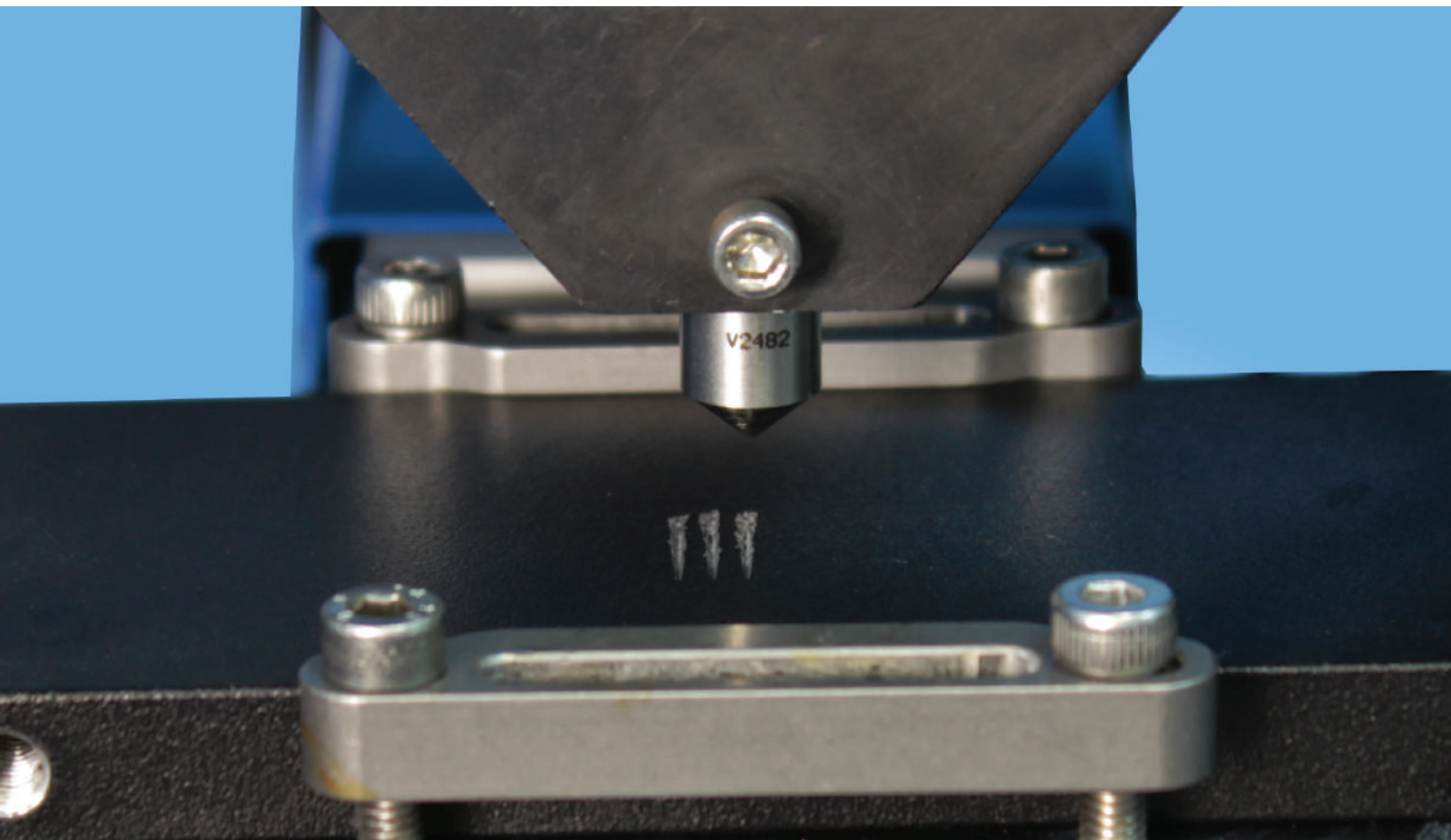


## *UNDERSTANDING COATING FAILURES*

— *USING* —

## *SCRATCH TESTING*



Prepared by  
**Jocelyn Esparza and Pierre Leroux**



## Introduction

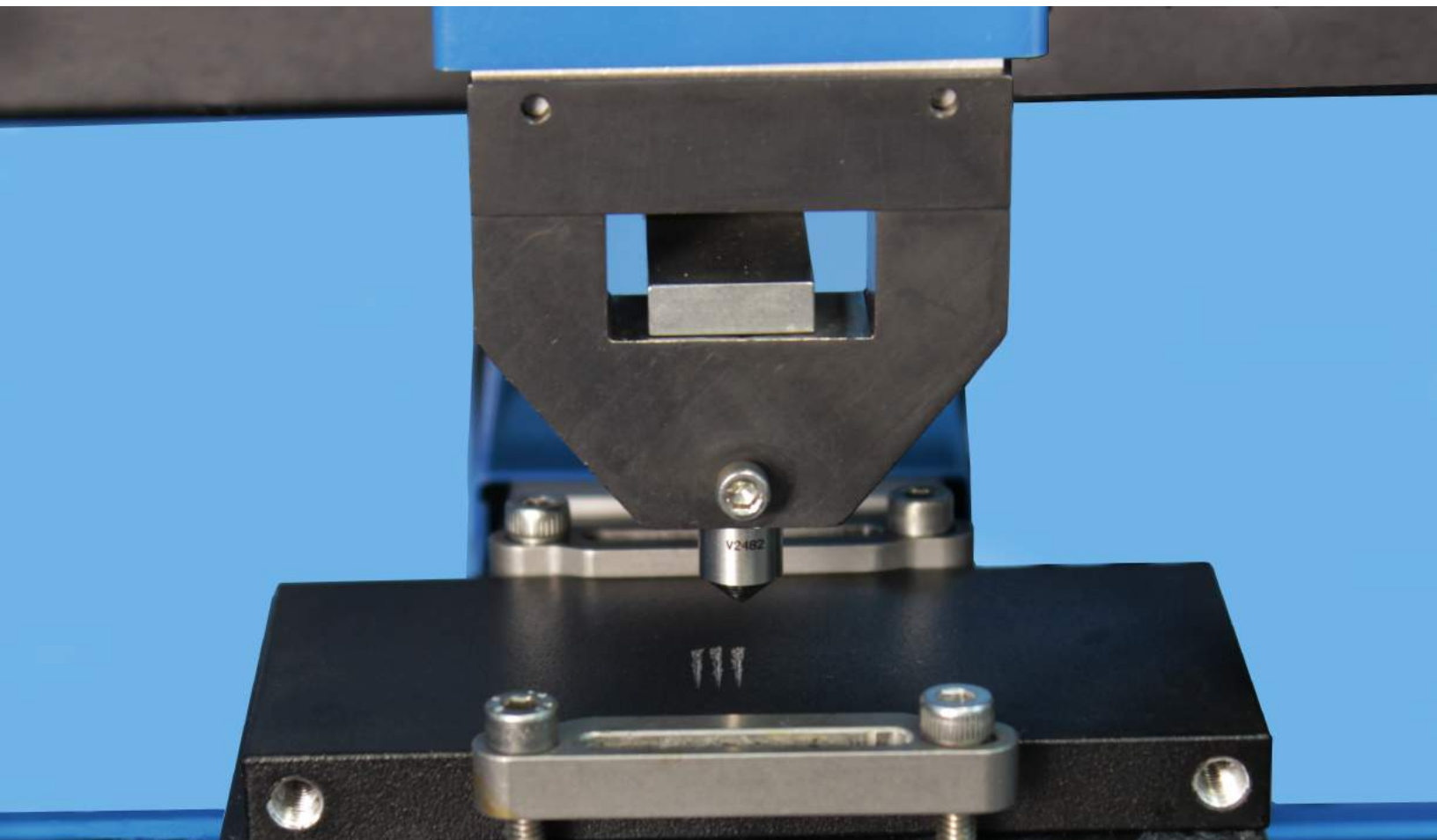
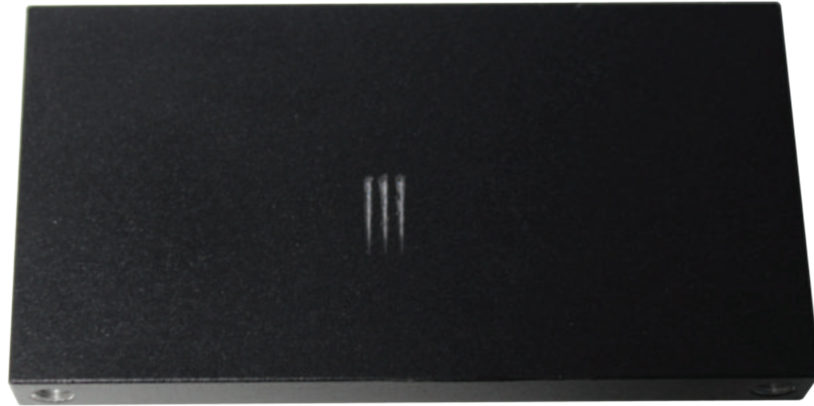
Surface engineering of materials plays a significant role in a variety of functional applications, ranging from decorative appearance to protecting substrates from wear, corrosion and other forms of deterioration. Important factors that determine the quality and service lifetime of coatings are their cohesive and adhesive characteristics.

## Importance of Understanding Coating Failures

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. Over the past two decades, this technique has been extensively used to assess the adhesion of PVD/CVD deposited super-hard nanocomposite coatings, which usually exhibits 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 environments, allowing us to detect premature adhesive/cohesive failure of coatings in real-life applications.

# MEASUREMENT OBJECTIVES

The process of scratching is carried out in a controlled and monitored manner to observe moments of adhesive and cohesive failures. In this study, different coatings were tested and compared using the Nanovea PB1000 Mechanical Tester (see Figure 1) to showcase the capacity of the instrument to investigate the adhesive and cohesive failures behaviors of different coatings during the scratch tests.



**Figure 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 $\mu\text{m}$ ) which travels at a constant speed across the sample, under a constant load, or a progressive load with a fixed loading rate.

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 often results in conformal or tensile cracking of the coating without full delamination. In the higher load regime, further damage usually comes from coating detachment from the substrate by spalling, buckling or chipping. Figure 2 illustrates the principle of scratch testing.

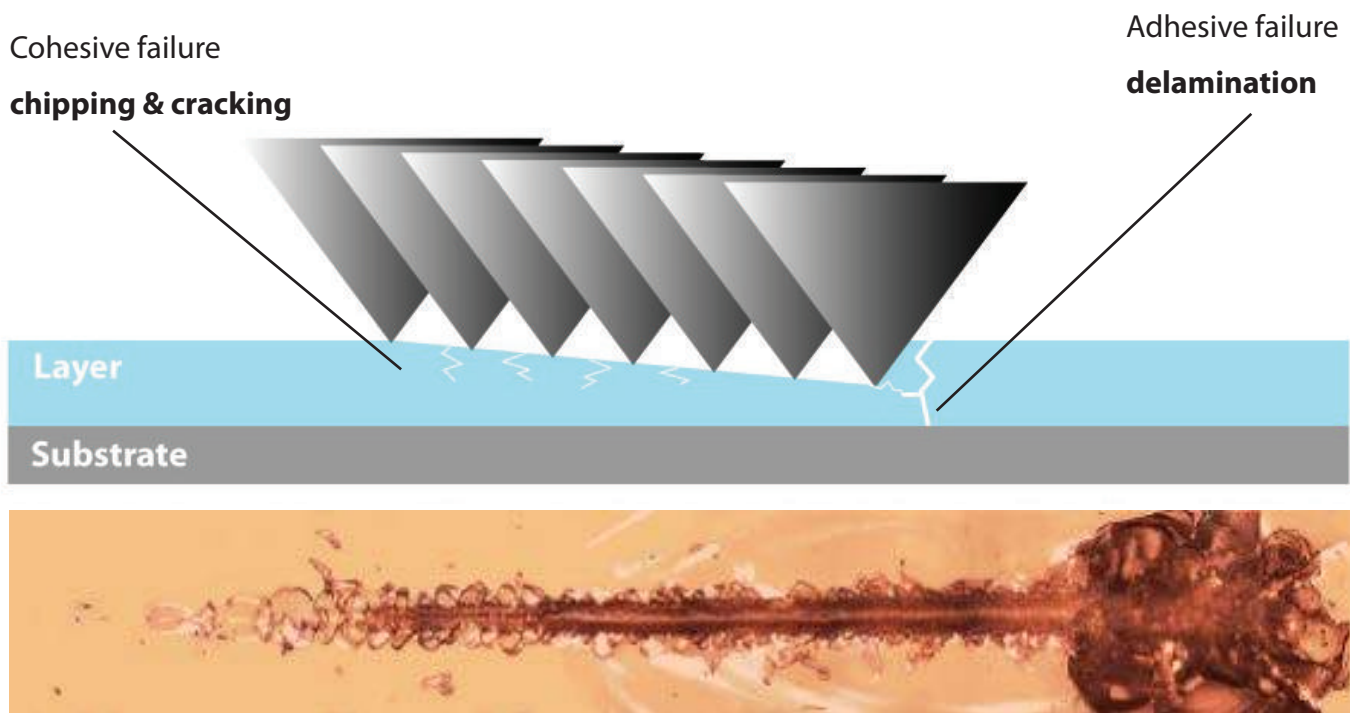


Figure 2: Principle of scratch testing.

## 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 influence the critical loads are summarized in Table 1.

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

**Table 1: List of parameters that influence the critical loads.**

The strength of scratch testing comes from the reproducibility of the test and the sensitivity to detect quantitatively small changes in the cohesive and adhesive properties that comes from changes in coating manufacturing processes or batch variations.

## Means for critical load determination

### ***Microscopic observation***

This is the most informative 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 sensitivity of this technique allows to detect the beginning of internal cracking before they become observable by microscope observation. Level of energy (aJ) produced by AE can be quantified.

### ***Depth Sensing***

Sudden change in the depth data can indicate delamination. True and residual scratch depth information with surface reference correction can also give information on plastic versus elastic deformation during the test. Chromatic 3D Non-Contact imaging and AFMs can be useful to image the full scratch profile after the test.

# Test Procedure

Nanovea Mechanical Tester (micro module) equipped with a sphero-conical diamond stylus was used to perform progressive load scratch tests on a variety of different coated samples. The test parameters of each material are listed in Table 2, and the tips geometries are given in Figure 3.

Coating	Aluminum Chromium Nitride (AlCrN)	Diamond Like Carbon (DLC)	Titanium Nitride (TiN)	CD
Load Type	Progressive	Progressive	Progressive	Progressive
Initial Load	0.2 N	0.01 N	0.5 N	0.1 N
Final Load	8 N	15 N	125 N	25 N
Loading rate	8 N/min	30 N/min	75 N/min	25 N/min
Scratch length	3.0 mm	3.0 mm	4.5 mm	20.0 mm
Scratch speed	3.0 mm/min	6.0 mm/min	2.7 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	50µm	20µm	200µm	200µm

Table 2: Test parameters.

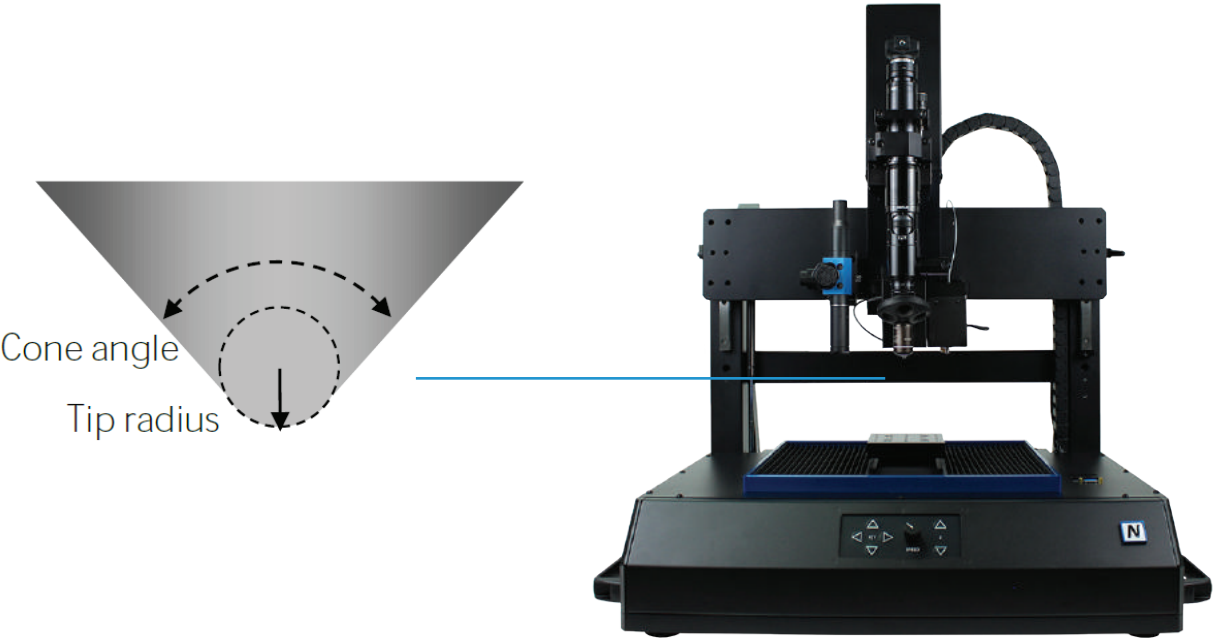


Figure 3: Sphero-conical indenter.

# RESULTS AND DISCUSSION

## AlCrN Coating

In the tooling industry, studying the adhesive and frictional properties of a coating that is applied to cutting tools supports R&D efforts of creating an optimal coating for every tooling application. One type of coating material known as aluminum chromium nitride (AlCrN) was measured for this study. After the scratch test was performed, cracking started to appear within the scratched region and the acoustic emissions showed a spike in energy when the coating first failed. This occurred at a critical load ( $L_{c1}$ ) of 1.36 N which is marked in the Figure 4 graph and Figure 6 micrograph. The acoustic emissions appear to stabilize once the indenter applies a normal force of 2.71 N or more, which is the second critical load ( $L_{c2}$ ). The scratch shows visual signs of complete delamination at this point as well. The coefficient of friction is constant until failure starts with increase coefficient of friction. As the tip passes through the coating and reaches the substrate in this zone, the coefficient of friction varies but in average it is fairly stable until the tip penetrates deeper in the substrate toward the end of the scratch. True and residual depths are shown in Figure 5. Full 3D profiles taken by white light chromatic technique are shown on Figure 7. 2D profiles along the scratch and perpendicular to the scratch are shown in Figure 8. This allows the study of pile up and the calculation of the volume displaced by the scratch test.

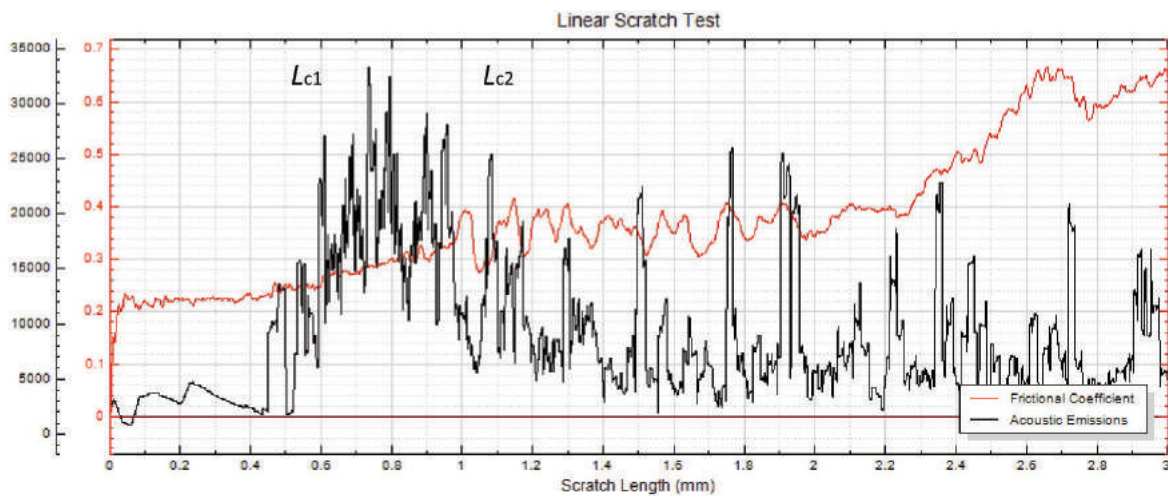


Figure 4: Coefficient of Friction and linear Acoustic Emissions vs Scratch Length.

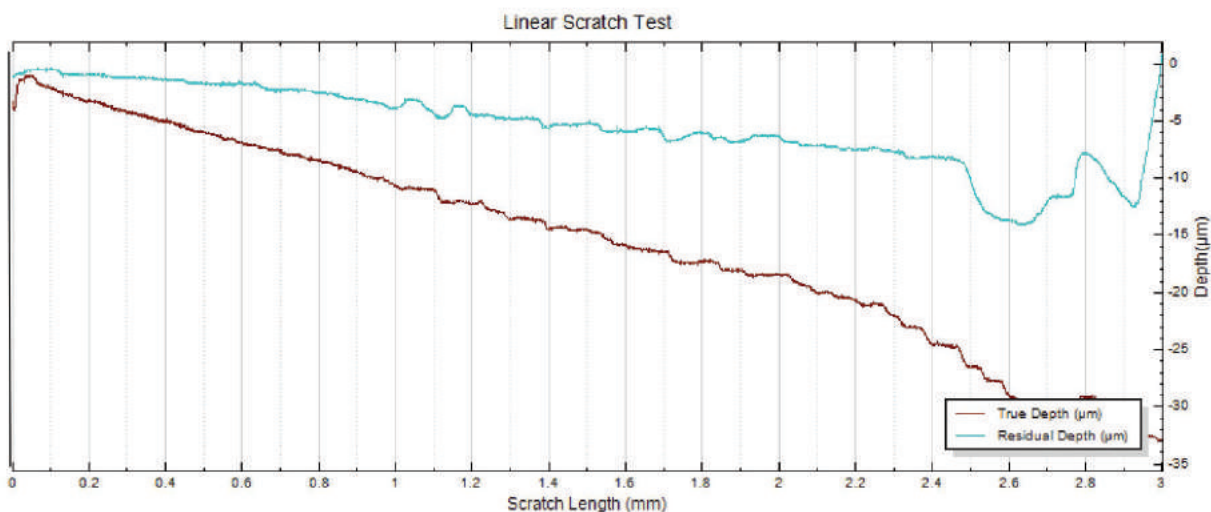


Figure 5: True and Residual Depth vs Scratch Length.



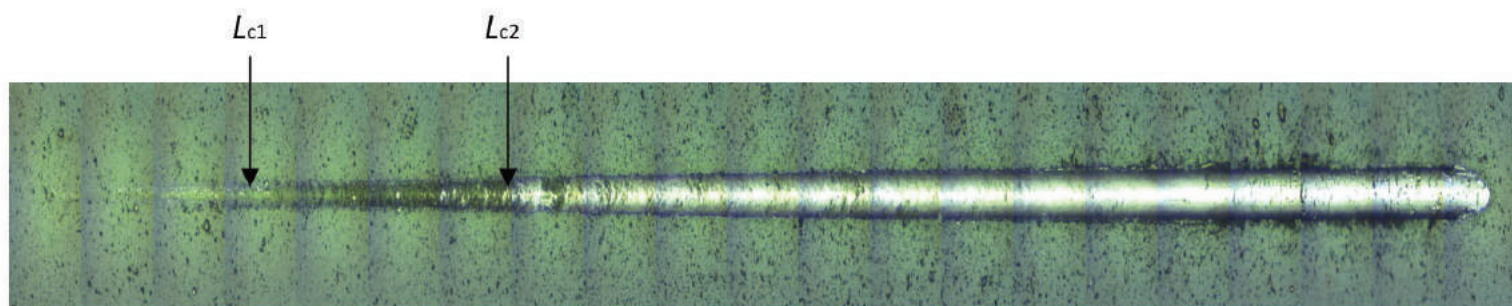


Figure 6: Full length micrograph of scratch AlCrN coating.

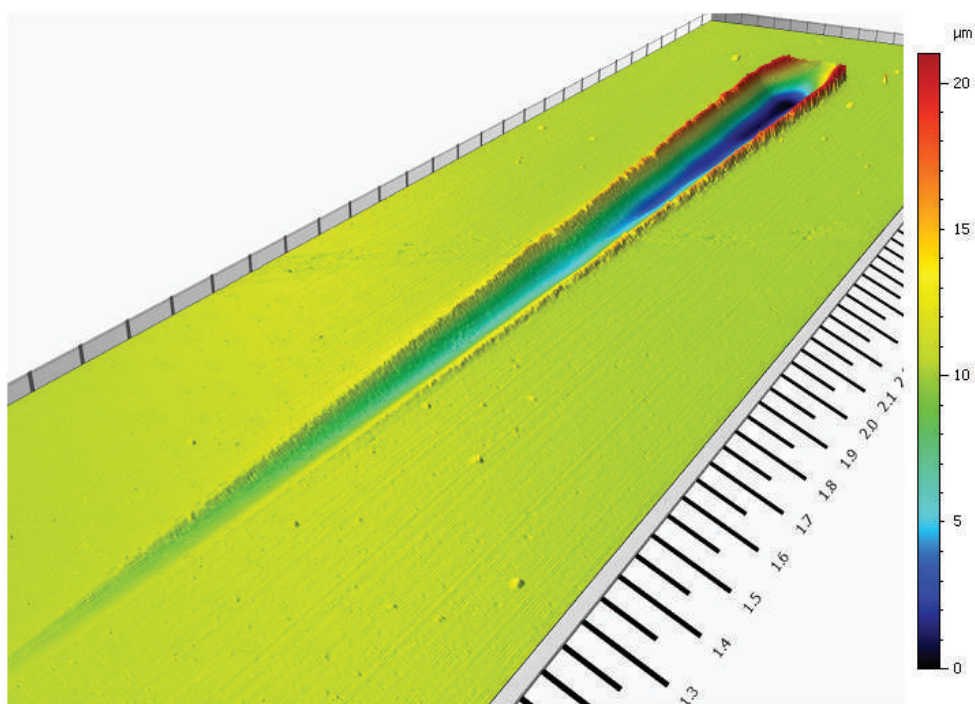
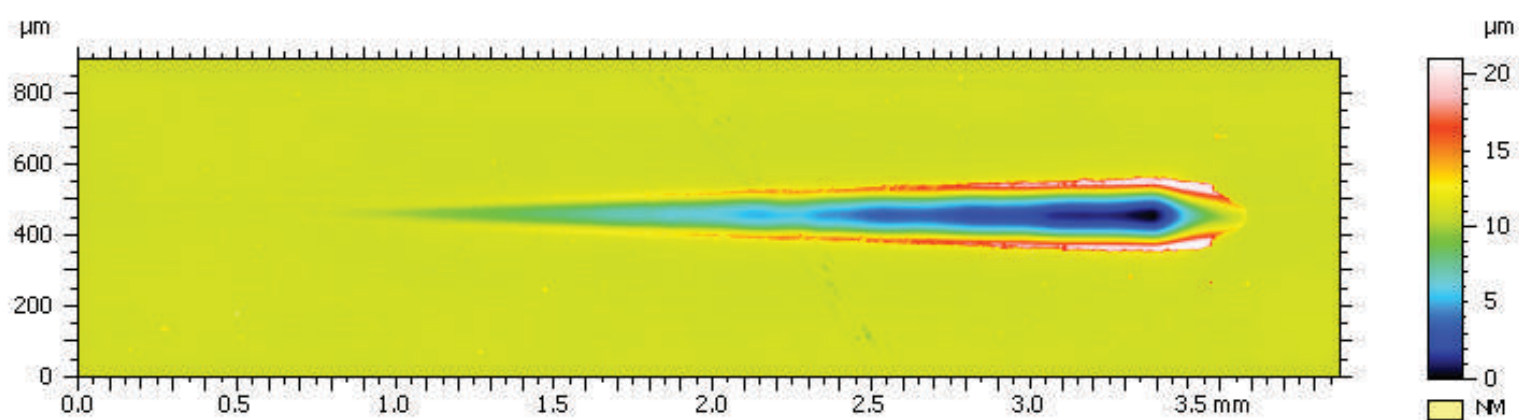
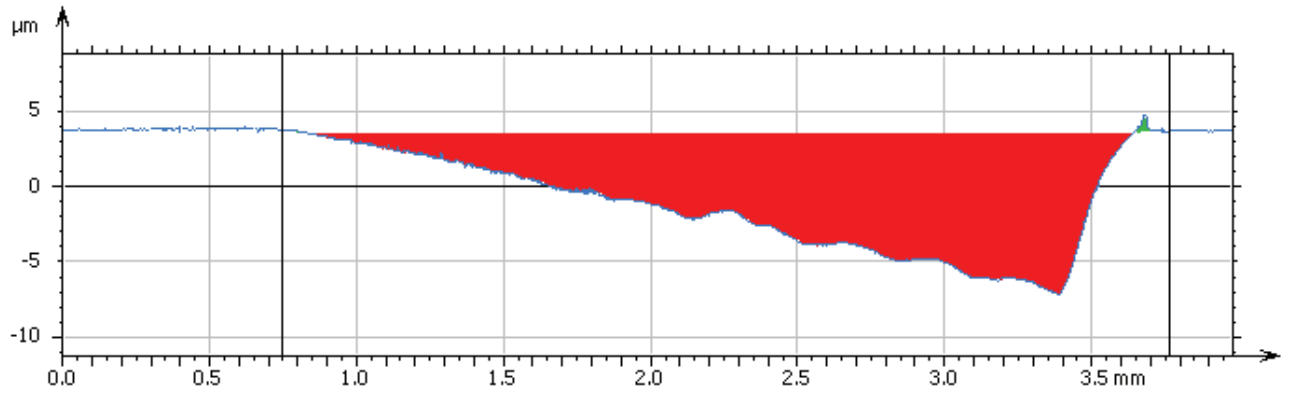
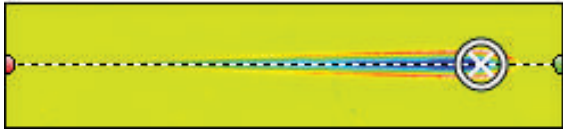
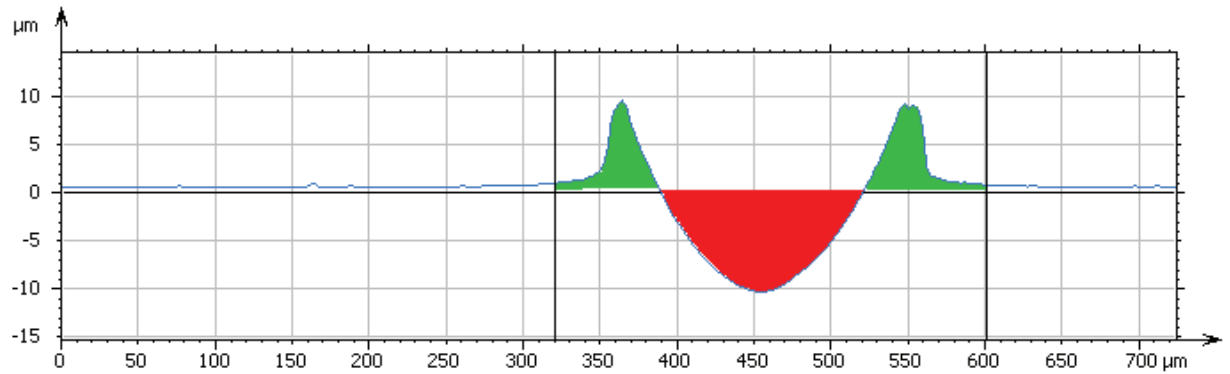
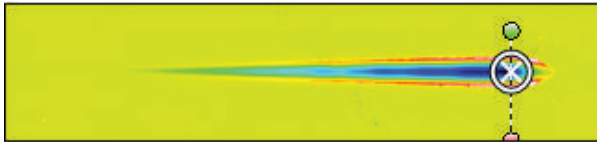


Figure 7: 3D Profile by chromatic technique (top view and 3D).





Parameters	Value	Unit
Maximum depth	10.96	μm
Maximum height	0.9699	μm



Parameters	Value	Unit
Maximum depth	11.00	μm
Maximum height	8.985	μm

Figure 8: Profiles taken perpendicular and along the scratch.

# 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 Figure 9. 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 Figure 10. A complete removal of DLC coating in the scratch track takes place at  $L_{c2}$  of 7.5 N.

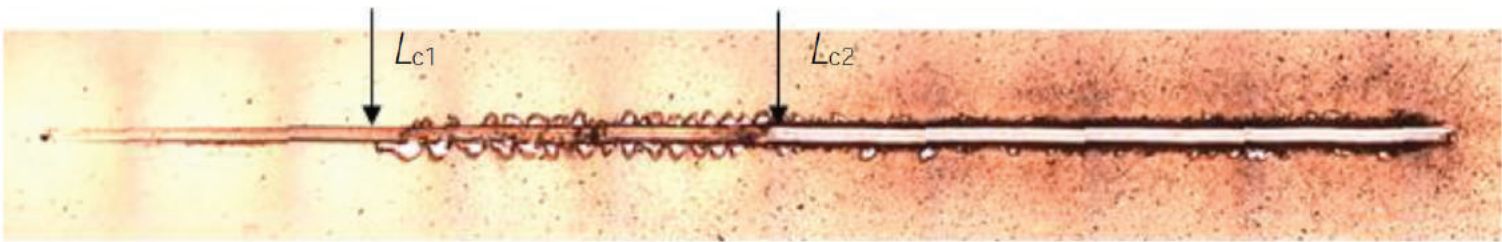


Figure 9: Micrograph of full scratch of DLC.

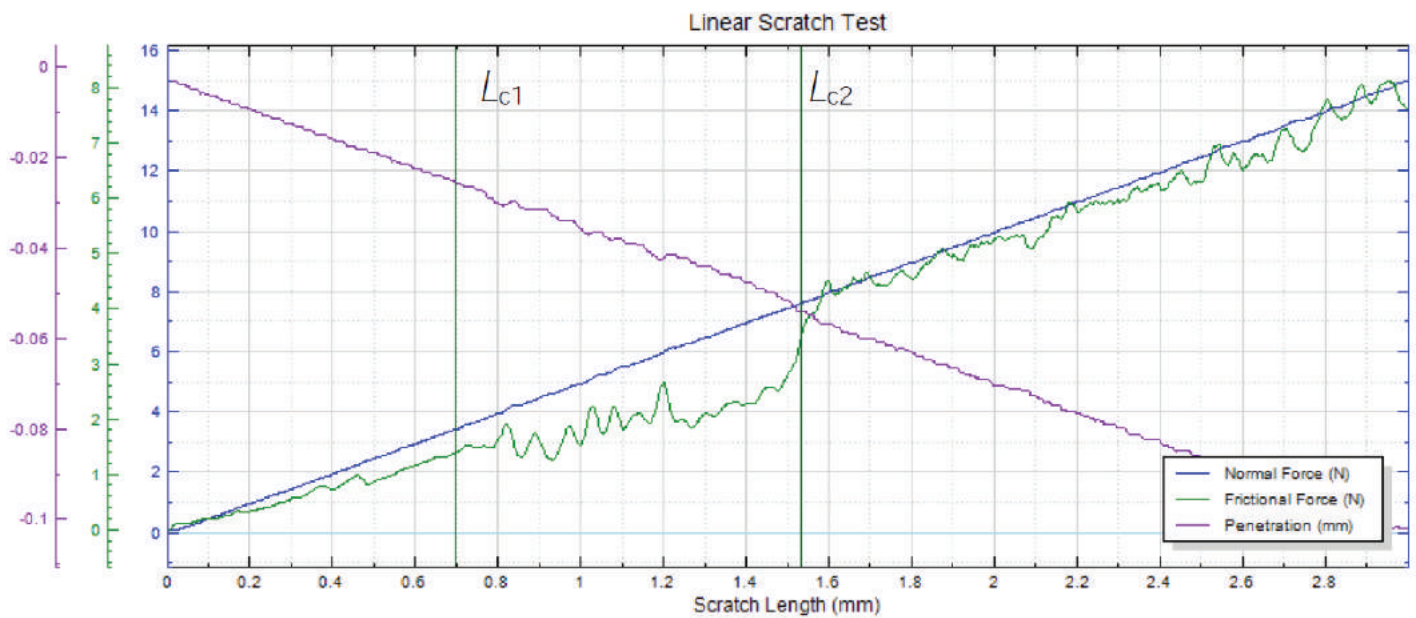
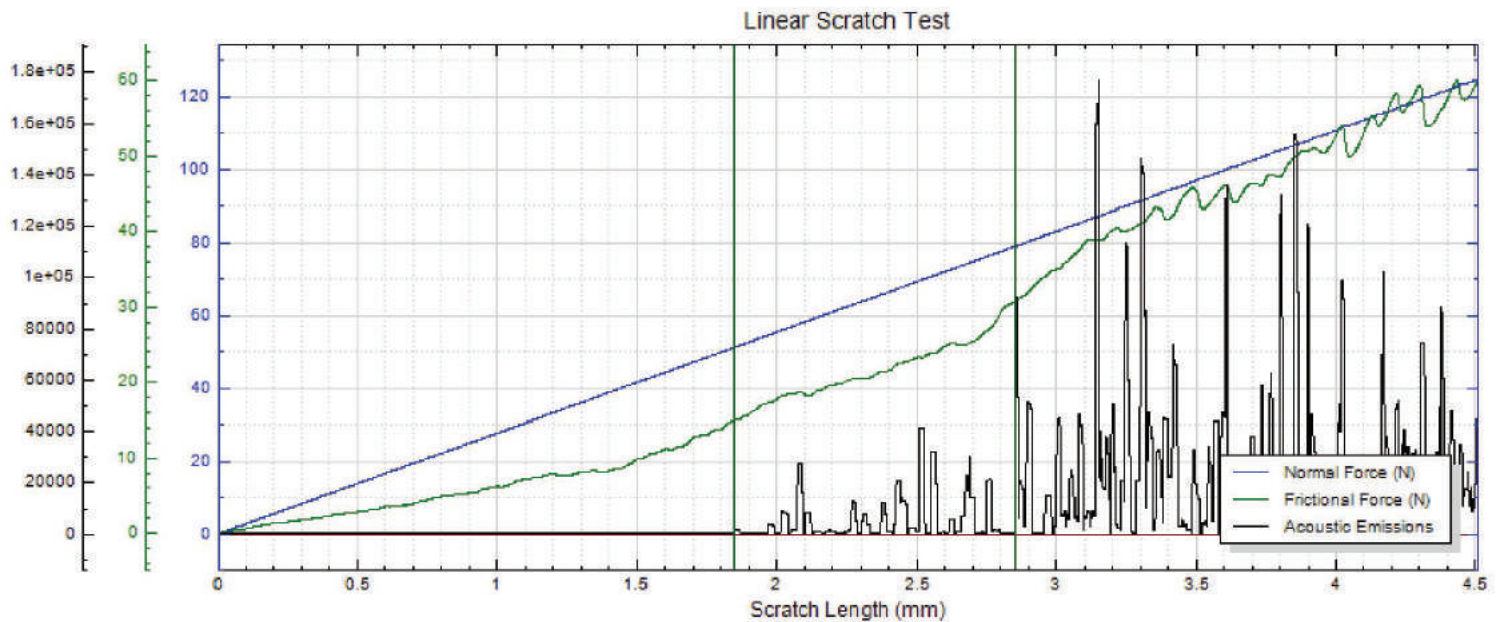


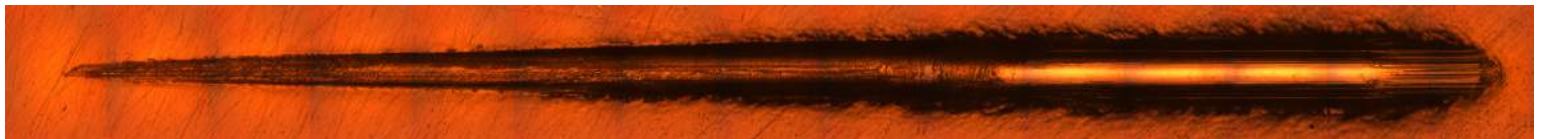
Figure 10: Normal force, frictional force and penetration as a function of scratch length of DLC.

## TiN Coating

The titanium nitride (TiN) coating showed signs of cracking based on the acoustic emissions data represented in Figure 11 at a critical load ( $L_{c1}$ ) of 51.1 N. Visual signs of cracking did not occur at this location but acoustic emissions allow the user to detect cracking when it is not easily visible under microscope inspection. The second critical load ( $L_{c2}$ ) location occurs at 79.1 N, visible signs of complete delamination occur on the scratch Figure 12 as well as increased spikes in the acoustic emissions after this point.



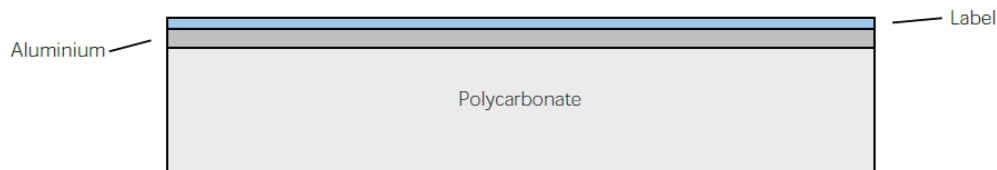
**Figure. 11: Normal force, frictional force and acoustic emissions as a function of scratch length.**



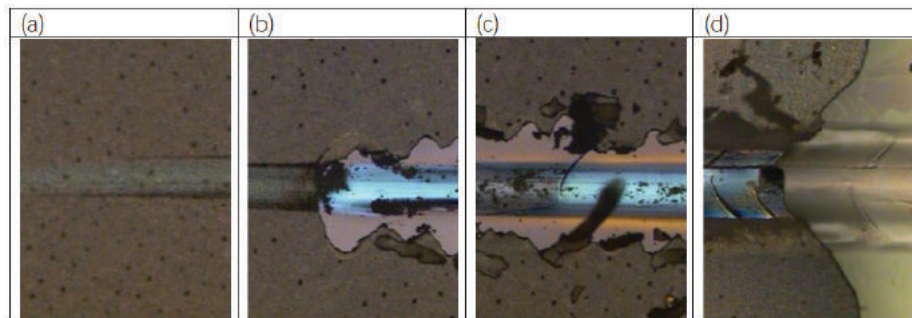
**Figure. 12: Full Image of full scratch of TiN Coating.**

## CD coating

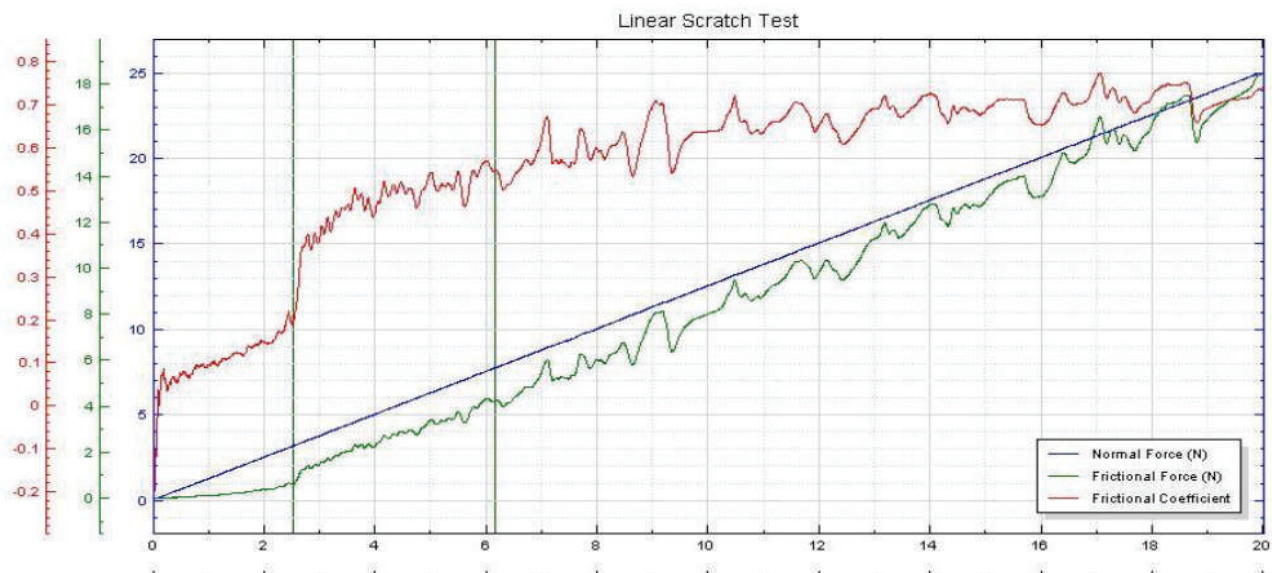
Scratch resistance is an important factor determining the service lifetime of CD/DVD. The CD is composed of a 1.2 mm disc of polycarbonate with a thin layer of aluminum. The aluminum is protected by a film of lacquer as illustrated in Figure 13. Figure 14 shows the evolution of coating behavior during 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 Figure 15.



**Figure 13: CD coating architecture.**



**Figure 14: Evolution of the coating during the scratch test**



**Figure 15: Friction coefficient, Normal Force and Frictional Force as a function of scratch length.**





## Conclusion

Nanovea Mechanical Tester is a superior tool for scratch and marring 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 the quality of their coatings.

The Nano & Micro 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, 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.



### Multi Module Platform

3 Testing Modes in 1 (Scratch/Indent/Wear)

Loading Ranges from 0.8uN to 400N

XYZ Motion with 0.20um Step Resolution

Fully Automated (Up to 100 indents in 15mins)

Integrated Imaging (AFM, Profilometer, Microscope)

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