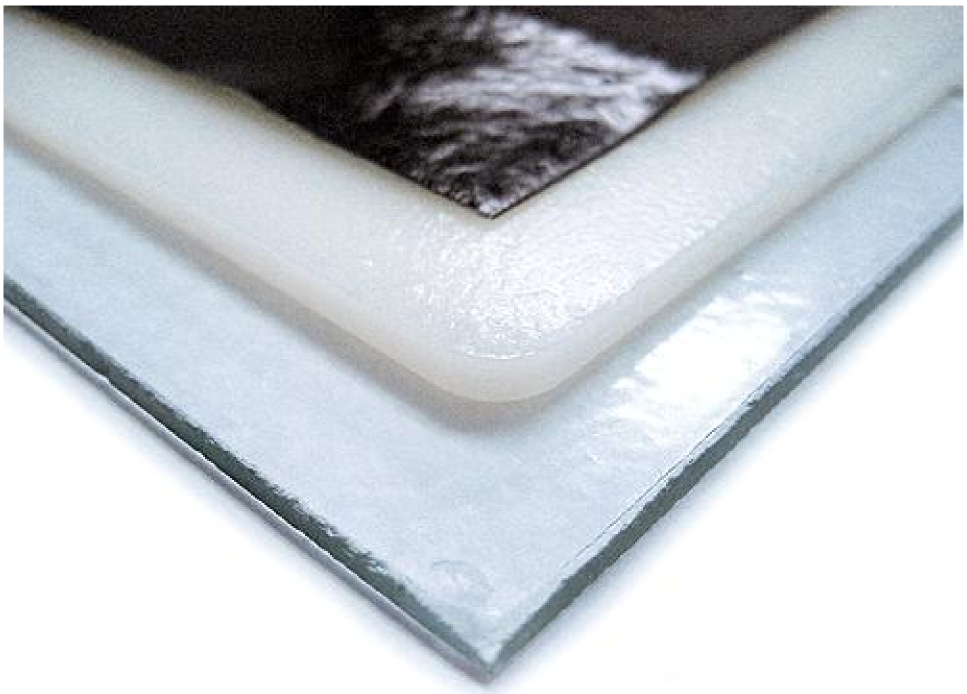
Nano Scratch Testing
Nano Coating Samples



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INTRO

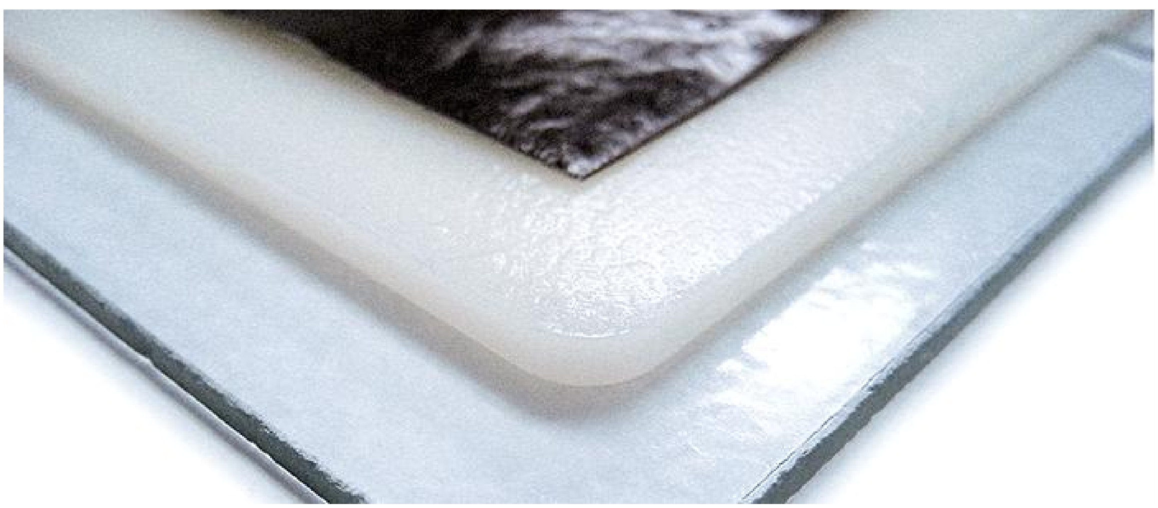
Coatings are applied to the surface substrate on endless applications, primarily to improve surface properties of the substrate, including appearance, adhesion, corrosion, wear resistance, and scratch/mar resistance. A “Nano” coating is a controlled process at the nano level to significantly enhance the ability of a coating to improve surface properties. Nano coatings can be applied with paint, thermal spray, vacuum technology and electroplating each with its own advantages and disadvantages. Ultimately, the success and quality of nano coatings, on a given substrate, must be reliable known and controlled.

IMPORTANCE OF NANO SCRATCH TESTING FOR QUALITY CONTROL

A major concern for the manufactures of nano coatings will be to insure a highly significant scratch/mar resistance level. Nano scratch testing is an ideal tool to measure scratch/mar resistance of the nano coating on each individual substrate. It is important for the manufacture to understand and monitor at what level marring can occur. Marring damage not only affects visual appearance but can lead to full adhesion failure as environmental conditions access through cracking.

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 a spray nano coating on a TPO polymer, glass, and aluminum coil substrate. A 10µm diamond tipped stylus is used at a progressive load ranging from 0.100 mN to 200.00 mN to scratch the coating. The point where the coating fails by cracking is taken as the point of failure. Three tests were done on each 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 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.

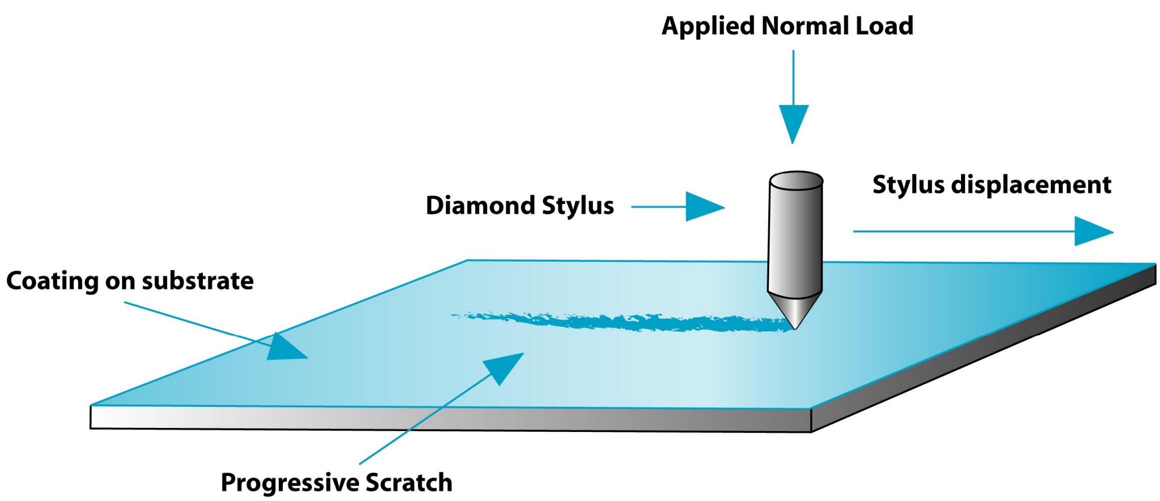


Figure 1 : Principle of scratch testing

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:
• Loading rate • Scratching speed • Indenter tip radius • Indenter material	• Friction coefficient between surface and indenter • Internal stresses in the material For bulk materials • Material hardness and roughness For coating-substrate systems • Substrate hardness and roughness • Coating hardness and roughness • 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.

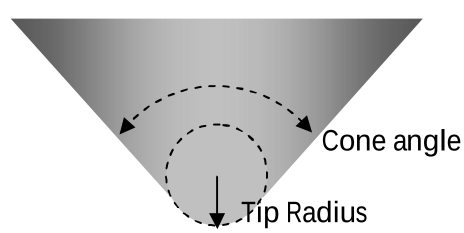
Tangential (frictional) force recording
This enables the force fluctuations along the scratch to be studied and correlated to the failures observed under the microscope. Typically, a failure in the sample will result in a change (a step, or a change in slope) in coefficient of friction. Frictional responses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

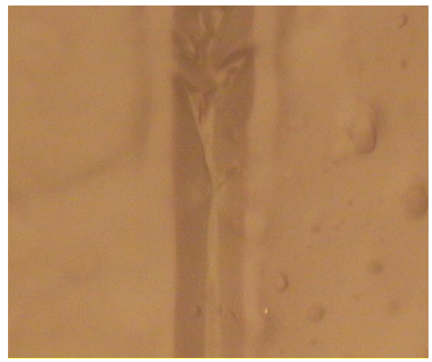
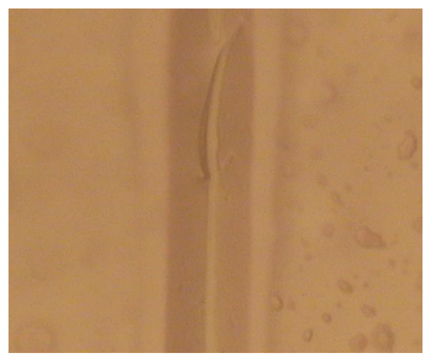
Load type	Progressive
Initial Load	0.100 mN
Final Load	200.00 mN
Loading rate	200.00 mN/min
Scratch Length	3.00 mm
Scratching speed, dx/dt	3.001 mm/min
Indenter geometry	90° conical
Indenter tip radius	10 µm
Indenter material (tip)	Diamond



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 Loads	Micrograph of failure
Initial Cracking This is the point at which first cracks are visible.	
Continuous Cracking This is the point at which cracks occur more frequently.	

Detailed results – Glass, Plastic, Aluminum

Critical loads		
Sample	**Initial Cracking [mN]**	**Continuous Cracking [mN]**
Glass	42.31	67.85
Plastic	58.57	60.72
Aluminum	29.07	48.71

GLASS

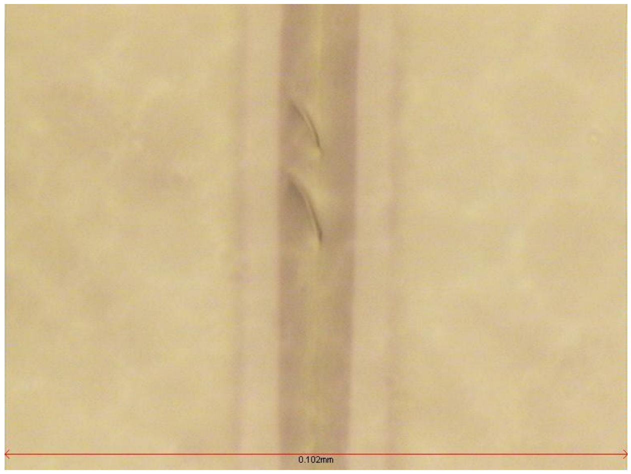


Figure 2 : Micrograph of the Initial Cracking – Glass 500x magnification (image width 0.102mm)

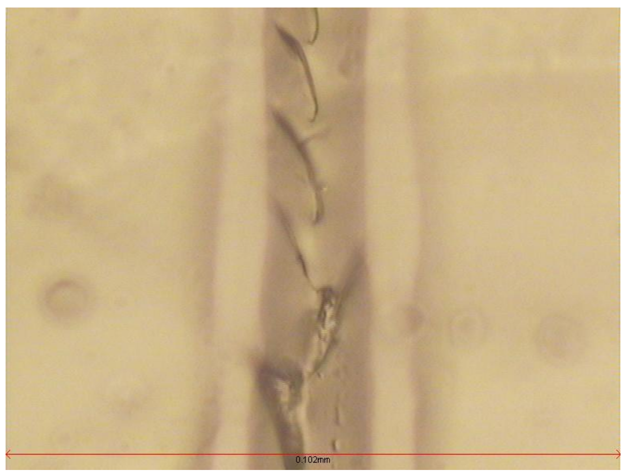


Figure 3 : Micrograph of Continuous Cracking – Glass 500x magnification (image width 0.102mm)

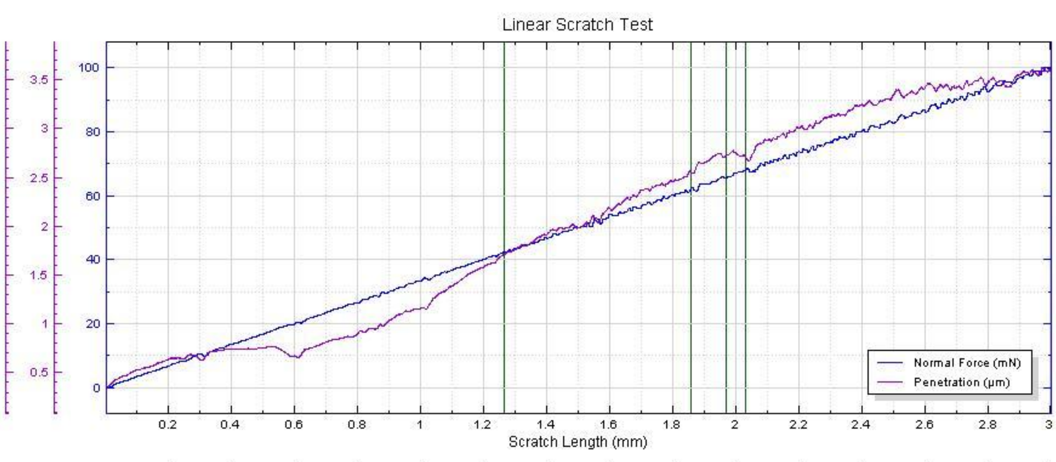


Figure 4 : Loading Curve – Glass

PLASTIC

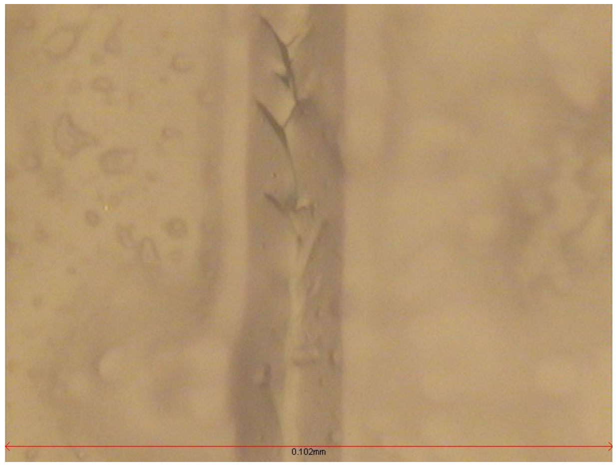


Figure 5 : Micrograph of the Initial Cracking – Plastic
500x magnification (image width 0.102mm)

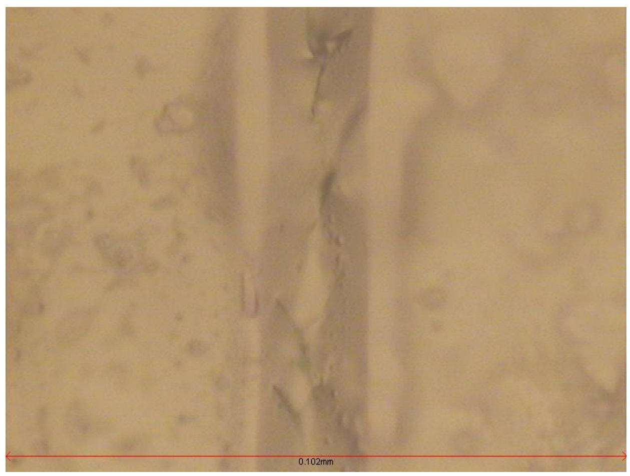


Figure 6 : Micrograph of Continuous Cracking – Plastic
500x magnification (image width 0.102mm)

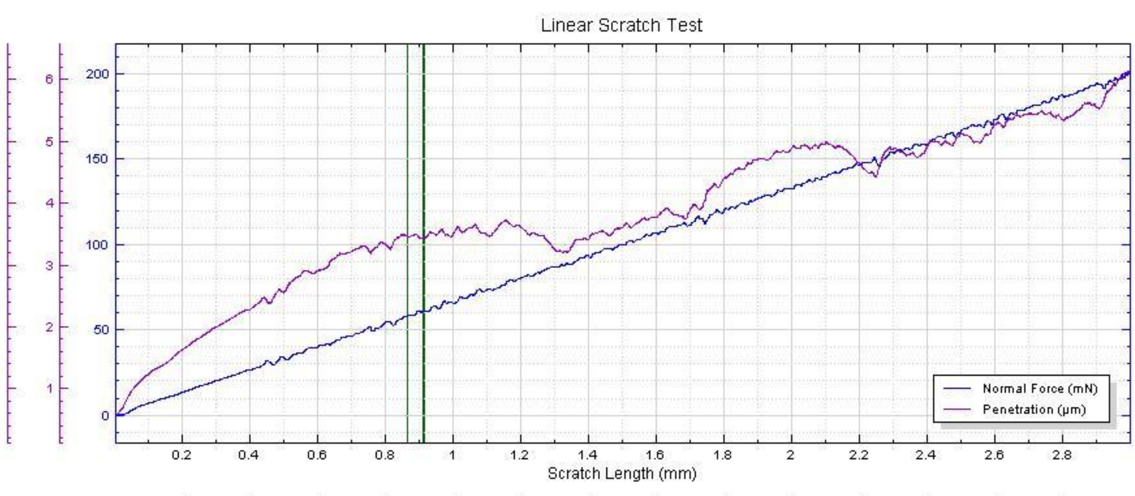


Figure 7 : Loading Curve – Plastic

ALUMINUM

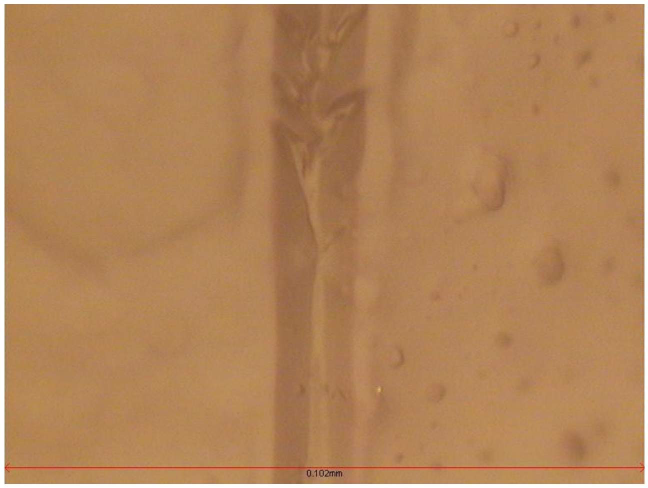


Figure 8 : Micrograph of the Initial Cracking – Aluminium 500x magnification (image width 0.102mm)

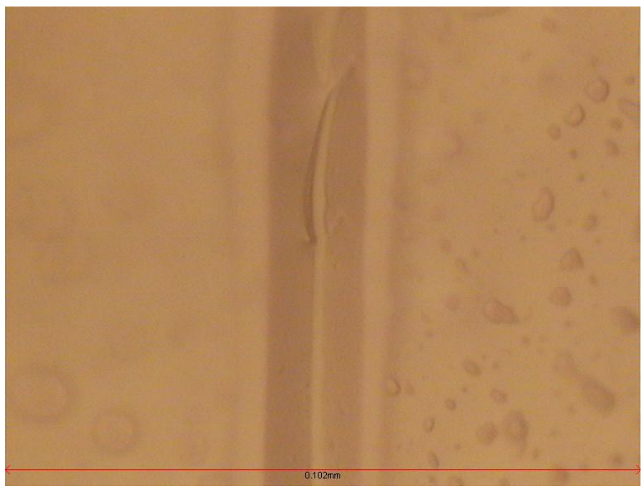


Figure 9 : Micrograph of Continuous Cracking – Aluminium 500x magnification (image width 0.102mm)

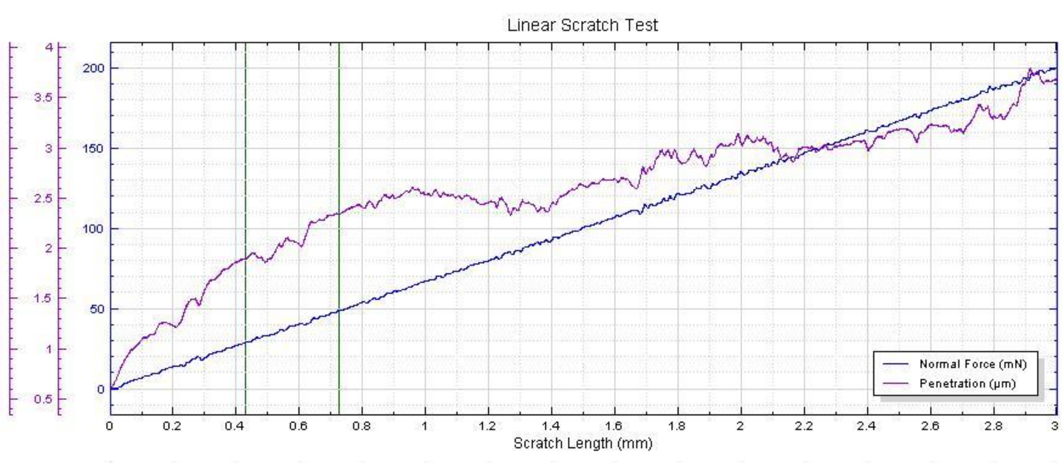


Figure 10 : Loading Curve – Aluminium

Conclusion

As seen in our analysis, the scratch/mar resistance level of nano coating has been identified on each substrate. The coating is prone to cracking and should be further investigated as to how it will relate to intended resistance level and performance. If the adhesion were not strong enough, full delamination or partial delamination would occur at the coating substrate interface. The value of nano scratch testing for this application is the ability to quantify with superior repeatability the scratch/mar resistance of the nano coating on each substrate. This test simulates either handling of the coating/substrate system or quantifies fracture toughness of the coating. The Nanovea Mechanical Tester, during Nano Scratch Tester Mode, is an excellent tool for the quality control of nano coatings on a wide range of substrates. The Failure points of nano coating can be quantitatively identified to insure a quality control and performance.