Using Nano Scratch Testing
For Composite Failure

Prepared by
Jorge Ramirez

6 Morgan, Ste156, Irvine CA 92618 · P: 949.461.9292 · F: 949.461.9232 · nanovea.com

INTRO

A Composite Material is a compilation of a short or long list of various other materials; referred to as constituent material. Constituent material is separated into two categories: matrix and reinforcement. The matrix material surrounds the reinforcement material and is enhanced by the properties of the reinforcement material. Essentially, by compiling various materials, in different formulas, a new material can be created and the resulting synergistic properties of the material can be controlled. Thus, the challenge is to create a formula from which each ingredient interacts with one another to yield intended results.

IMPORTANCE OF NANO SCRATCH TESTING FOR QUALITY CONTROL

One of the major concerns for composite material is it's durability in final form. The size of particles that form the strengthening additive can drastically affect the overall final performance of the composite material. By using the nano scratch testing method the failure of composite material can be compared to identify the most durable formula.

MEASUREMENT OBJECTIVE

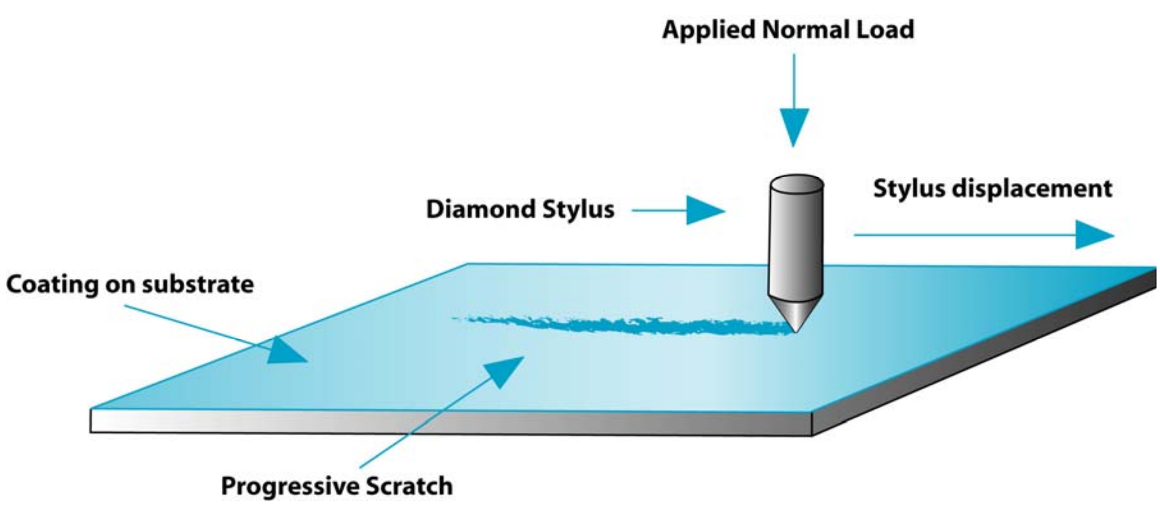
We must simulate the process of scratching in a controlled and monitored manner to observe sample behavior effects. In this application, the Nanovea Mechanical Tester in its nano scratch testing mode is used to measure the load required to cause failure to composite material samples. Two of the samples have micro size and one sample has nano size hard phase additive. A 5µm diamond tipped stylus is used at a progressive load ranging from 0.06 mN to 120 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 load.

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.



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.

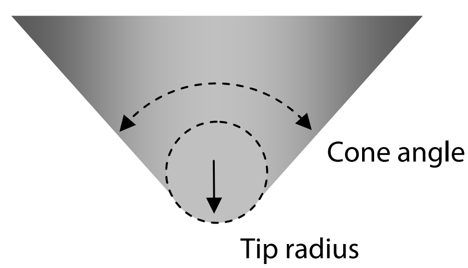
Tangential (frictional) force recording
This enables the force fluctuations along the scratch to be studied and correlated to the failures observed under the microscope. Typically, a failure in the sample will result in a change (a step, or a change in slope) in coefficient of friction. Frictional responses 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	0.06 mN
Final Load	120 mN
Loading rate	120 mN/min
Scratch Length	2 mm
Scratching speed, dx/dt	2.001 mm/min
Indenter geometry	90° cone
Indenter material (tip)	Diamond
Indenter tip radius	5 µm

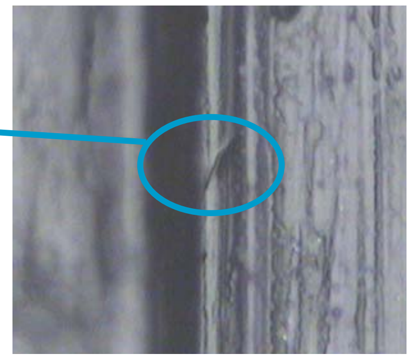


Sphero-conical indenter

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.	

Detailed results – Sample 1, Sample 2 & Sample 3

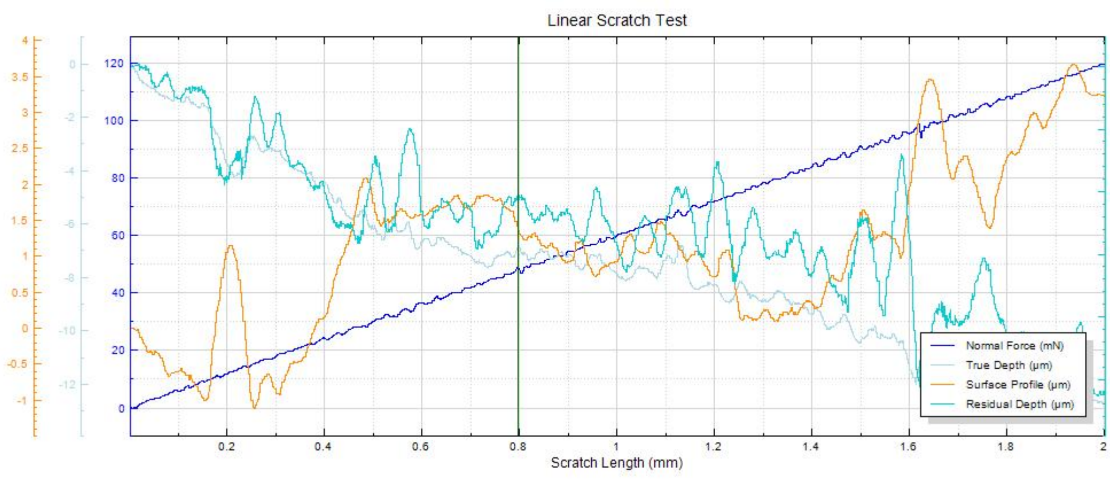
Sample	Initial Damage [mN]		
	Value		Std Deviation
1 (Micro)	37.989	±	9.108
2 (Micro)	36.402	±	2.999
3 (Nano)	77.202	±	11.386

Sample	Depth Study		
	Depth at 80mN	Residual Depth at 80mN	% of plasticity
1 (Micro)	8.6	2.9	34%
2 (Micro)	8.8	3.0	34%
3 (Nano)	6.9	1.5	22%

RESULTS - SAMPLE 1

SAMPLE 1	Scratch	Initial Damage [mN]
	1	32.253
	2	33.224
	3	48.491
	Average	**37.989**
	Std dev	**9.108**

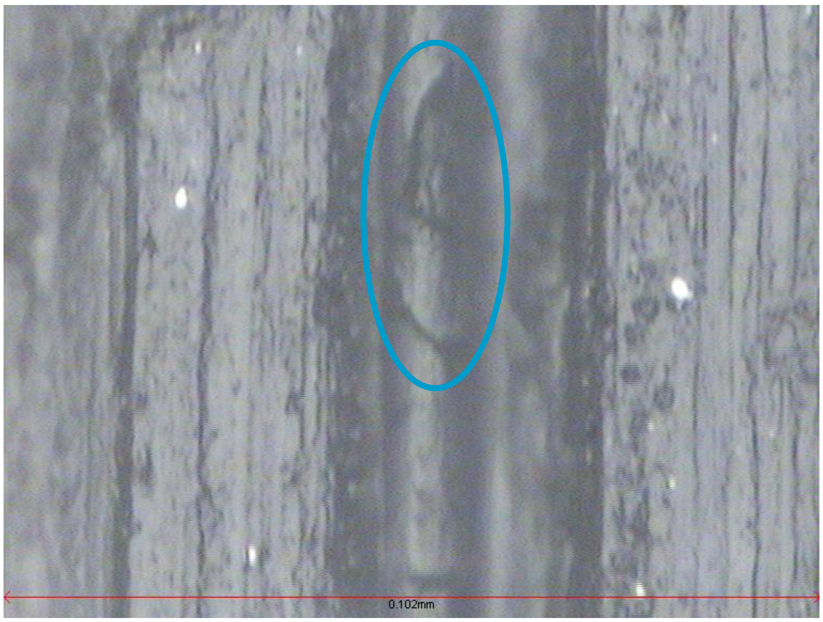
Micrograph of Initial Damage – SAMPLE 1, Scratch 3
500x magnification (image width 0.102mm)



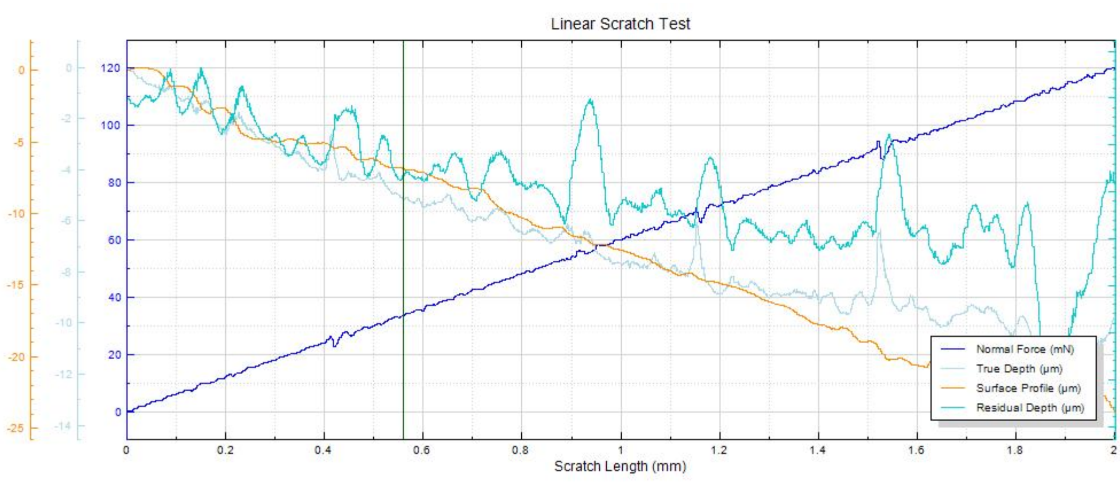
Loading Curve – SAMPLE 1, Scratch 3: Surface Profile shown in yellow is the profile of the surface measured at 0.06mN prior to the scratch test. This is used as the reference which is automatically subtracted from the other depth curves. The light grey curve is the profile of depth during the scratch. The green curve is the residual depth which is measured at 0.06mN after the scratch is performed.

RESULTS – SAMPLE 2

SAMPLE 1	Scratch	Initial Damage [mN]
	1	35.914
	2	39.615
	3	33.676
	Average	**36.402**
	Std dev	**2.999**



Micrograph of Initial Damage – SAMPLE 2, Scratch 3
500x magnification (image width 0.102mm)

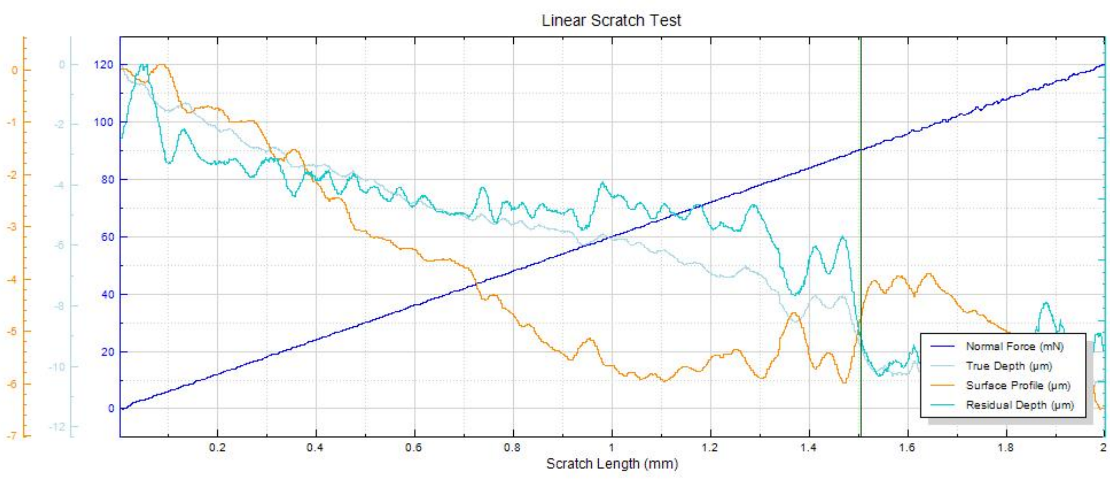


Loading Curve – SAMPLE 1, Scratch 3: **Surface Profile shown in yellow is the profile of the surface measured at 0.06mN prior to the scratch test. This is used as the reference which is automatically subtracted from the other depth curves. The light grey curve is the profile of depth during the scratch. The green curve is the residual depth which is measured at 0.06mN after the scratch is performed.**

RESULTS – SAMPLE 3

SAMPLE 1	Scratch	Initial Damage [mN]
	1	70.434
	2	90.347
	3	70.825
	Average	**77.202**
	Std dev	**11.386**

Micrograph of Initial Damage – SAMPLE 3, Scratch 2
500x magnification (image width 0.102mm)



Loading Curve – SAMPLE 3, Scratch 2: **Surface Profile shown in yellow is the profile of the surface measured at 0.06mN prior to the scratch test. This is used as the reference which is automatically subtracted from the other depth curves. The light grey curve is the profile of depth during the scratch. The green curve is the residual depth which is measured at 0.06mN after the scratch is performed.**

Conclusion

As seen in our analysis, the scratch/mar resistance level of composite materials has been identified. All the samples have shown cracking but the composite with the nano size hard phase additive has shown better resistance by cracking at more than double the value of the composite made with micro size hard phase additive. By measuring depth during the scratch and measuring the residual depth after the scratch, it also confirmed less deformation for the sample composite with nano size particles. It is interesting to note however that the two micro samples have a higher percentage of plasticity.

The Nanovea Mechanical Tester, during Nano Scratch Tester Mode, is an excellent tool for the quality control of composite materials. The use of the instrument in nano indentation mode would lead to additional information such as elastic modulus and creep information. Combined with AFM or Optical profiling, the instrument can extend the studies to roughness and 3D mapping of indents and scratches.