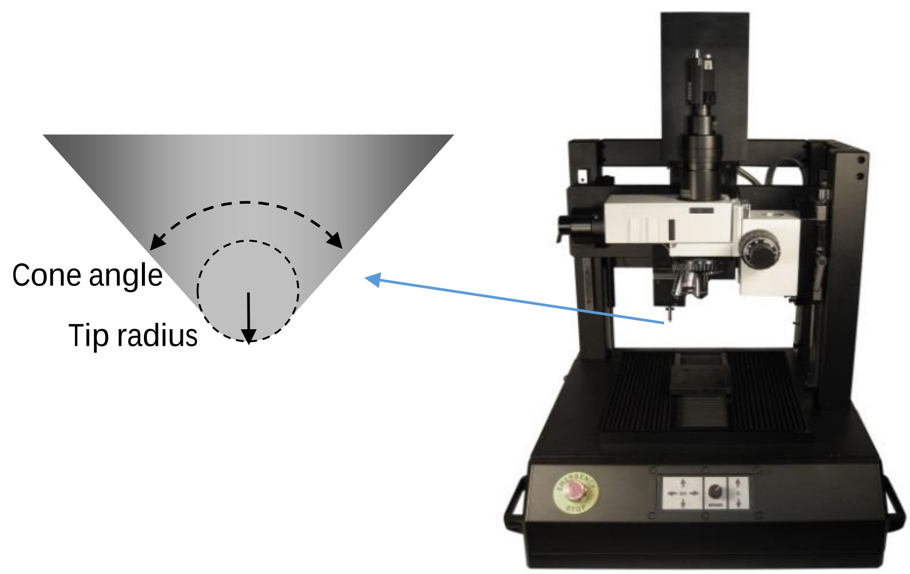

Fig. 3: Sphero-conical indenter.

RESULTS AND DISCUSSION:

Anodized Aluminum

As anodic films are generally much stronger and more adherent than most types of paint and metal plating, we performed the scratch test using a high final load of 100 N. As displayed in Fig.

4, we don't observe any coating failure until a high load of ~37 N is reached, where the anodized Al layer is removed and the metal substrate is exposed.

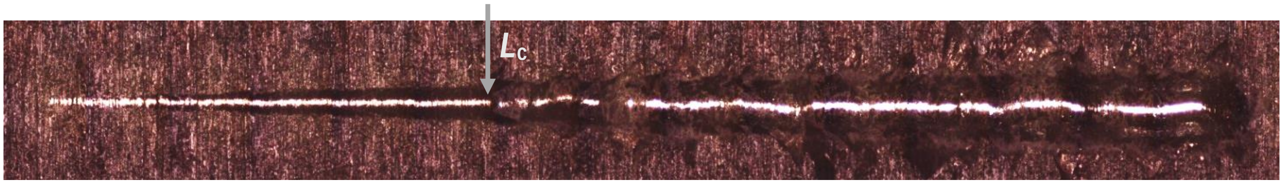

Fig. 4: Micrograph of full scratch of anodized Al.

Fig. 5 shows the plot of normal force, frictional force and penetration as a function of scratch length for the anodized Al sample. As the normal load progressively increases, the indention tip gradually sinks into the sample, indicated by the nearly-linear change of Penetration at the beginning of the test. At a scratch length of ~3.8 mm, which corresponds to a high load of ~37 N, a sudden change in the slopes of Frictional Force and Penetration curves takes place, followed by fluctuation of these two values. Such a behavior can be used as one of the implications that coating failure starts to occur.

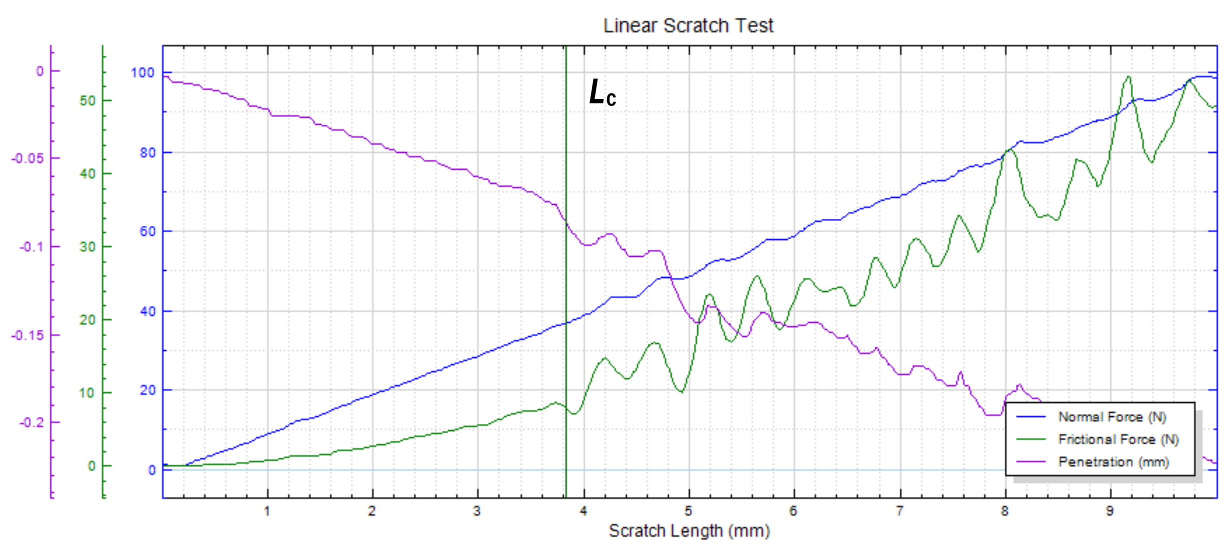

Fig. 5: Normal force, frictional force and penetration as a function of scratch length.

Diamond-Like Carbon

The diamond-like carbon, DLC, film exhibits a scratch track with a peculiar serrated pattern above L_{c1} of 3.5 N as shown in Fig. 6. Small periodic localized hemispherical coating chipping occurs at the side of the scratch track, indicating weak cohesive strength of this sample. Such a chipping phenomenon results in oscillations in the Friction Force as presented in Fig. 7. A complete removal of DLC coating in the scratch track takes place at L_{c2} of 7.5 N.

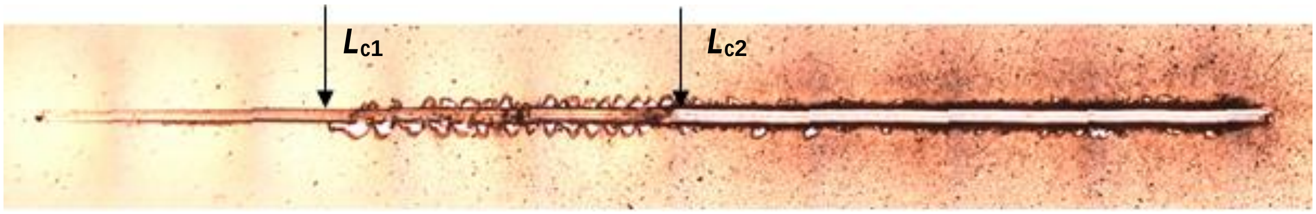

Fig. 6: Micrograph of full scratch of DLC.

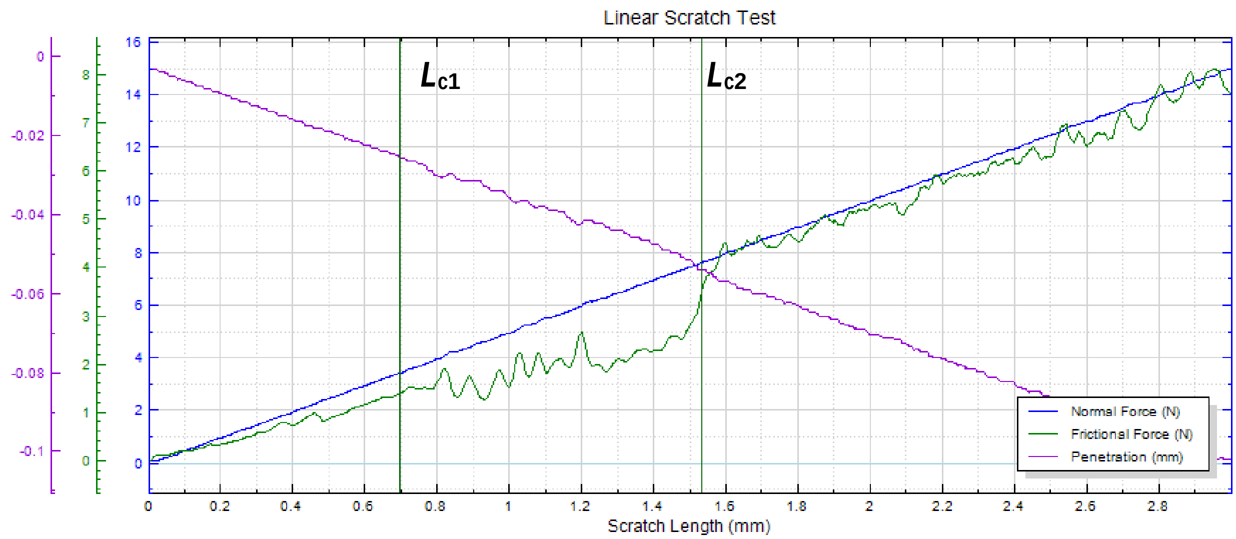

Fig. 7: Normal force, frictional force and penetration as a function of scratch length of DLC.

Brittle Ceramic Coating

Fig. 8 shows the micrograph of full scratch of a thick brittle ceramic coating – catastrophic coating failure is observed when L_{c1} of ~9 N is reached. This is also reflected by the intense fluctuation of frictional force and penetration during the test as shown in Fig. 9. When a critical coating thickness is reached, the coating becomes too stiff to buckle [3]. However, this leads to formation of compressive shear cracks through the thickness of the coating as illustrated in Fig. 10. The wedge crack firstly forms at the weak coating/substrate interface ahead of the moving indenter due to compressive shear stress. The continued forward motion of the stylus increases the stress and leads to growth of the interfacial cracks. Eventually the built-up high compressive stress lifts and bends the coating and results in spallation. Such a spallation behavior during the scratch test is often observed on brittle coatings with weak interfacial adhesion to the substrates.

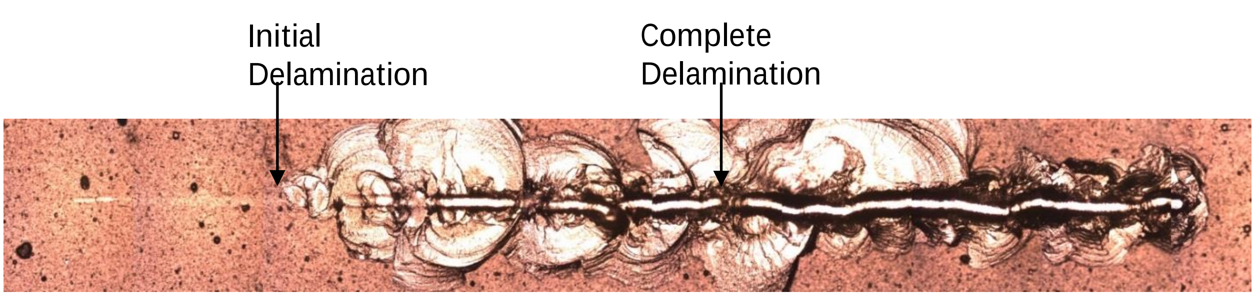

Fig. 8: Micrograph of full scratch of Brittle Ceramic Coating.

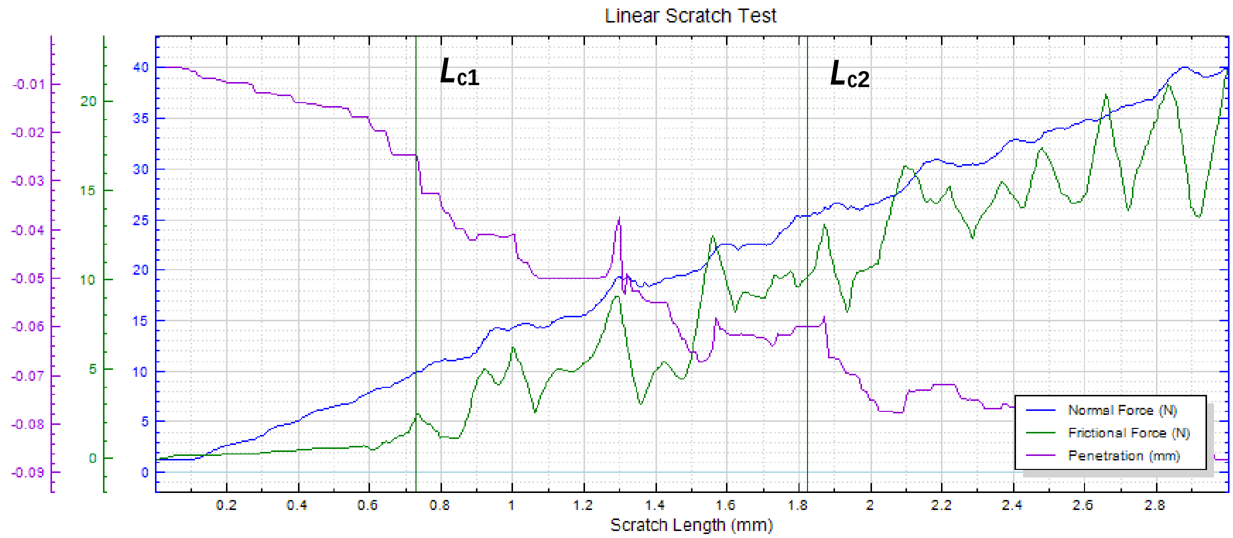

Fig. 9: Normal force, frictional force and penetration as a function of scratch length.

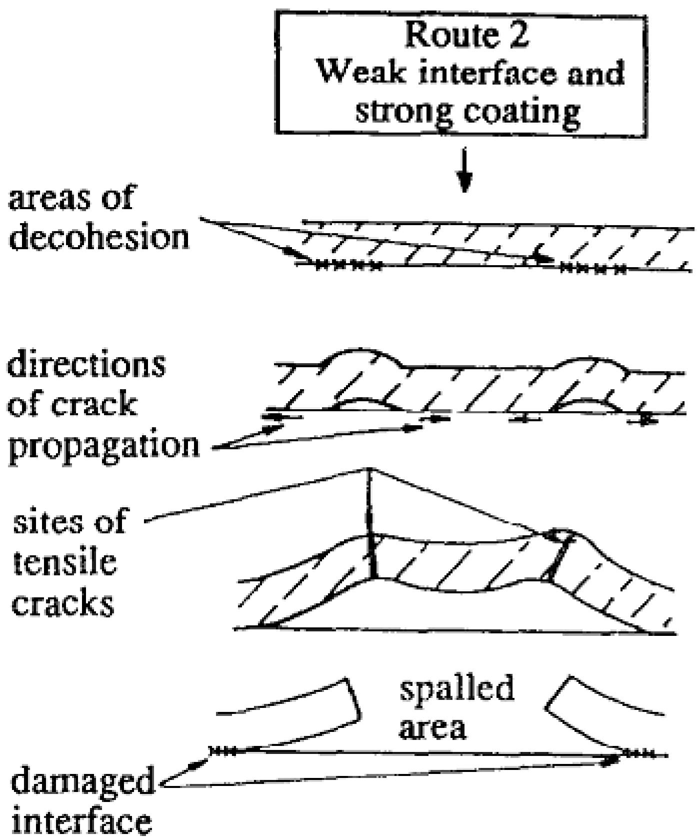

Fig. 10: Wedge spallation failure mode in the scratch test [3].

CD coating

Scratch resistance is an important factor determining the service lifetime of CD/DVD. The CD is composed of a 1.2 mm disc of polycarbonate with a thin layer of aluminum. The aluminum is protected by a film of lacquer as illustrated in Fig. 11. Fig. 12 shows the evolution of coating behavior during the scratch test. The initial mark on the label layer appears at the beginning of the test (a). As the stylus continues to penetrate into the coating, failure of the label (top) layer takes place at 3.2 N (b). Between 3 and 6 N, the label continues to delaminate while the aluminum layer remains adherent (c), until a load of 6.2 N is reached where large spallation of the aluminum layer takes place (d). Such a coating evolution can also be indicated by the progressive increase of friction coefficient as shown in Fig. 13.

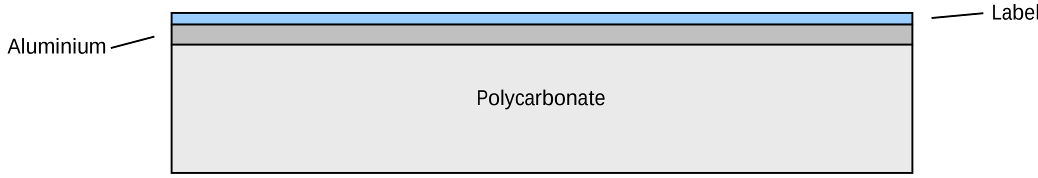

Fig. 11: CD coating architecture.

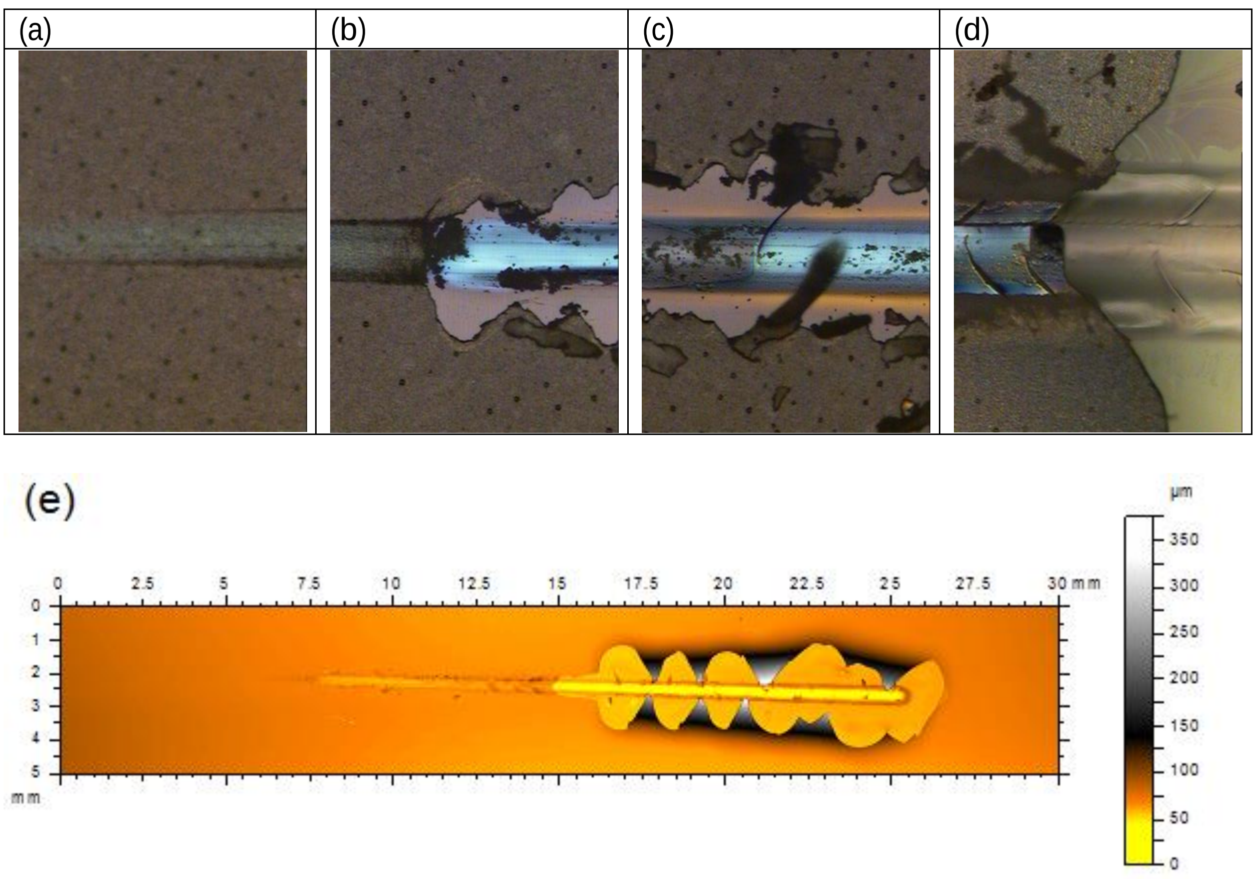

Fig. 12: Evolution of the coating during the scratch test and 3D image of the scratch.

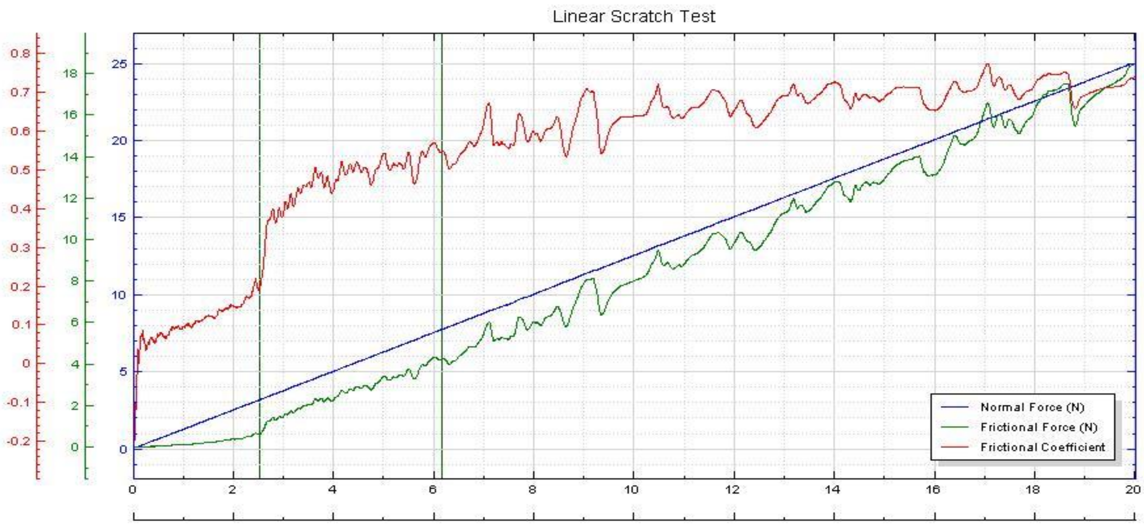

Fig. 13: Friction coefficient vs. Scratch distance.

CONCLUSIONS:

Nanovea Mechanical Tester at Scratch Tester Mode is a superior tool for evaluation and quality control of a variety of coatings. Different coatings exhibit drastically different adhesion/cohesion failures as demonstrated in the scratch tests. By applying loads in a controlled and closely monitored fashion, the tool allows users to identify quantitative and reproducible critical load failures. This type of information can help manufacturers improve and control coating quality.

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.

Learn More about the [Nanovea Mechanical Testers]

REFERENCES:

1. Heavens, O.S., *Some factors influencing the adhesion of films produced by vacuum evaporation.* J. Phys. Radium, 1950. **11**: p. 355-360.
2. Benjamin, P. and C. Weaver, *Measurement of Adhesion of Thin Films.* Proceedings of the Royal Society, 1960. **254**: p. 163-176.
3. Bull, S.J., *Failure mode maps in the thin film scratch adhesion test.* Tribology International, 1997. **30**(7): p. 491-498.