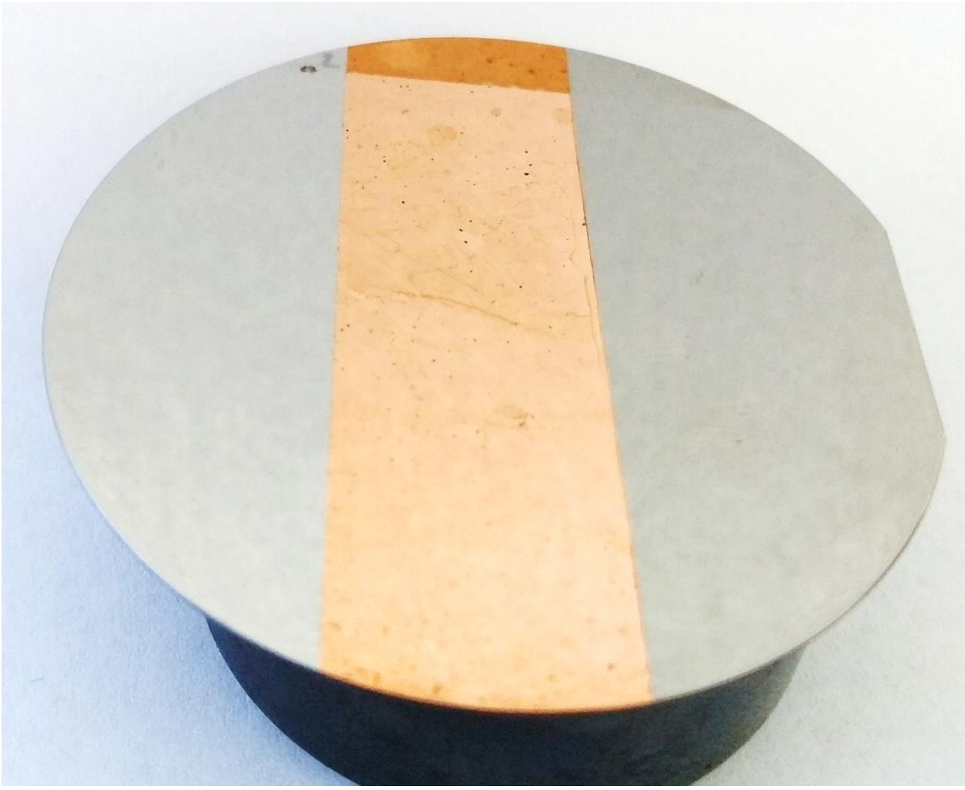
Scratch Testing on Multi-Layered Thin Film



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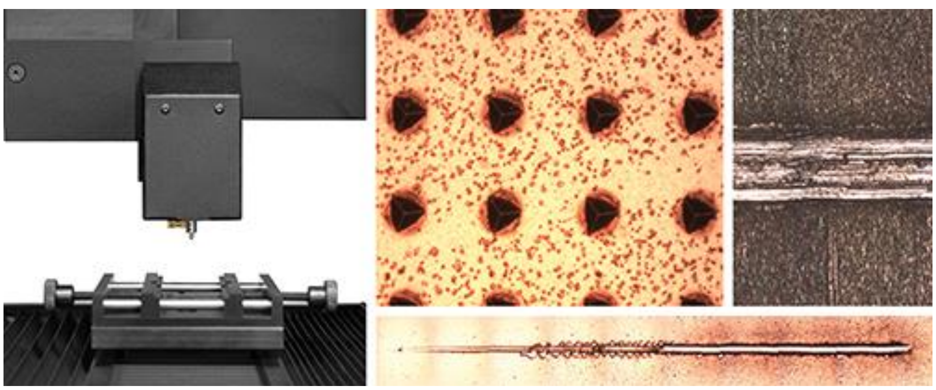
INTRO

Coatings used extensively throughout multiple industries to preserve the underlying layers, to create electronic devices, or to improve surface properties of materials. Due to their numerous uses coatings are extensively studied, but their mechanical properties can be difficult to understand. Failure of coatings can occur in the micro/nanometer range from surface-atmosphere interaction, cohesive failure, and poor substrate-interface adhesion. A consistent method to test for coating failures is scratch testing. By applying a progressively increasing load, cohesive (e.g. cracking) and adhesive (e.g. delamination) failures of coatings can be quantitatively compared.

IMPORTANCE OF SCRATCH TESTING ON MUTLILAYERED COATINGS

Scratch testing is a modern destructive testing method to determine the adhesive and cohesive properties of coatings. Adhesive properties refer to how well the substrate binds to the coating at the interface, while cohesive properties refer failure within the coating region. These properties of coatings are important for failure analysis and can be used to compare results from different manufacturing processes. Most common are single coating/substrate samples. Consistent data can still be obtained from multi-layered samples via scratch testing with Nanovea's Mechanical Tester.



MEASUREMENT OBJECTIVE

Scratch testing was conducted on a sample coated with multiple thin layers of materials on top of a silicon substrate. Failures were identified visually using an optical microscope.

TEST CONDITIONS AND PROCEDURE

Test Parameters	
Load type	Progressive
Initial Load (N)	0.02
Final Load (N)	2
Loading rate (N/min)	10
Scratch Length (mm)	3
Scratching speed (mm/min)	15
Indenter geometry	120° cone
Indenter material (tip)	Diamond
Indenter tip radius (µm)	20

Table 1: Scratch test parameters for multi-layered sample

RESULTS

The scratch tests done on the sample consistently shows two adhesive failures. The first delamination of the first coating occurs at 0.039 ± 0.012N. The second delamination point occurs at 1.068 ± 0.102N. No other delamination points were identifiable under an optical microscope. In addition to the two adhesive failures, a cohesive failure of the silicon wafer was also found to occur consistently at 1.779 ± 0.043N.

Scratch	First Delamination (N)	Second Delamination (N)	Cohesive Failure (N)
1	0.025	1.157	1.727
2	0.055	1.123	1.833
3	0.038	0.925	1.778
Average	**0.039**	**1.068**	**1.779**
Standard Deviation	**0.012**	**0.102**	**0.043**

Table 2: Result summary of scratch test on multi-layered sample

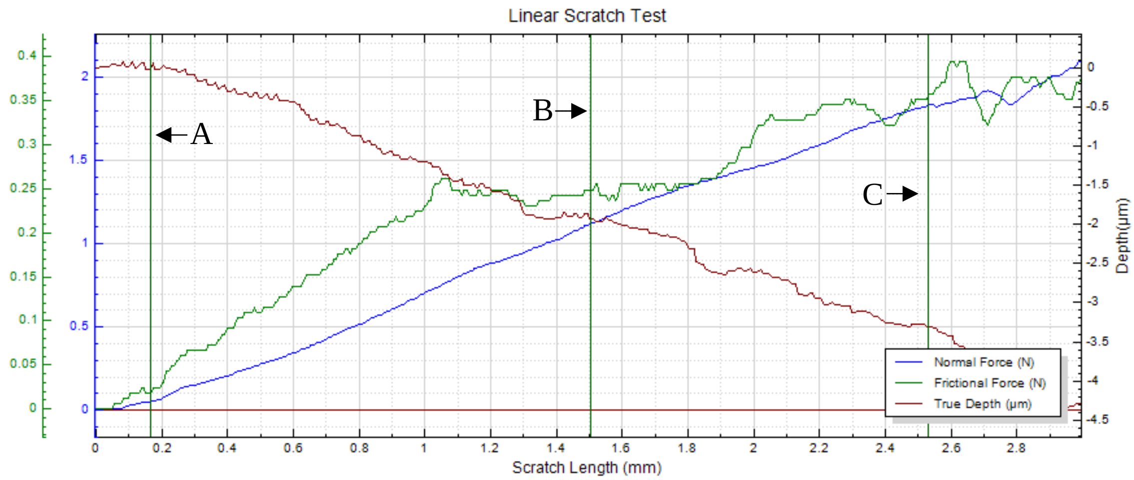
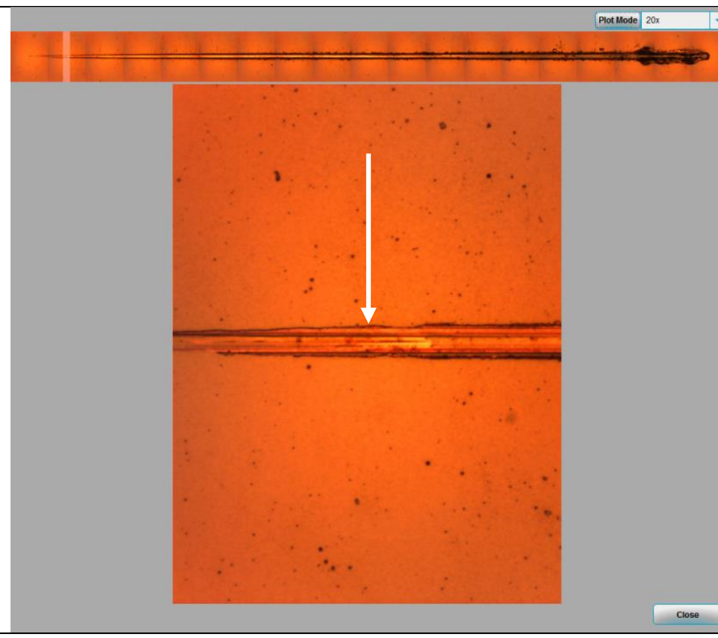


Figure 1: Friction graph of scratch test on multi-layered sample. A) First delamination point, B) Second delamination point, C) Cohesive Failure.

A) First Delamination Point The first delamination point was identified by the change in color. The underlying layer starts becoming exposed at this point.	

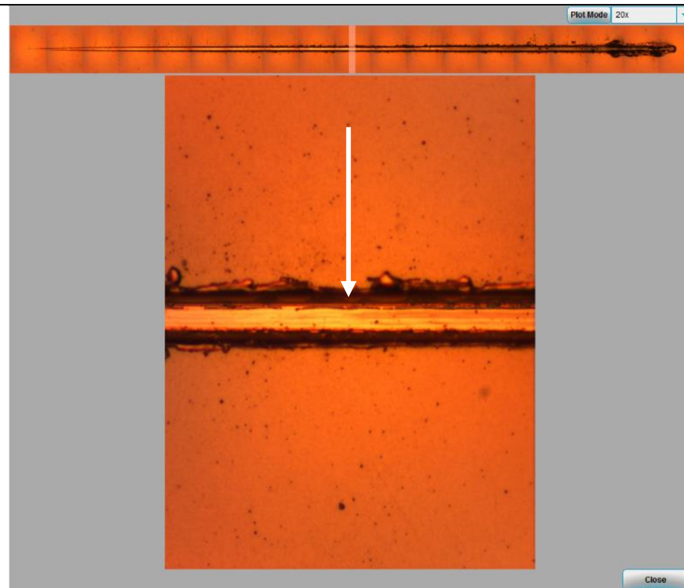
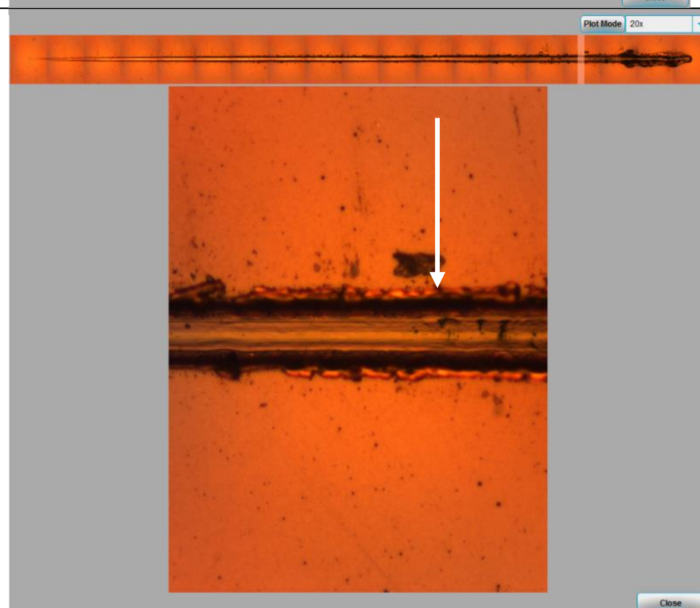
B) Second Delamination Point The second delamination point was identified by the change in color.	
C) Cohesive Failure The cohesive failure was identified by the change in texture on the surface.	

Table 3: Failure analysis of scratch test on multi-layered sample

Figure 2: Full length image of scratch test on multi-layered sample.

The friction graph above, figure 1, shows the data collected over the course of the scratch. The data is often used as a supplement to optical inspection, but did not show clear signs of critical failures for our sample (see means for critical load determination pg.8). With our precise stage movements, we can identify the critical load of the failure by mapping an image of the scratch with all the data collected, including load. By matching the image with the data, the load, depth, and frictional force can be pinpointed at various positions in the scratch.

Description of the failure and how they were identified are listed in Table 3.

CONCLUSION:

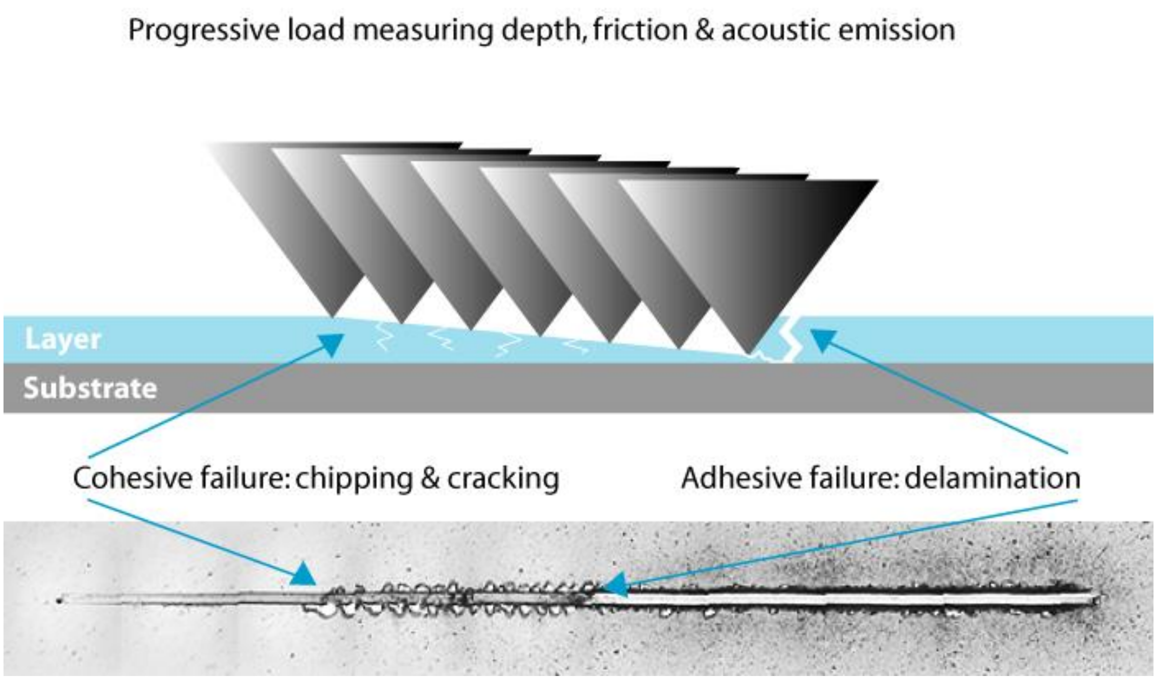
The controlled and monitored fashion the Nanovea Mechanical Tester conducts its scratch test was able to create clear, consistent delaminations on a sample with multiple layers of thin films. In three different scratches, the sample clearly displays two points of delamination at 0.039 ± 0.012N and 1.068 ± 0.102N. Cohesive failure was found to be at 1.779 ± 0.043N. These failures were able to be clearly distinguished in all scratches conducted.

Theory of Scratch Testing

Principle

The scratch testing method is a quantitative test in which critical loads at which failures appear in the samples are used to evaluate the relative cohesive or adhesive properties of a coating or the scratch resistance of a bulk material. During the test, scratches are made on the sample with a sphero-conical stylus 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 styluses are 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.



Comments on the critical load

The scratch test is a quantitative test with high repeatability. The critical load depends on the mechanical strength (adhesion, cohesion) of a combined coating-substrate system 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.

Test parameters affecting critical load:

- Loading rate
- Scratching speed
- Indenter tip radius
- Indenter material (and also indenter tip wear)

Sample specific parameters affecting critical load:

- Friction coefficient between surface and indenter
- Internal stresses in the material
- Substrate hardness and roughness
- Coating hardness and roughness
- Coating thickness

By keeping the test parameters constant one can obtain very repeatable data to quantifiably compare samples.

Means for critical load determination

Microscopic observation 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.

The friction force recording 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.

The depth sensor recording can also sometimes indicate where a failure occurs. Typically, a significant fall in the depth will indicate that the indenter has broken through one layer of a sample down to the next. The depth recording can also be used to study deformation of a sample surface. Plastic and elastic deformation can be studied by performing pre- and post-scans of the scratch.