# DLC Coating Failure
# Using Micro Scratch Testing


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## INTRO

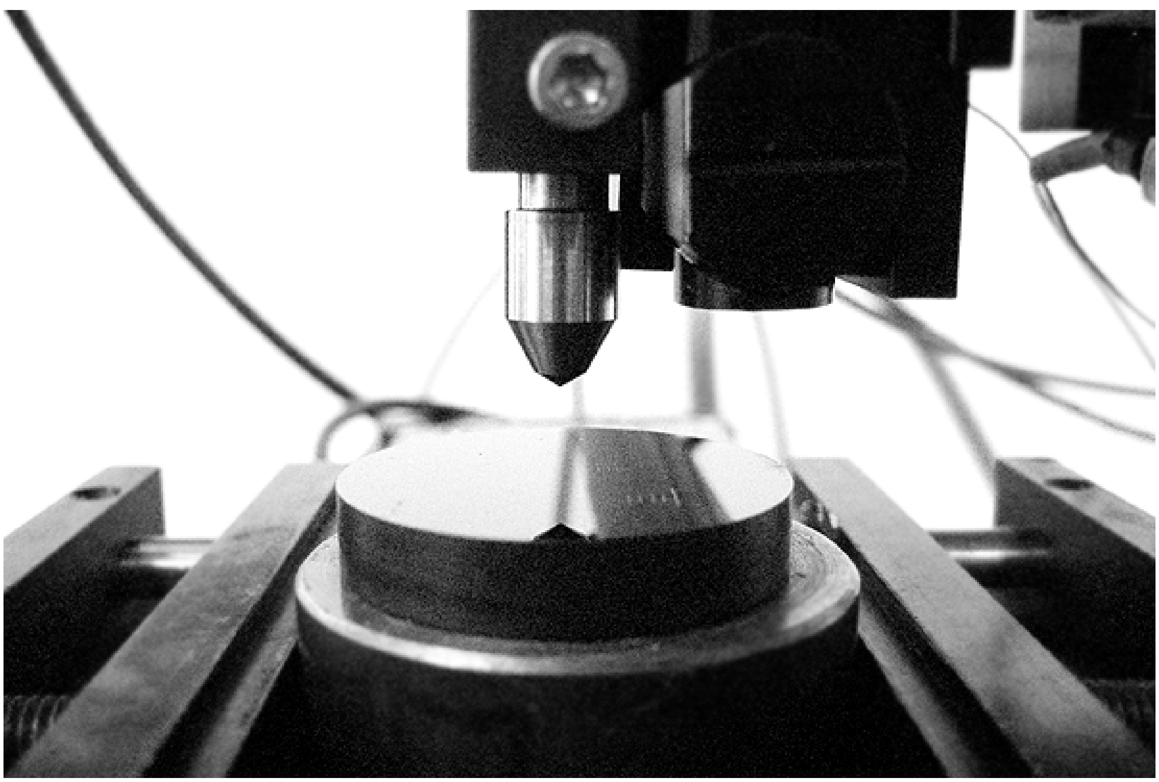
Diamond-like carbon (DLC) coatings are well known for their use in creating surfaces with diamond-like properties on nearly any material. The primary associated properties of interest are extreme hardness, wear resistance and low friction coefficient. These favorable tribological properties of DLC coatings are in high demand in industries such as Aerospace, Automotive and Tooling.

### IMPORTANCE OF MICRO SCRATCH TESTING FOR QUALITY CONTROL

A major concern for DLC coatings is to insure strong coating adhesion and or ability to withstand marring/cracking. The same internal stress that enhances the hardness of DLC coatings conversely makes it difficult to bond to the substrate being coated. It is for this reason that DLC coatings require quality assurance when establishing a reliable coating process. Although adhesion and or cracking failure may be inevitable over time, it is crucial that these failures are investigated, known and controlled. Using the Micro Scratch Test, precisely controlled loads can be used to investigate DLC coating for these very failures.

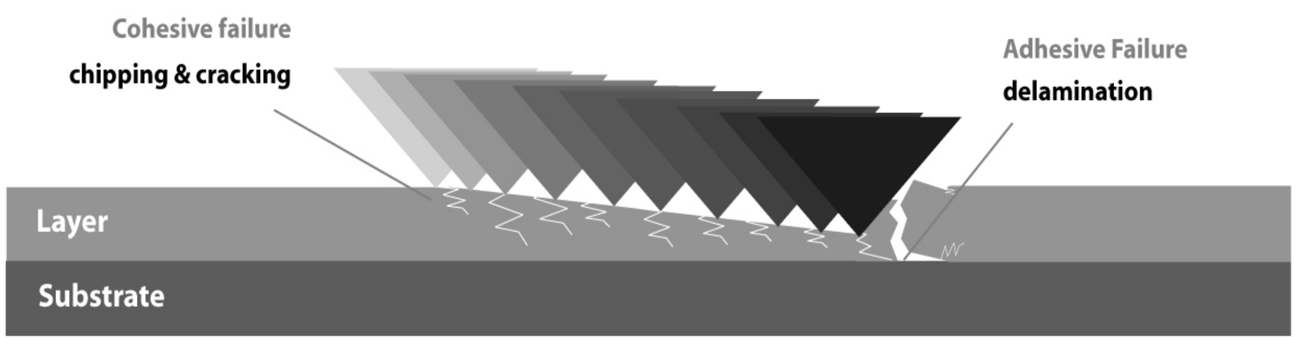
### MEASUREMENT OBJECTIVE

We must simulate the process of scratching in a controlled and monitored manner to observe sample behavior effects. In this application, the Nanovea Mechanical Tester, in its micro scratch mode, is used to measure the load required to cause the cracking and adhesion to three separately processed DLC coatings. A 90° Cone, 20µm diamond tipped stylus is used at a progressive load ranging from 0.01 mN to 15 N to scratch the DLC coating. The point where the coating fails by cracking is taken as the point of failure.



## MEASUREMENT PRINCIPLE

The scratch testing method is a very reproducible quantitative technique in which 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 20µm) 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 200µm for micro/macro scratch tests, and 1 to 20µm 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.




### 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 test specific parameters include: | The sample specific parameters include: |
|---|---|
| Loading rate<br>Scratching speed<br>Indenter tip radius<br>Indenter material | Friction coefficient between surface and indenter<br>Internal stresses in the material<br>For bulk materials<br>Material hardness and roughness<br>For coating-substrate systems<br>Substrate hardness and roughness<br>Coating hardness and roughness<br>Coating thickness |

### 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.

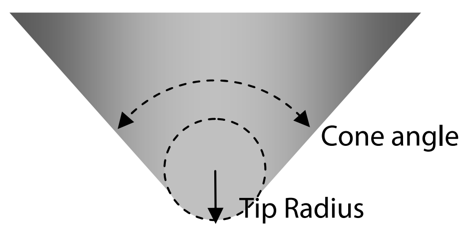
**Tangential (frictional) force recording**
This enables the force fluctuations along the scratch to be studied and correlated to the failures observed under the microscope. Typically, a failure in the sample will result in a change (a step, or a change in slope) in coefficient of friction. Frictional 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 AFM's can be useful to measure exact depth of scratch after the test.

### Test parameters-Scratch

| | |
|---|---|
| Load type | Progressive |
| Initial Load (N) | 0.01 mN |
| Final Load (N) | 15 N |
| Loading rate ((N/min) | 30 N/min |
| Scratch Length (mm) | 3mm |
| Indenter geometry | 90° Cone |
| Indenter tip radius | 20µm |
| Indenter material (tip) | Diamond |



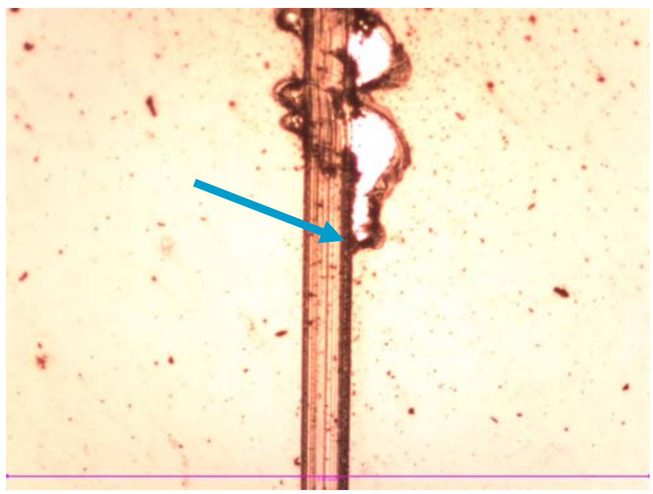
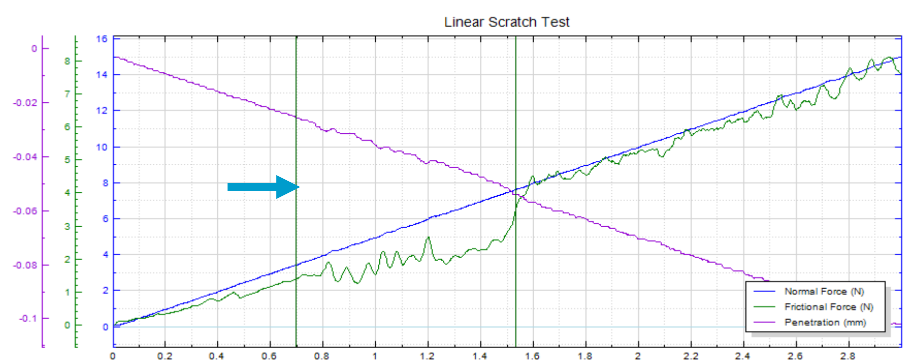

## RESULTS

Summary table of main numerical results

| Sample | Chipping (N) | Complete Delamination (N) |
|---|---|---|
| A | 3.52 ± 0.10 | 7.59 ± 0.13 |
| B | 4.60 ± 0.09 | 5.81 ± 0.26 |
| C | 3.51 ± 0.33 | 10.15 ± 0.05 |

## DETAILED RESULTS – Sample A

| Sample A | Chipping (N) | Complete Delamination (N) |
|---|---|---|
| 1 | 3.41 | 7.63 |
| 2 | 3.57 | 7.44 |
| 3 | 3.59 | 7.70 |
| Average | 3.52 | 7.59 |
| Standard Deviation | 0.10 | 0.13 |

| Critical failure | Micrograph of failure |
|---|---|
| **Chipping**<br>This is the first evidence at which damage is done to the coating. Here we see the coating being chipped on the right side of the scratch. | <br>**Figure 1: Chipping – Sample A**<br>**100x magnification (image width 0.474 mm)**<br><br><br>**Figure 2: Friction graph showing line of chipping – Sample A** |

**Complete Delamination**
This is the point where the coating is being completely removed to the substrate.

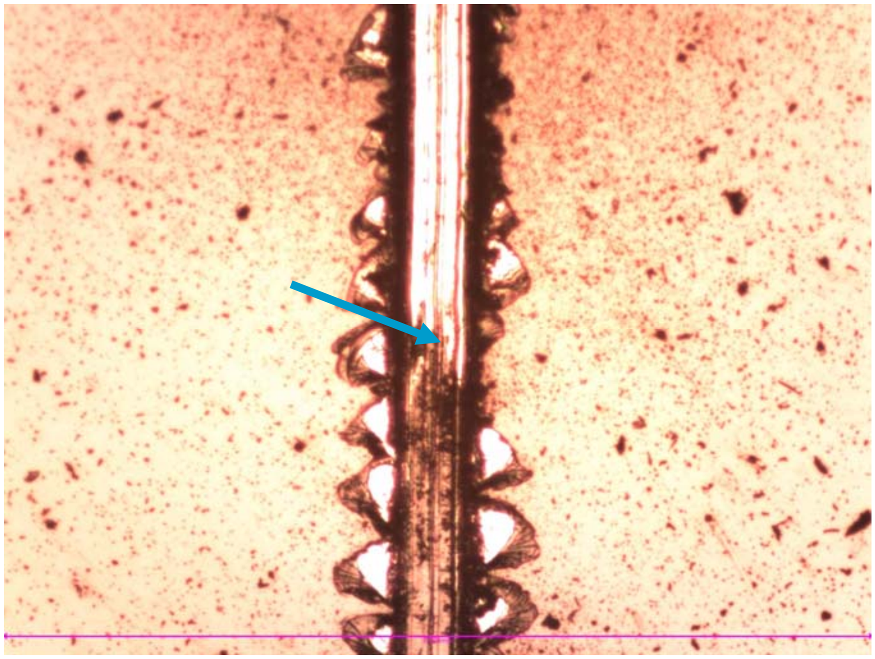


**Figure 3: Complete delamination- Sample A 100x magnification (image width 0.474mm)**

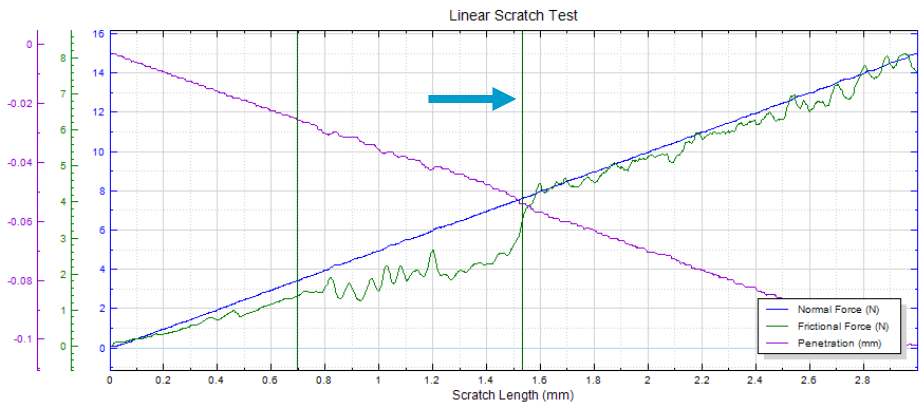



**Figure 4: Friction graph showing line of complete delamination – Sample A**

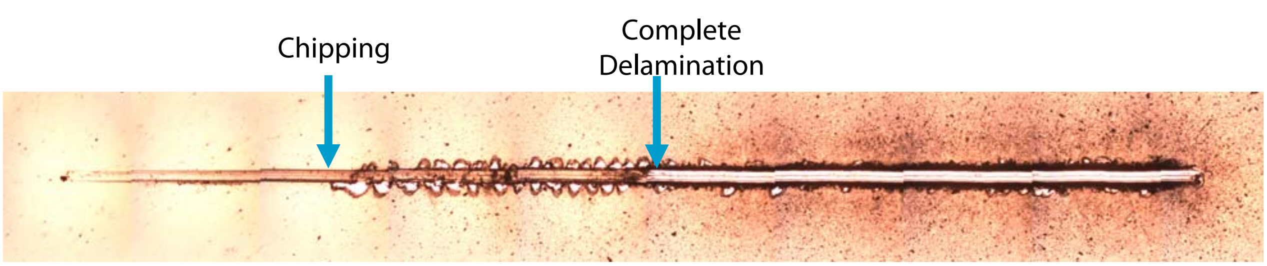
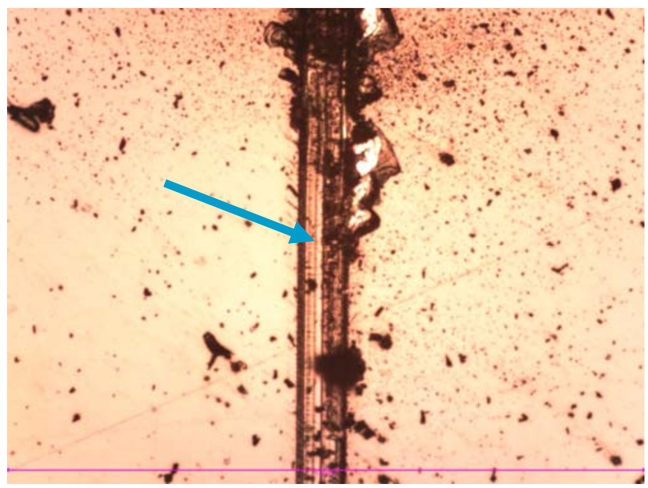
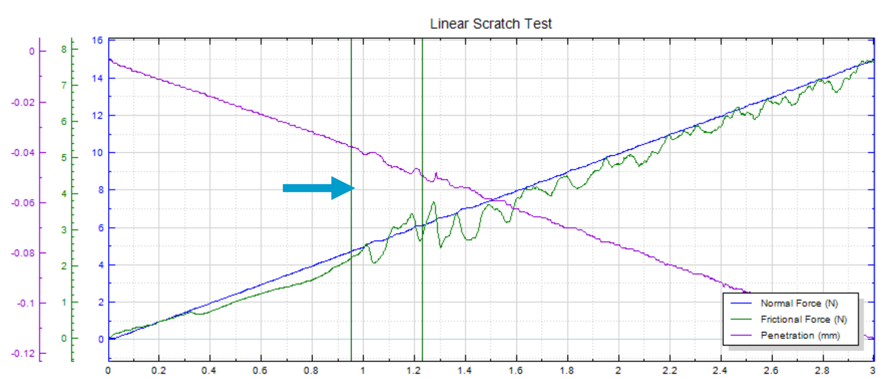



**Figure 5: Micrograph of full scratch – Sample A**

## DETAILED RESULTS – Sample B

| Sample B | Chipping (N) | Complete Delamination (N) |
|---|---|---|
| 1 | 4.70 | 6.11 |
| 2 | 4.53 | 5.68 |
| 3 | 4.57 | 5.65 |
| Average | 4.60 | 5.81 |
| Standard Deviation | 0.09 | 0.26 |

| Critical failure | Micrograph of failure |
|---|---|
| **Chipping**<br>This is the first evidence at which damage is done to the coating. Here we see the coating being chipped on the right side of the scratch. | <br>**Figure 6: Chipping – Sample B**<br>**100x magnification (image width 0.474 mm)**<br><br><br>**Figure 7: Friction graph showing line of chipping – Sample B** |

**Complete Delamination**
This is the point where the coating is being completely removed to the substrate.

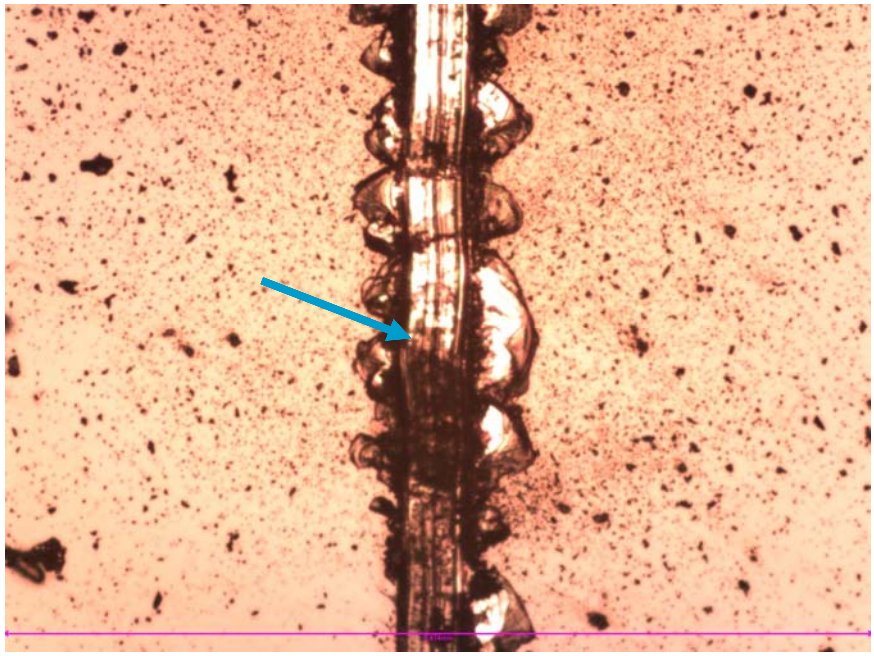


**Figure 8: Complete delamination- Sample B 100x magnification (image width 0.474mm)**

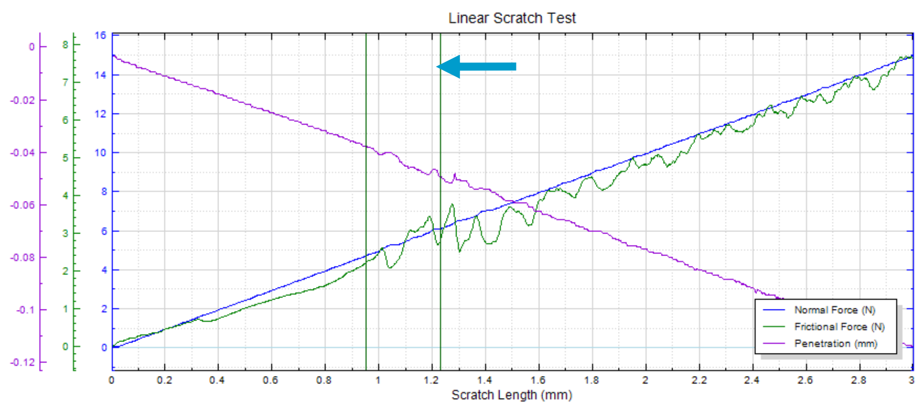



**Figure 9: Friction graph showing line of complete delamination – Sample B**


**Figure 10: Micrograph of full scratch – Sample B**

## DETAILED RESULTS – Sample C

| Sample C | Chipping (N) | Complete Delamination (N) |
|---|---|---|
| 1 | 3.14 | 10.18 |
| 2 | 3.79 | 10.17 |
| 3 | 3.61 | 10.10 |
| Average | 3.51 | 10.15 |
| Standard Deviation | 0.33 | 0.05 |

| Critical failure | Micrograph of failure |
|---|---|
| **Chipping**<br>This is the first evidence at which damage is done to the coating. Here we see the coating being chipped on the right side of the scratch. | See Figures 11 and 12 below |

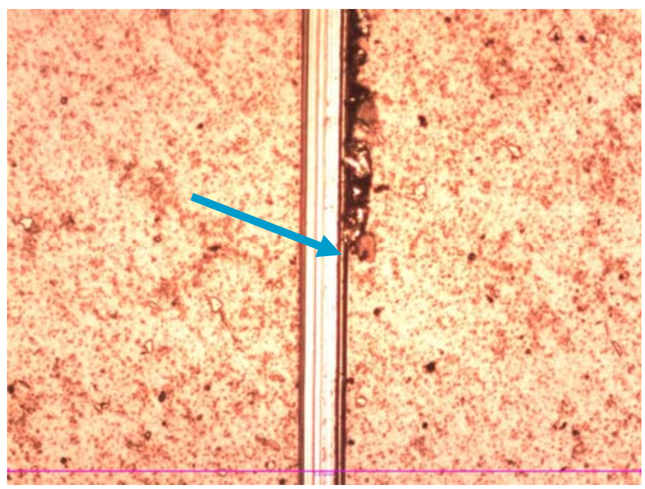


**Figure 11: Chipping – Sample C**
**100x magnification (image width 0.474 mm)**

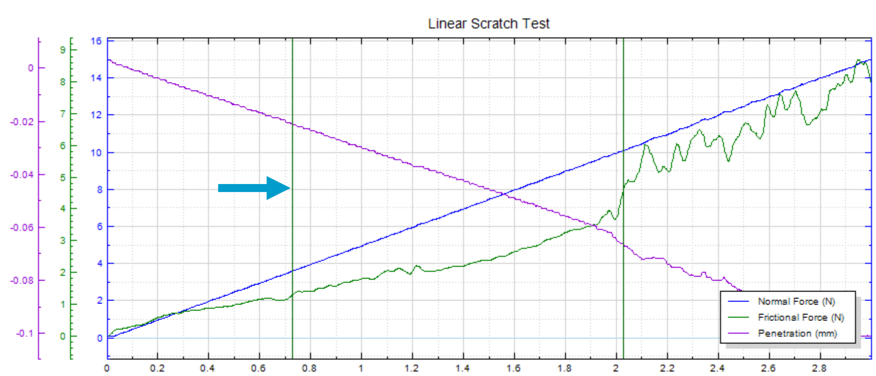



**Figure 12: Friction graph showing line of chipping – Sample C**

**Complete Delamination**
This is the point where the coating is being completely removed to the substrate.

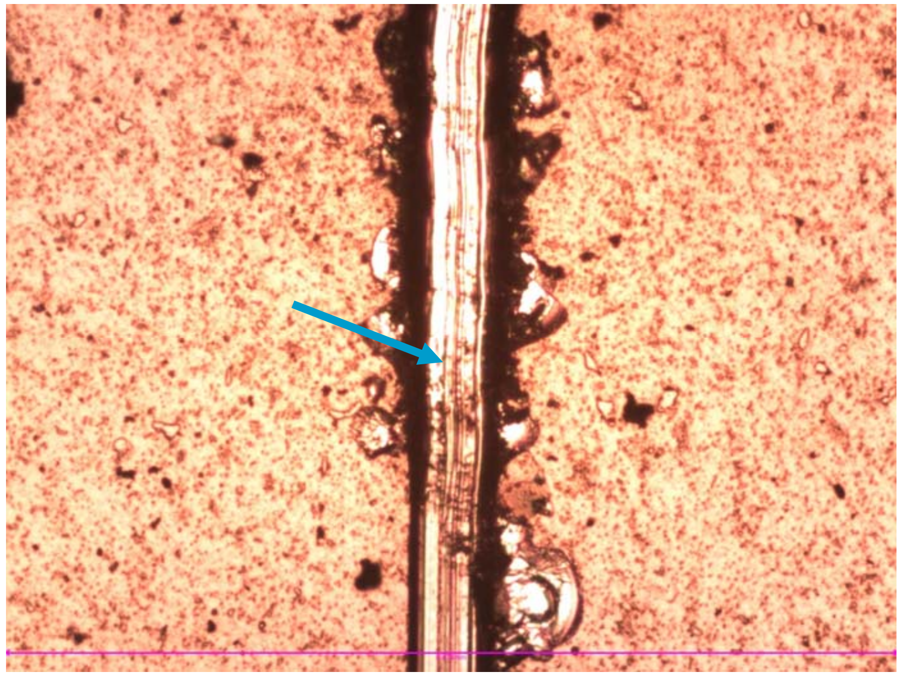


**Figure 13: Complete delamination- Sample C 100x magnification (image width 0.474mm)**

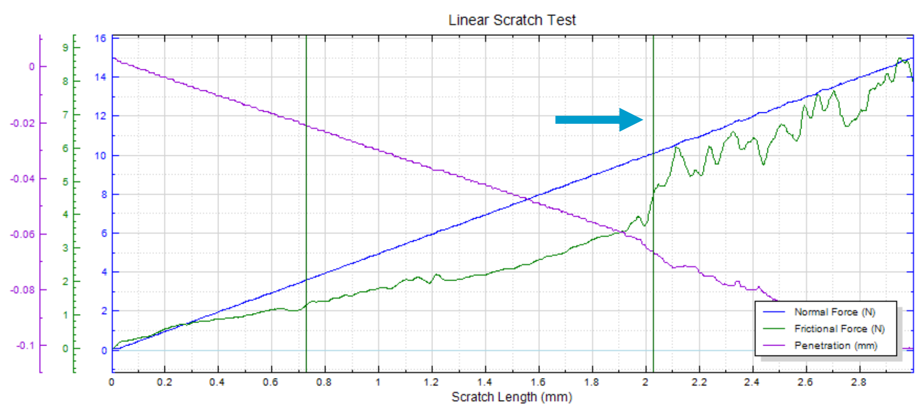



**Figure 14: Friction graph showing line of complete delamination – Sample C**


**Figure 15: Micrograph of full scratch – Sample C**

## CONCLUSION

Using the Nanovea Mechanical Tester, in micro scratch mode, the cohesive & adhesive failure of the three DLC samples was comparatively evaluated. The samples exhibit a large variation in adhesion strength compared to cracking properties. Micro scratch testing has the ability to quantify adhesive and cohesive failures of DLC coatings with high repeatability. There are many indenter tips that can be used to simulate various levels of scratches. Additionally, the Nanovea Mechanical Tester could have also been used to measure hardness, elastic modulus, wear, friction coefficient, fracture toughness, roughness and many others. It is a complete and powerful tool for DLC coating research and control.
To learn more about: Nanovea Mechanical Tester. or Lab Services