

**NANO SCRATCH/MAR TESTING  
OF PAINT ON METAL SUBSTRATE**



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## INTRO

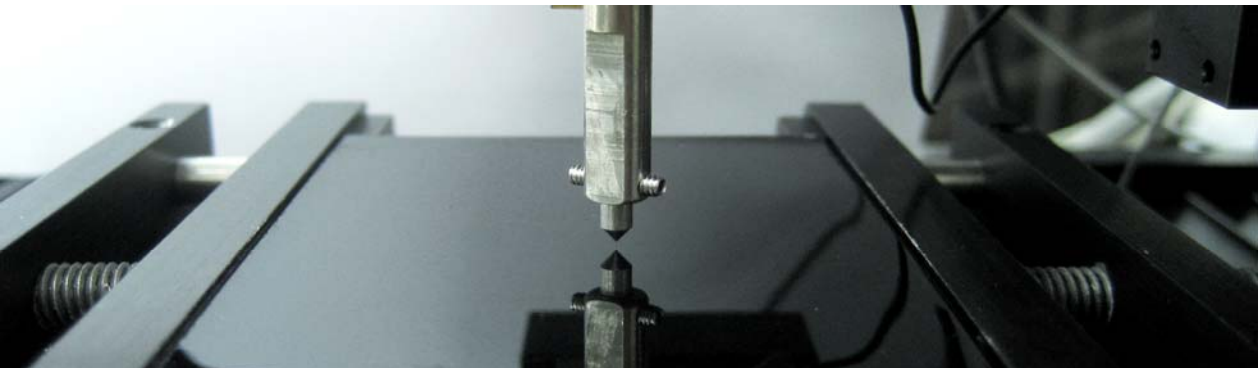
Paint with or without hard coat is one of the most commonly used coatings. We see it on cars, on walls, on appliances and virtually anything that needs some protective coatings or simply for aesthetic purposes. The paints that are meant for the protection of the underlying substrate often have chemicals that prevent the paint from catching on fire or simply that prevent it from losing its color or cracking. Often the paint used for aesthetic purposes comes in various colors, but may not be necessarily meant for the protection of its substrate or for a long lifetime. Nevertheless, all paint suffers some weathering over time. Weathering on paint can often change the properties from what the makers intended it to have. It can chip quicker, peel off with heat, lose color or crack. The different property changes of paint over time is why makers offer such a wide selection. Paints are tailored to meet different requirements for individual clients.

### IMPORTANCE OF NANO SCRATCH TESTING FOR QUALITY CONTROL

A major concern for paint makers is the ability for their product to withstand cracking. Once paint begins to crack, it fails to protect the substrate that it was applied on; therefore, failing to satisfy their client. For example, if a branch happens to stroke the side of a car and immediately after the paint begins to chip off the makers of the paint would lose business due to their poor quality of paint. The quality of the paint is very important because if the metal under the paint becomes exposed it may begin to rust or corrode due to its new exposure. Reasons like this apply to several other spectrums such as household and office supplies and electronics, toys, research tools and more. Although the paint may be resistant to cracking when they first apply it to metal coatings, the properties may change over time when some weathering has occurred on the sample. This is why it's very important to have the paint samples tested at their weathered stage. Although cracking under a high load of stress may be inevitable, the maker must predict how weakening the changes may be over time and how deep the affecting scratch must be in order to provide their consumers with the best possible products.

### 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 nano scratch testing mode is used to measure the load required to cause failure to an approximately 7 year old 30-50 $\mu$ m thick paint sample on a metal substrate. A 2  $\mu$ m diamond tipped stylus is used at a progressive load ranging from 0.015 mN to 20.00 mN to scratch the coating. We performed a pre and post scan of the paint with 0.2 mN load in order to determine the value for the true depth of the scratch. The true depth analyzes the plastic and elastic deformation of the sample during testing; whereas, the post-scan only analyzes the plastic deformation of the scratch. The point where the coating fails by cracking is taken as the point of failure. We used the ASTM-D7187 as a guide to determine our testing parameters. We can conclude that having used a weathered sample; therefore, testing a paint sample at its weaker stage, presented us with lower points of failure. Five tests were done on this sample in order to determine the exact failure critical loads.



## 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 20 $\mu\text{m}$ ) 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 $\mu\text{m}$  for micro/macro scratch tests, and 1 to 20 $\mu\text{m}$  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.

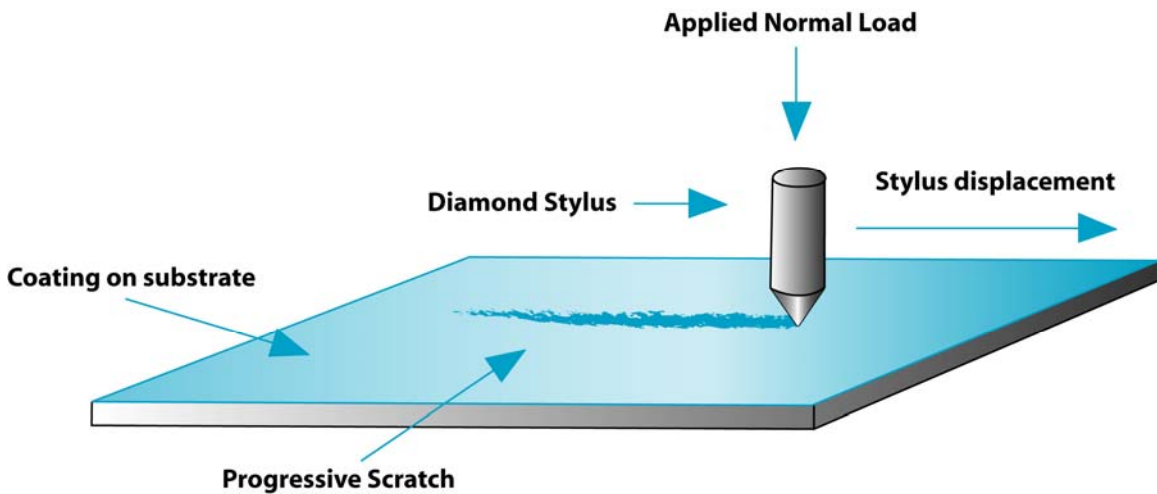


Figure 1 : Principle of scratch testing

## 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 test specific parameters include:	The sample specific parameters include:
<ul style="list-style-type: none"> <li>• Loading rate</li> <li>• Scratching speed</li> <li>• Indenter tip radius</li> <li>• Indenter material</li> </ul>	<ul style="list-style-type: none"> <li>• Friction coefficient between surface and indenter</li> <li>• Internal stresses in the material For bulk materials</li> <li>• Material hardness and roughness For coating-substrate systems</li> <li>• Substrate hardness and roughness</li> <li>• Coating hardness and roughness</li> <li>• Coating thickness</li> </ul>

## 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

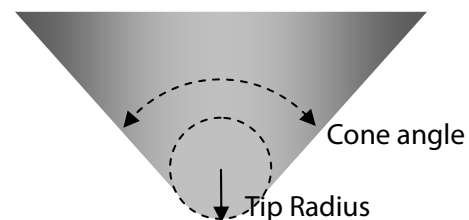
Detection of elastic waves generated as a result of the formation and propagation of microcracks. The AE sensor is insensitive to mechanical vibration frequencies of the instrument. This method of critical load determination is mostly adequate for hard coatings that crack with more energy.

### 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 following ASTM D 7027

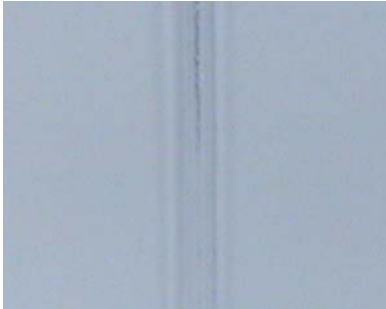

Load type	Progressive
Initial Load	0.015 mN
Final Load	20.00 mN
Loading rate	20.00 mN/min
Scratch Length	1.6 mm
Scratching speed, dx/dt	1.601 mm/min
Pre-Scan Load	0.200 mN
Post-Scan Load	0.200 mN
Indenter geometry	90° conical
Indenter tip radius	2 μm
Indenter material (tip)	Diamond



## Results

This section presents the data collected on the failures during the scratch test. The first section describes the failures observed in the scratch and defines the critical loads that were reported. The next part contains a summary table of the critical loads for all samples, and a graphical representation. The last part presents detailed results for each sample: the critical loads for each scratch, micrographs of each failure, and the graph of the test.

### Failures observed and definition of critical loads

Critical failure	Micrograph of failure
<p><b>Initial Damage</b> This is the first point at which the damage is observed along the scratch track.</p>	
<p><b>Complete Damage</b> At this point, the damage is more significant where the paint is chipping and cracking along the scratch track.</p>	

## Detailed results – Paint on Metal

\* Failure values taken at point of substrate cracking.

Critical loads – Paint on Metal		
Scratch	Initial Damage [ mN ]	Complete Damage [ $\mu\text{m}$ ]
1	4.153	4.932
2	3.895	4.838
3	3.917	4.930
Average	<b>3.988</b>	<b>4.900</b>
Std dev	<b>0.143</b>	<b>0.054</b>



Figure 2 : Micrograph of Full Scratch – Paint on Metal  
1000x magnification

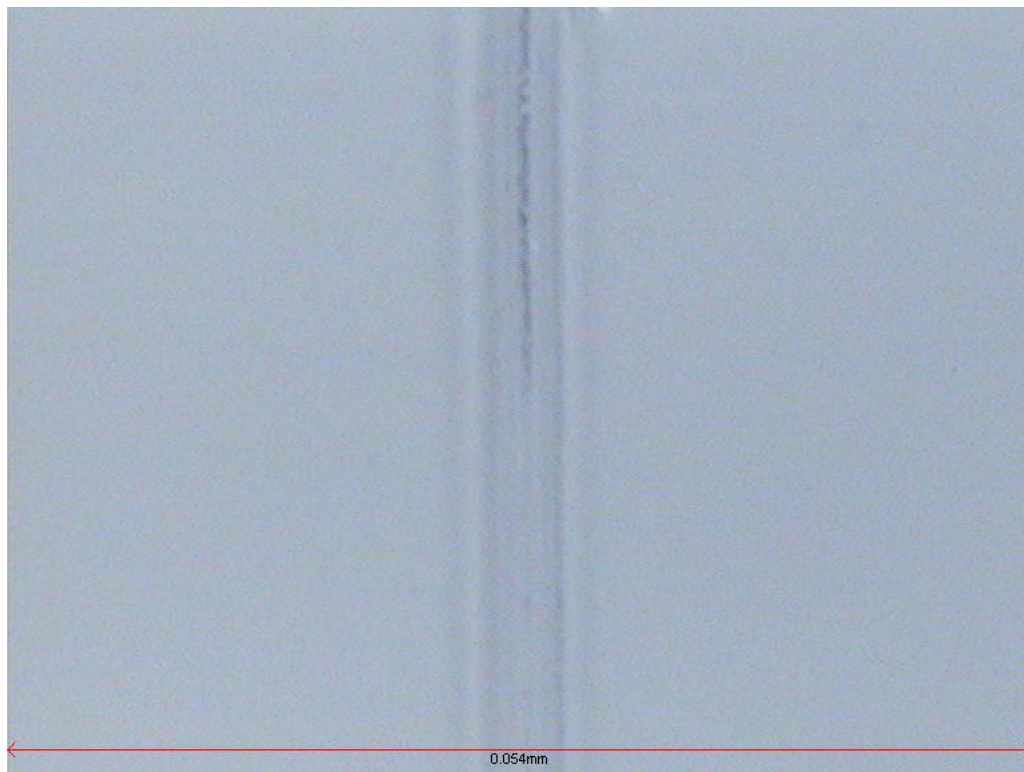
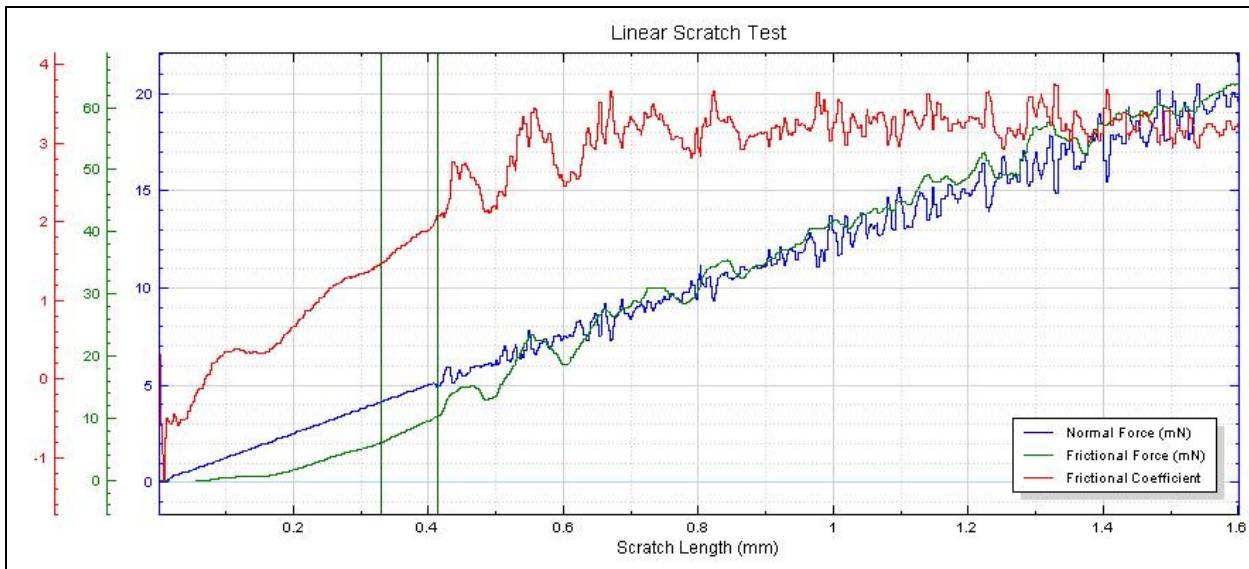


Figure 3 : Micrograph of Initial Damage – Paint on Metal  
1000x magnification (image width 0.054mm)

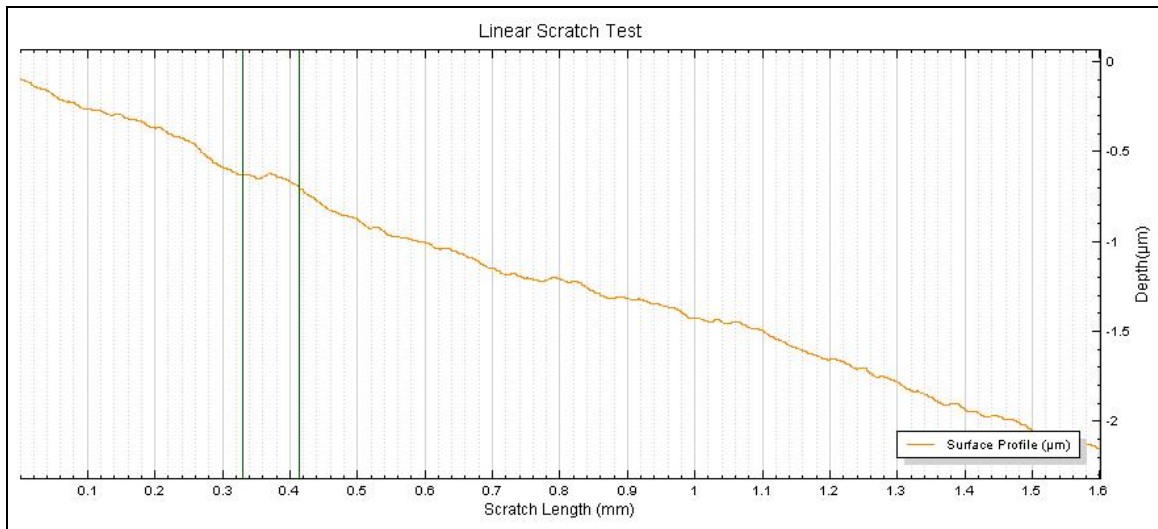




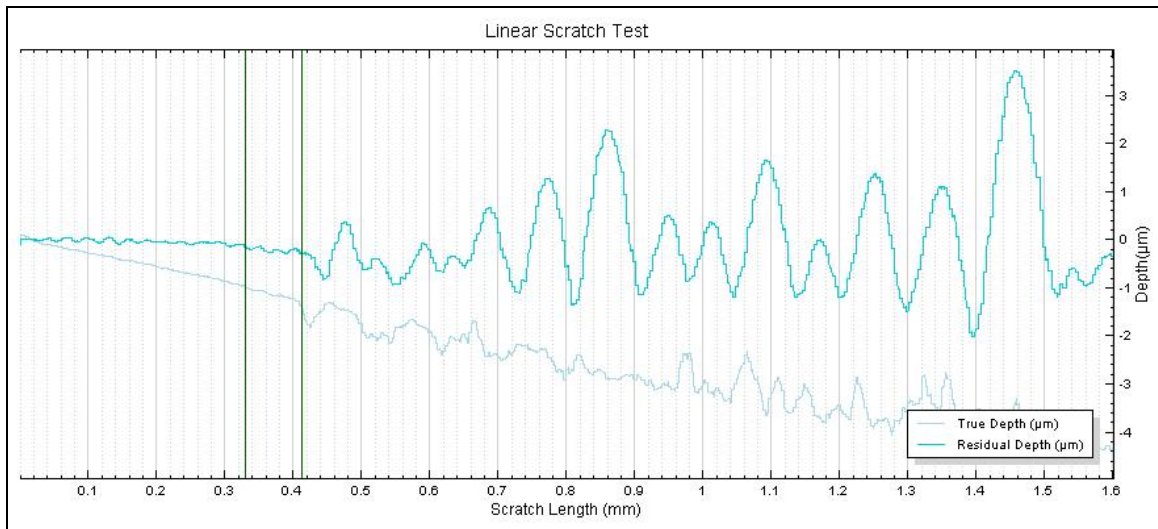
**Figure 4 : Micrograph of Complete Damage – Paint on Metal  
1000x magnification (image width 0.054mm)**



**Figure 5 : Friction Force and Coefficient of Friction Graph – Paint on Metal**



**Figure 6 : Surface Profile Graph – Paint on Metal**



**Figure 7 : True Depth and Residual Depth Graph – Paint on Metal**

## Conclusion

Nanovea Mechanical Tester, during Nano Scratch Tester Mode, allows simulation of many real-life failures of paint coatings and hard coats. By applying increasing loads in a controlled and closely monitored fashion, the instrument allows to identify at what load failures occur. This can then be used as a way to determine quantitative values for scratch resistance. The coating tested, with no weathering, is known to have a first crack at about 22mN. With values closer to 5mN, it is clear that the 7 year lap has degraded the paint. Compensating for the original profile allows to obtain corrected depth during the scratch and also to measure the residual depth after the scratch. This gives extra information on the plastic versus elastic behavior of the coating under increasing load. Both cracking and the information on deformation can be of great use for improving the hard coat. The very small standard deviations also show the reproducibility of the technique of the instrument which can help manufacturers improved the quality of their hard coat / paint and study weathering effects.