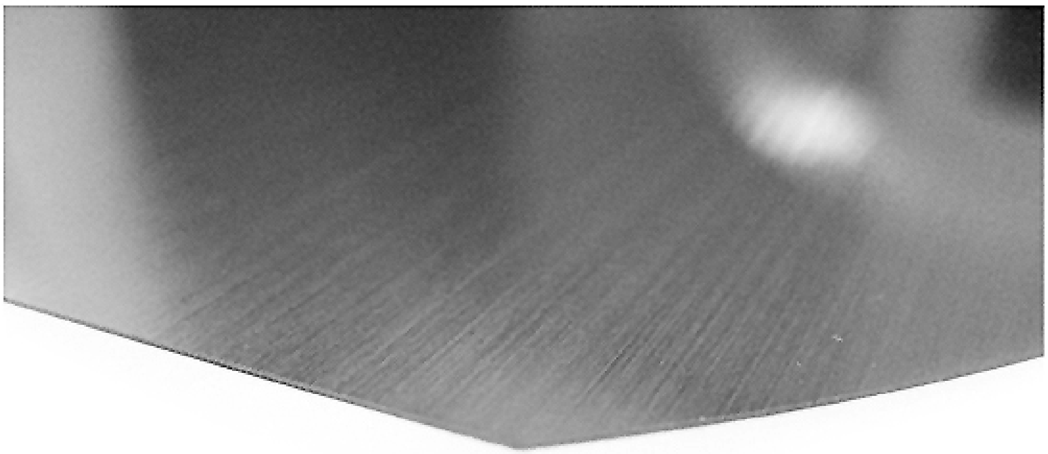
Fracture Resistance of Silicon With Nano Scratch Testing



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INTRO

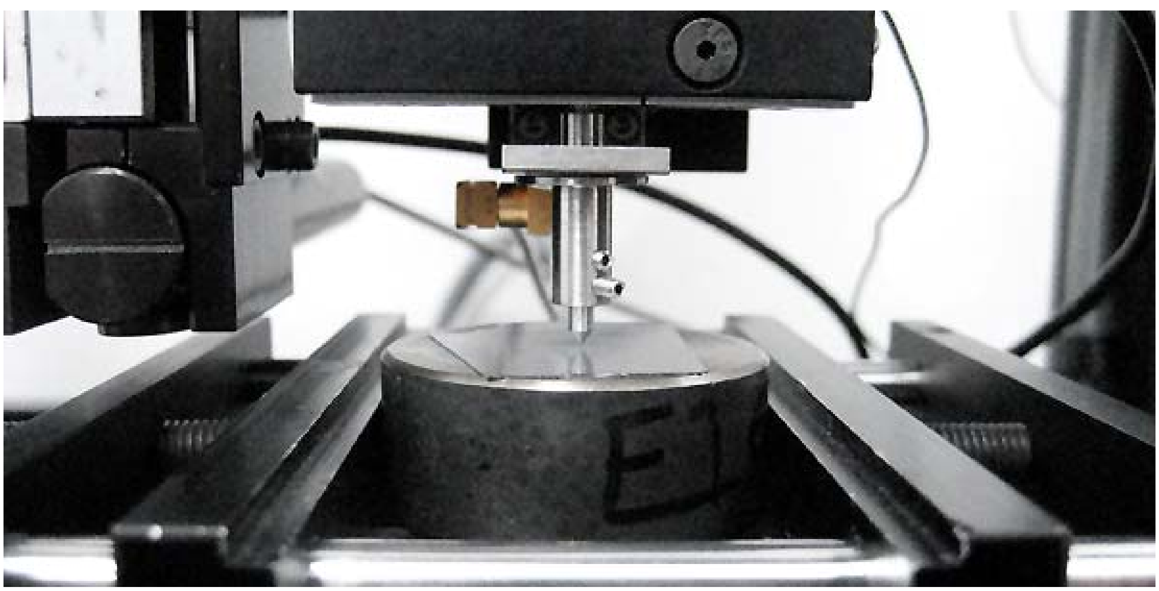
The scratch test, using nano loads, has become widely used for the study of adhesion strength and marring resistance of thin films and coatings. This same test can also be used to study fracture behavior on brittle substrates similar to a marring resistance test approach. This can provide an in-depth observation of failure on delicate substrate materials at precisely controlled and recorded loads.

IMPORTANCE OF NANO SCRATCH TESTING FOR QUALITY CONTROL

One of the major concerns for sensitive and brittle material is it's durability during intended use. Applications such as silicon, thin gold, glass, copper and others are widely used in microelectronics and require certain failure resistant levels. The chosen formula used to create the solid material can drastically affect the overall final resistance. By using the nano scratch testing method the failure of silicon, among others, can be tested and compared to identify the most durable formulation. Controlling failure on materials that are easily subject to "complete failure", such as silicon, highlights the sensitive control of the test and the possibilities for further applications.

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 nano scratch testing mode, is used to measure the load required to cause failure to 170μm thick silicon sample. A 2μm diamond tipped stylus is used at a progressive load ranging from 0.5 mN to 400 mN to scratch the silicon surface. Points of failure will be reviewed.

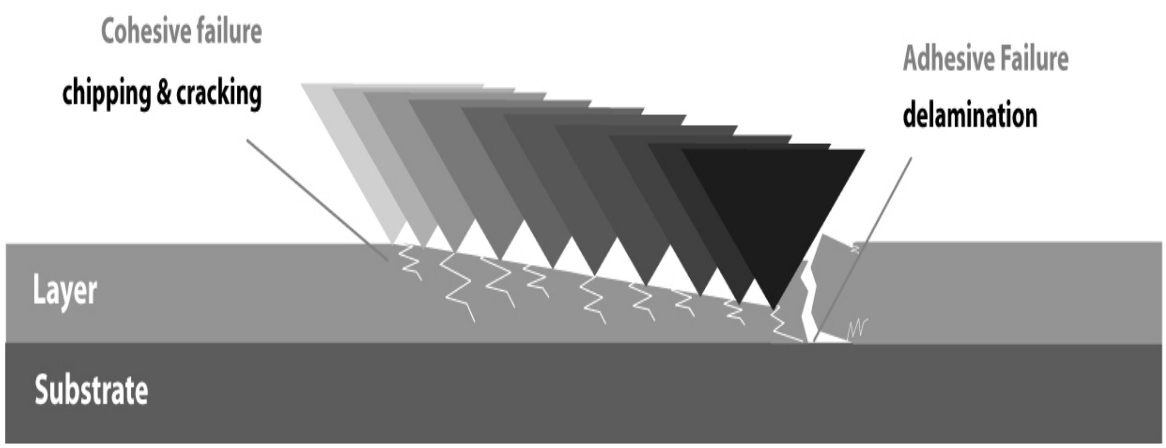


MEASUREMENT PRINCIPLE:

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.



The scratch test gives very reproducible quantitative data that can be used to compare the behavior of various coatings and substrate materials. 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

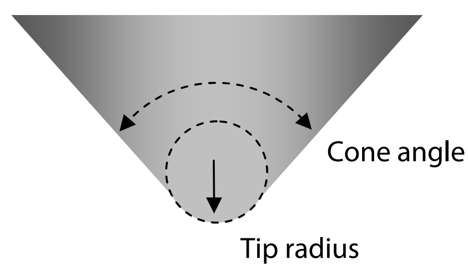
Microscopic observation
This is the most reliable method to detect surface damage. This technique is able to differentiate between cohesive failure within the coating and adhesive failure at the interface of the coating-substrate system.

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.

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.

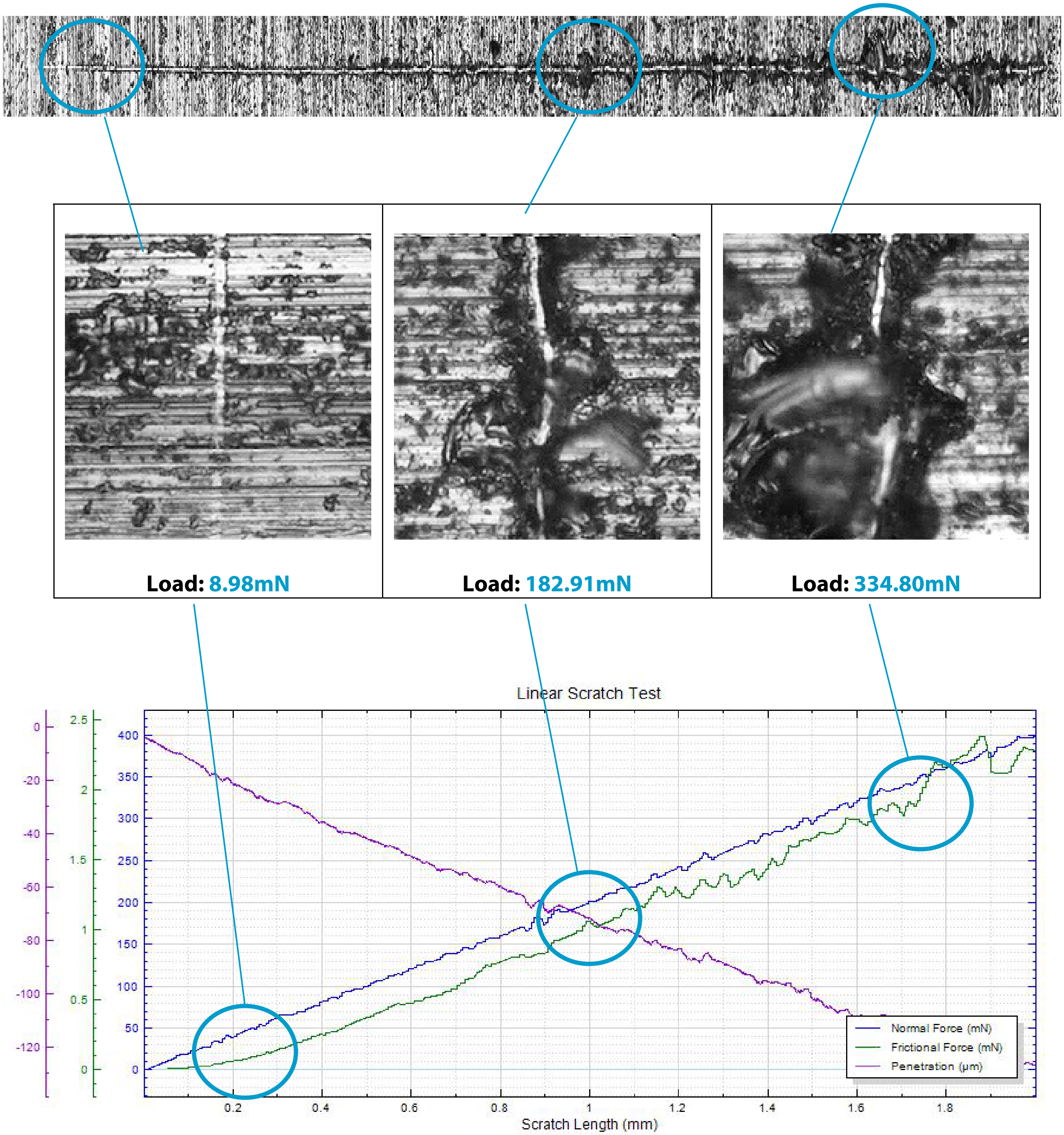
Test parameters

Load type	Progressive
Initial Load	0.5 mN
Final Load	400 mN
Loading rate	800 mN/min
Scratch Length	2 mm
Scratching speed, dx/dt	4.001 mm/min
Indenter geometry	90° cone
Indenter material (tip)	Diamond
Indenter tip radius	2 µm



Sphero-conical indenter

Results



Load: 8.98mN
Load: 182.91mN
Load: 334.80mN
Linear Scratch Test
Scratch Length (mm)
Normal Force (mN)
Frictional Force (mN)
Penetration (µm)
0
-20
-40
-60
-80
-100
-120
2.5
2
1.5
1
0.5
0
400
350
300
250
200
150
100
50
0
0.2
0.4
0.6
0.8
1
1.2
1.4
1.6
1.8

Conclusion

As seen in our analysis, the surface fracture resistance level of silicon has been identified with chipping failure. The quick and precise load control possible with the combination of the ultra sensitive load cell and the fast piezo table provides a smooth loading curve as the load increases even when intense chipping occurs on the surface. The Friction curve shows increase variation at points of major chipping as seen in the graph. Depth can also provide useful information by scanning prior to the test to obtain the leveling of the sample. Another possibility is the use of acoustic emission during the scratch using an AE sensor connected to the bottom of the sample material.

Unlike nanoindentation, the nano scratch test provides an understanding of mechanical behavior of the silicon surface at a progressive load while moving with linear force induced by the movement. Here we obtain a better understanding of mechanical failure with linear forces that would more closely resemble contact damage in the real world application. Again, it is important to note that 170μm thick silicon is extremely delicate making the precise load control of the nano scratch test critical to investigate the mechanical behavior of sensitive substrate materials. Traditional micro level tests would simply crack the entire silicon sample while the nano range allows progressive marring without provoking complete failure.

The Nanovea Mechanical Tester, during nano scratch mode, is a superior tool for the quality control of sensitive and brittle substrate materials. The use of the instrument in nanoindentation mode would lead to additional information such as elastic modulus, hardness and or fracture toughness information. Combined with AFM the instrument can extend the studies to roughness and 3D mapping of indents and scratches. To learn more about Nanovea Nanoindentation.