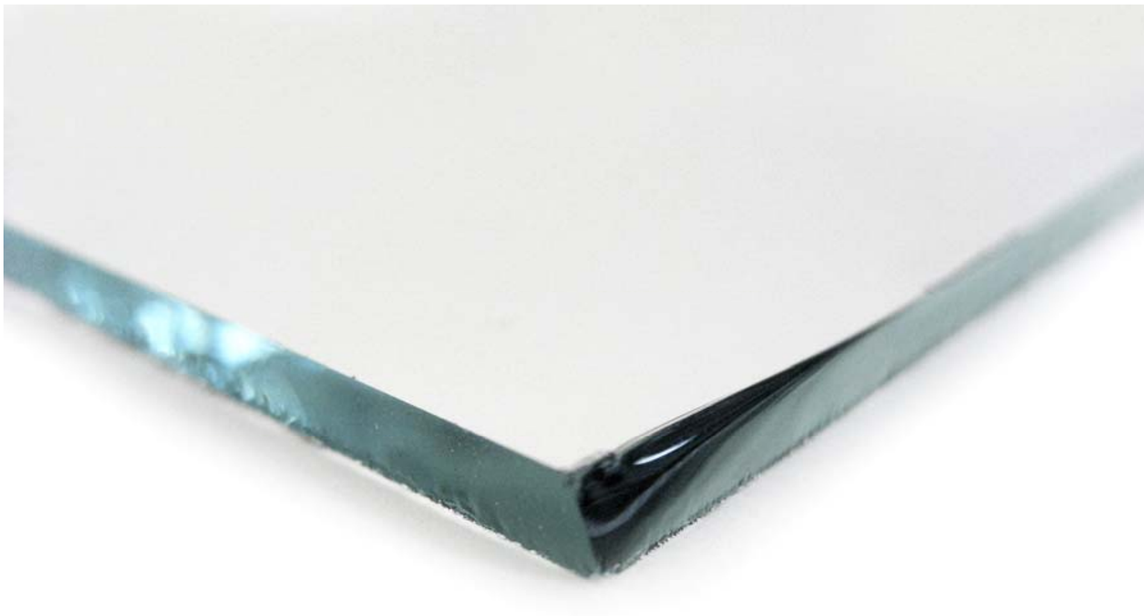
NANO SCRATCH TESTING
OF THIN FILM ON GLASS SUBSTRATE



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INTRO

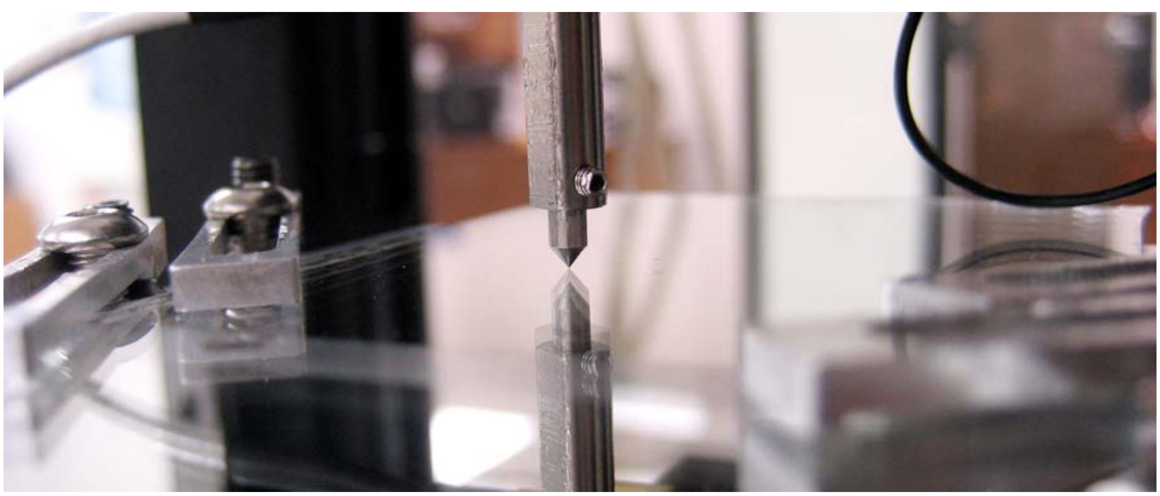
Substrates range from thin polymers, such as Teflon or PC, to thick refractory metals like Tungsten or Molybdenum. Glass substrates are getting more and more attention for specific area of research. One of the major uses is the manufacturing of power electronics and microelectronics. Often a specific layer are deposited on the surface of the glass to act as an interconnect between the circuit board and the electronic components. These layers are capable to carry current along with operating over a wide range of temperatures. Another role these layers play in power electronics is to act as an insulator or chemical barrier during the manufacturing of integrated circuits. In this research we focus on Aluminum and SiN thin layers on glass.

SUBSTRATE FABRICATION CONCERNS

Depending on the application a large number of different types of coating layers will be deposited onto the substrate. The different types of coating techniques for thin-film deposition include but are not limited to chemical vapor deposit (CVD), physical vapor deposit (PVD), electroplating, plasma spraying and spin coating. During processing these techniques can place a large amount of stress on the film. To insure the film does not fail during manufacturing, the max force required to damage these coatings and induce failure becomes valuable information. One method to obtain this critical value is through scratch testing.

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 of two different commonly used thin film materials, Aluminum (Al) and Silicon Nitrite (SiN), on glass. Each sample has a 200 nm coating on 3 mm thick glass. A 2μm diamond tipped stylus is used at a progressive load ranging from 0.05 mN to 80.00 mN to scratch the coating. The point where the coating fails by cracking or where delimitation from the glass occurs first is taken as the point of failure. Five tests where 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.

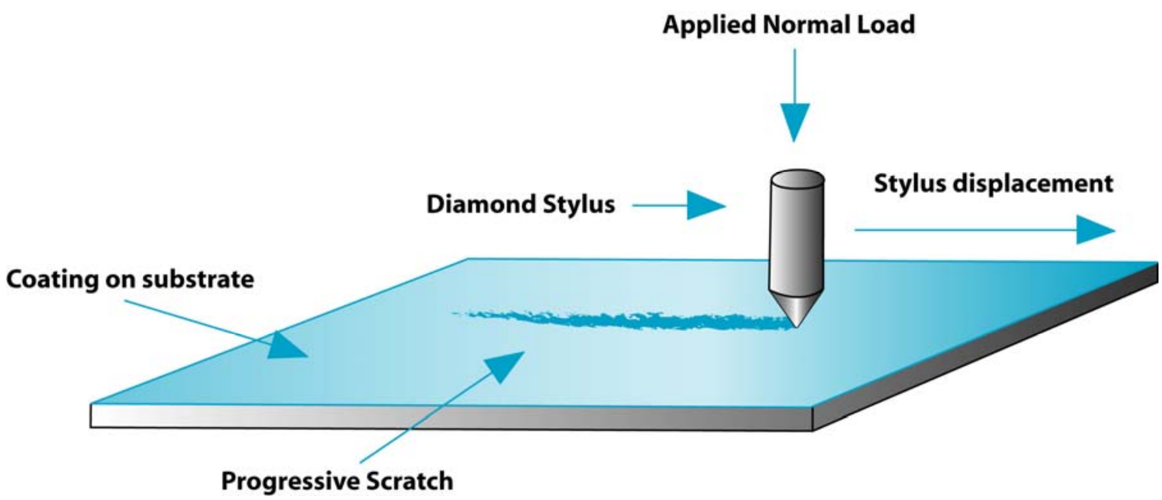


Figure 1 : Principle of scratch testing

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.

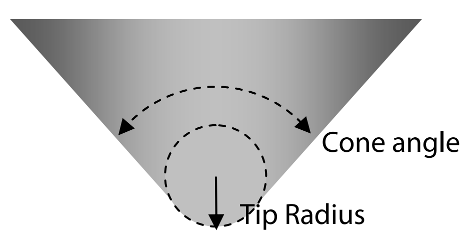
Tangential (frictional) force recording
This enables the force fluctuations along the scratch to be studied and correlated to the failures observed under the microscope. Typically, a failure in the sample will result in a change (a step, or a change in slope) in coefficient of friction. Frictional responses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

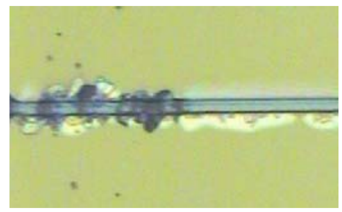
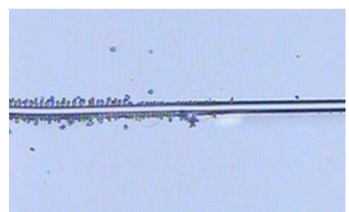
Load type	Progressive
Initial Load	0.050 mN
Final Load	80.00 mN
Loading rate	80.00 mN/min
Scratch Length	3 mm
Scratching speed, dx/dt	3 mm/min
Indenter geometry	90° conical
Indenter material (tip)	Diamond
Indenter tip radius	2 µm



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
SiN: Failure is the point where the coating fails in such a way that debris are visible for the remainder of the scratch track.	
Al: Failure is the point where the glass substrate actually fails. Aluminum adhesion is better than the glass resistance.	

Summary table of main numerical results

Sample	Failure [mN]		
	Value		Std Deviation
Al on Glass	50.945	±	1.042
SiN on Glass	34.346	±	0.518

Detailed results –Al on Glass

Critical loads - Al on Glass	
Scratch	**Failure* [mN]**
1	49.340
2	51.729
3	51.946
4	51.114
5	50.596
Average	**50.945**
Std dev	**1.042**

* Failure values taken at point of substrate cracking. No indications of the Aluminum film delamination was observed. Separate test of substrate was preformed to validate data.

Figure 2 : Micrograph of the Scratch at 20 mN – Al on Glass
500x magnification (image width 0.0615mm)

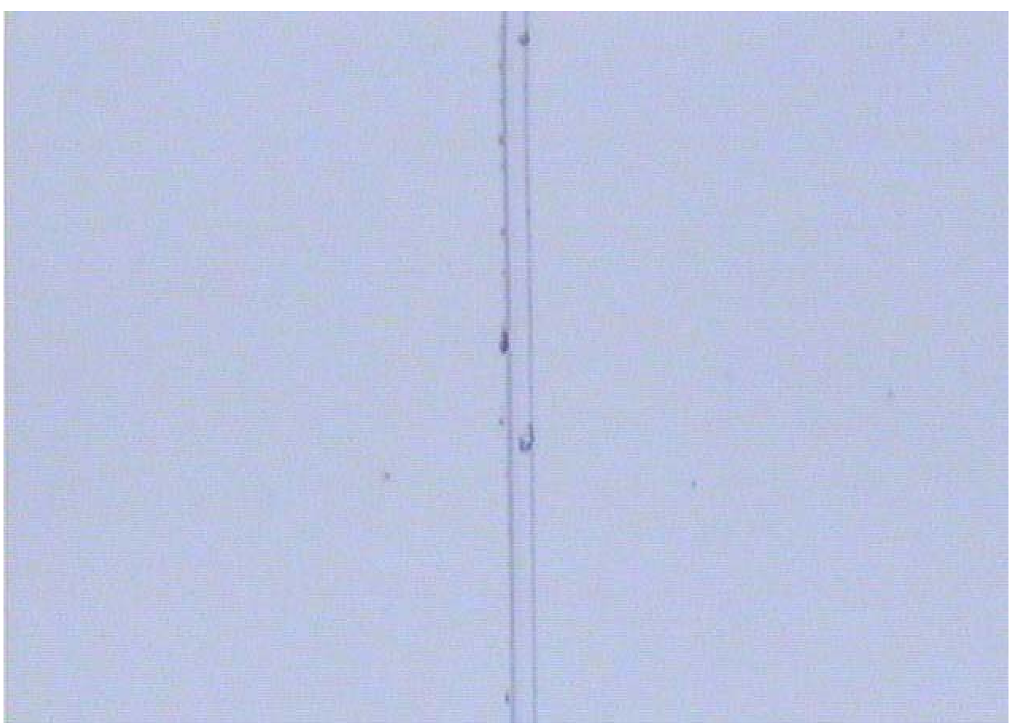


Figure 3 : Micrograph of the Scratch at Failure– Al on Glass
500x magnification (image width 0.0615mm)

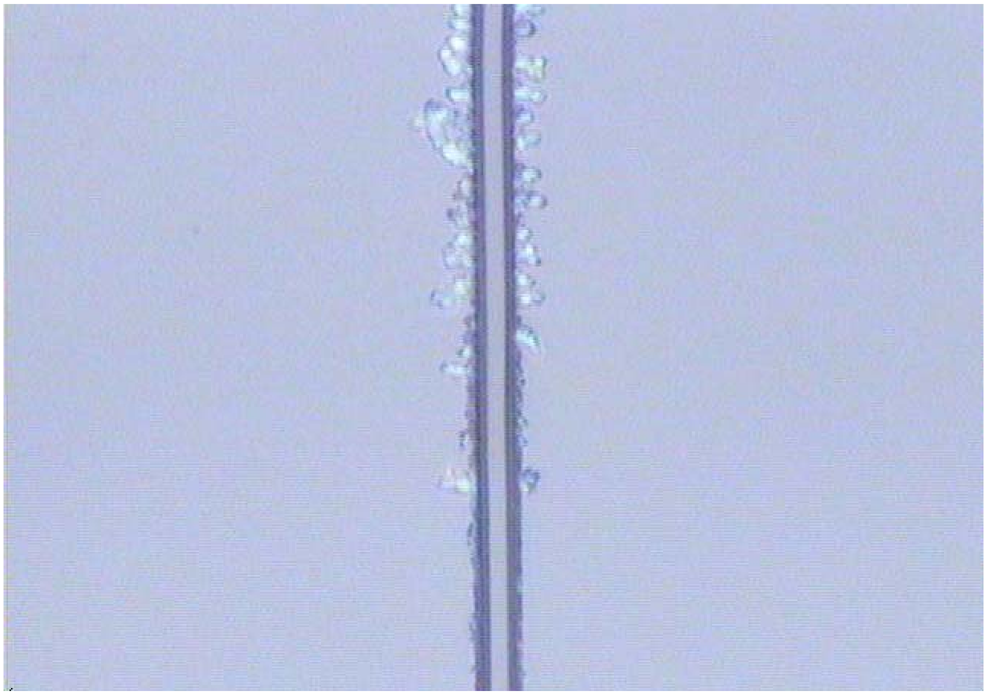


Figure 4 : Micrograph of the Scratch at 60 mN– Al on Glass
500x magnification (image width 0.0615mm)

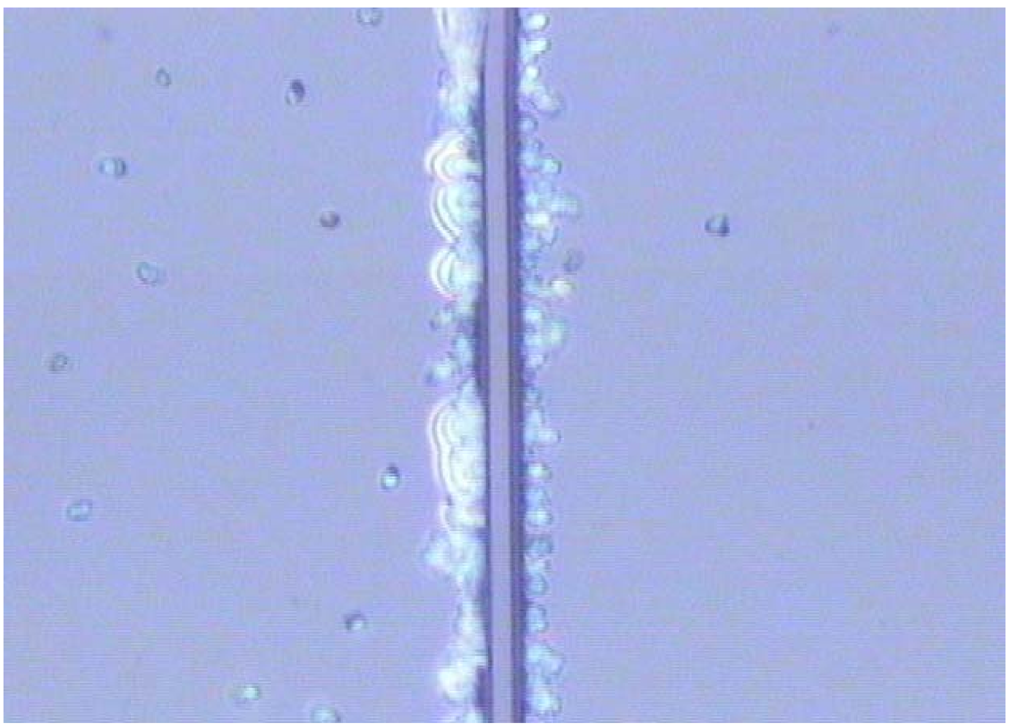
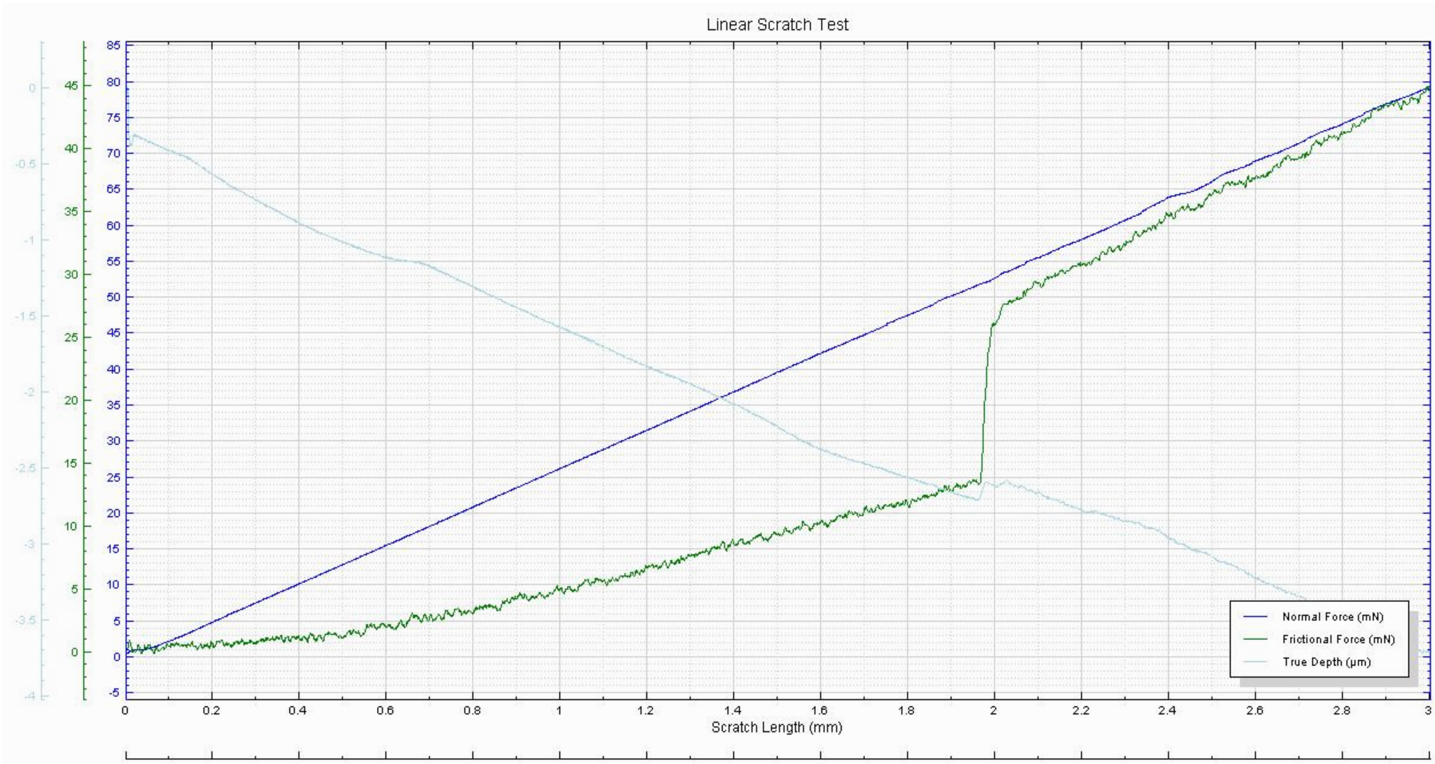


Figure 5 : Friction graph – Al on Glass



Note

The results of the scratch testing showed that failure of the aluminum coating could not be determined, rather, testing showed failure of the glass substrate. The adhesion capability of the aluminum coating on the glass allowed for coating to remain on the substrate during the scratch testing. This indicates that the substrate should fail before the coasting delaminates from the surface. It is because of this high adhesion property, along with the ability to withstand failure upon applied. In this case, it is clear that trying to improve adhesion of the coating by changing interface treatment or properties of the Aluminum layer will not improve mechanical performance since the substrate is what is prone to failure first.

Detailed results –SiN on Glass

Critical loads - SiN on Glass	
Scratch	**Failure[mN]**
1	34.327
2	34.957
3	34.207
4	33.581
5	34.657
Average	**34.346**
Std dev	**0.518**

Figure 6 : Micrograph of the Scratch at 20 mN – SiN on Glass
500x magnification (image width 0.0615mm)

Figure 7 : Micrograph of the Scratch at Failure – SiN on Glass
500x magnification (image width 0.0615mm)

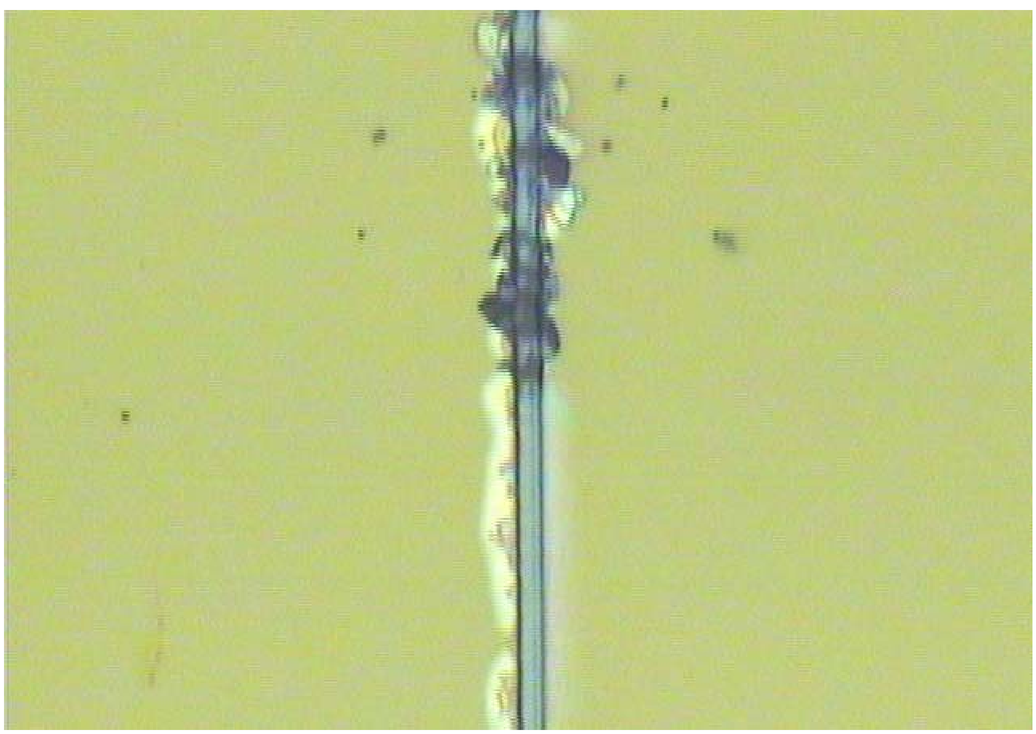


Figure 8 : Micrograph of the Scratch at 60 mN– SiN on Glass
500x magnification (image width 0.0615mm)

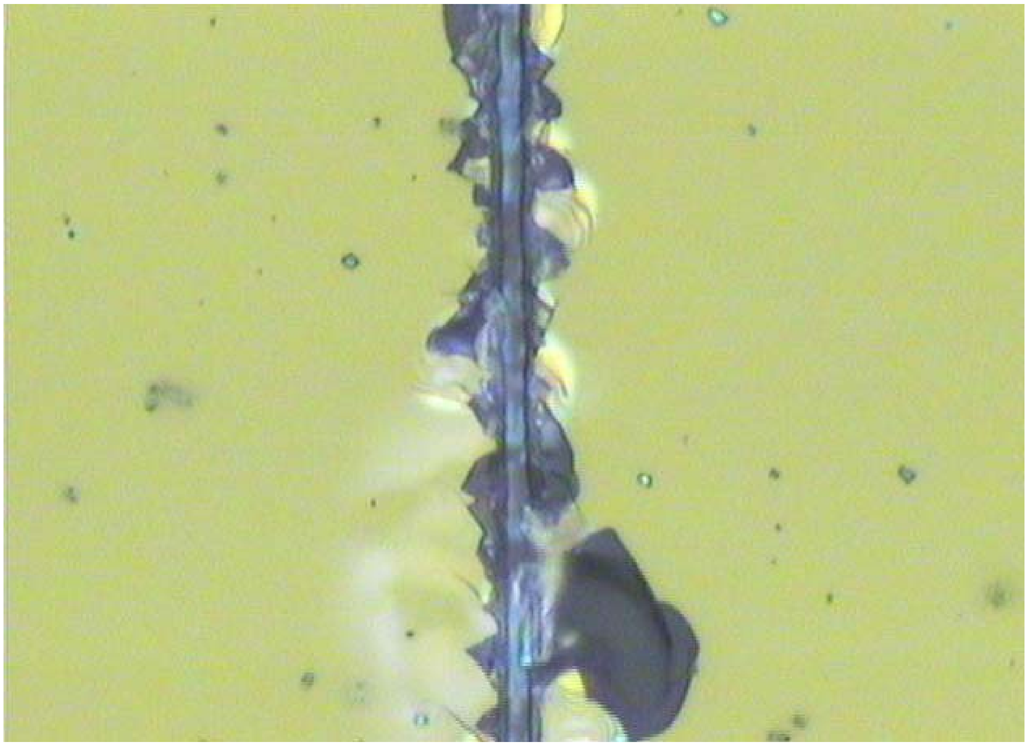
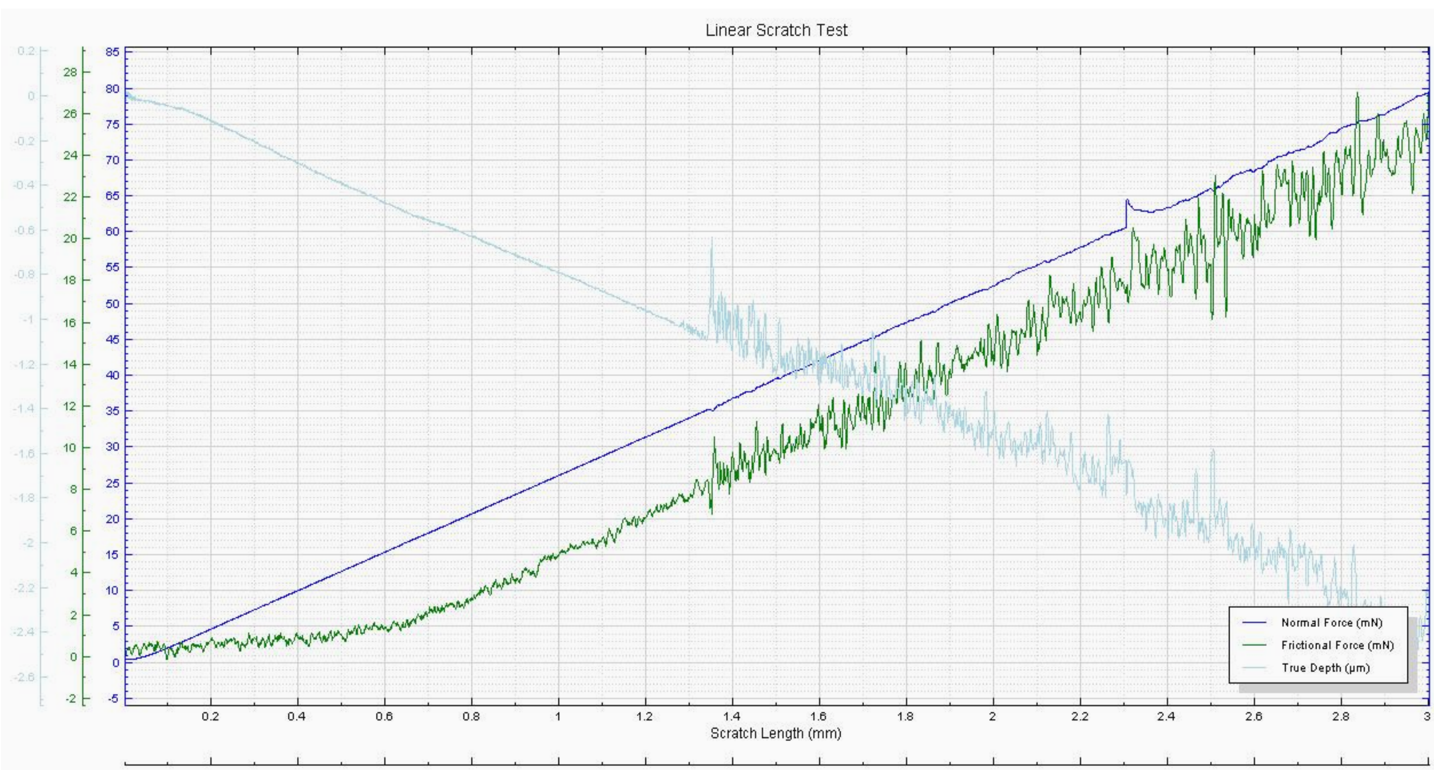


Figure 9 : Friction graph – SiN on Glass



Conclusion

Nanovea Mechanical Tester, during Nano Scratch Tester Mode, allows simulation of many real-life failures in thin coatings. By applying loads in a controlled and closely monitored fashion, the instrument allows to identify at what load failure in the scratch occurs. This can then be as a quantitative value for comparing scratch resistance between samples. A clear difference in the force required to fail is observed between the two samples. The very small standard deviations also show the reproducibility of the technique and of the instrument. This type of information can help manufactures improved the quality of their thin films.