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A Better Measure.

Tablet Coating Failure
Using Micro Scratch Testing

Prepared by
Jorge Ramirez

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

INTRO

Tablets are covered by various coatings for many reasons, such as how the pill will dissolve once ingested. These requirements inside the body sometime conflicts with the mechanical properties required to keep appearance and durability before use. During the manufacturing and packaging process of pharmaceutical tablets there are many environmental situations that can harm the coverage and appearance of the coating. Two main possibilities of damages occur either when the pill is travelling on the metallic conveyer or when the pill is inside the container where it is in contact with other tablets. The coating is intended to withstand the process of conveying and packaging with minimal failure while maintaining the requirement during ingesting. The reliability of the tablet coating process is critical to existing tablet production and will be even more so with new or improved pharmaceutical coatings.

IMPORTANCE OF MICRO SCRATCH TESTING FOR QUALITY CONTROL

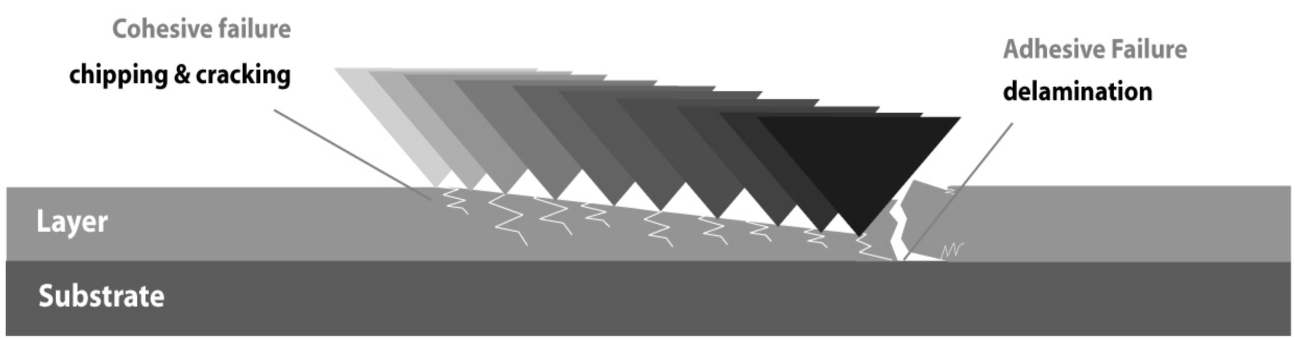
A major concern for the manufactures of pharmaceutical tablets will be to insure strong coating adhesion and or ability to withstand marring/cracking. A tablet coating that losses its adhesion and or begins to crack is a serious product failure when thousands of tablets will be coated with the same process. As tablet medication continues to grow, quality assurance will play a crucial role in establishing a reliable coating process. Although adhesion and or cracking failure may be inevitable or even intended over time, it is crucial that these failures are investigated, known and controlled. Using the Micro Scratch Test, precisely controlled loads can be used to investigate a tablets coating for these very failures.

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 micro scratch mode, is used to measure the load required to cause the failure to a generic and brand tablet coating. A 20µm diamond tipped stylus is used at a progressive load ranging from 4 N to 8 N to scratch the tablet coating. The point where the coating fails by cracking is taken as the point of failure. Hardness and elastic modulus will also be evaluated in nanoindentation mode.

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.



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.

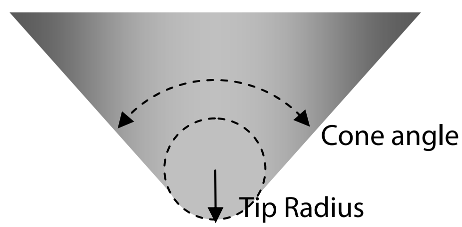
Tangential (frictional) force recording
This enables the force fluctuations along the scratch to be studied and correlated to the failures observed under the microscope. Typically, a failure in the sample will result in a change (a step, or a change in slope) in coefficient of friction. Frictional responses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

Load type	Progressive
Initial Load (N)	0.08
Final Load (N)	4.00
Loading rate ((N/min)	8.00
Scratch Length (mm)	3.00
Indenter geometry	Rockwell C, 120°
Indenter tip radius	20µm
Indenter material (tip)	Diamond

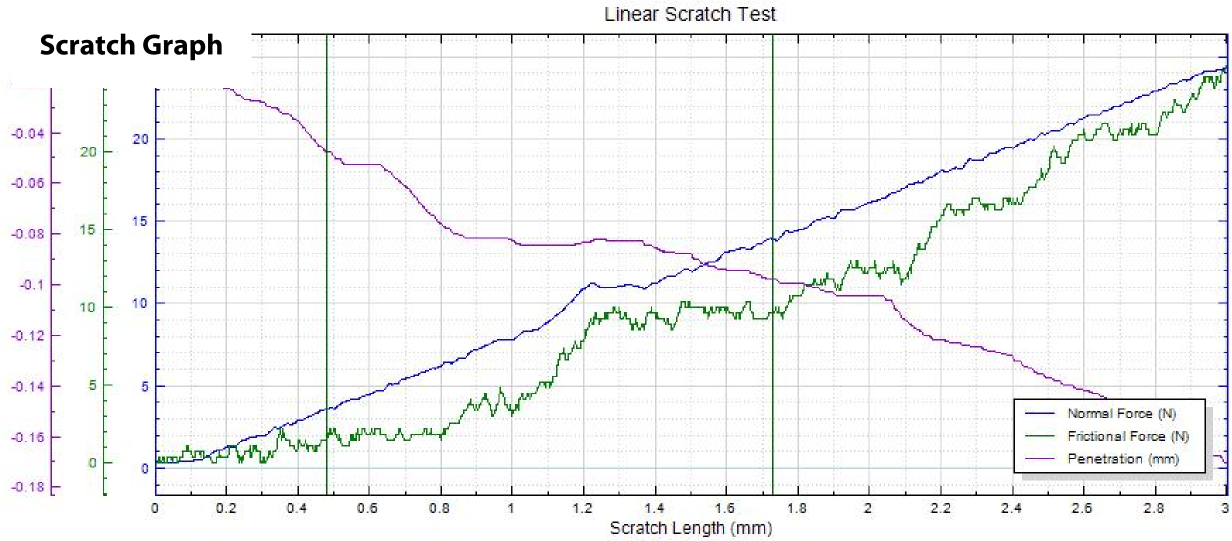


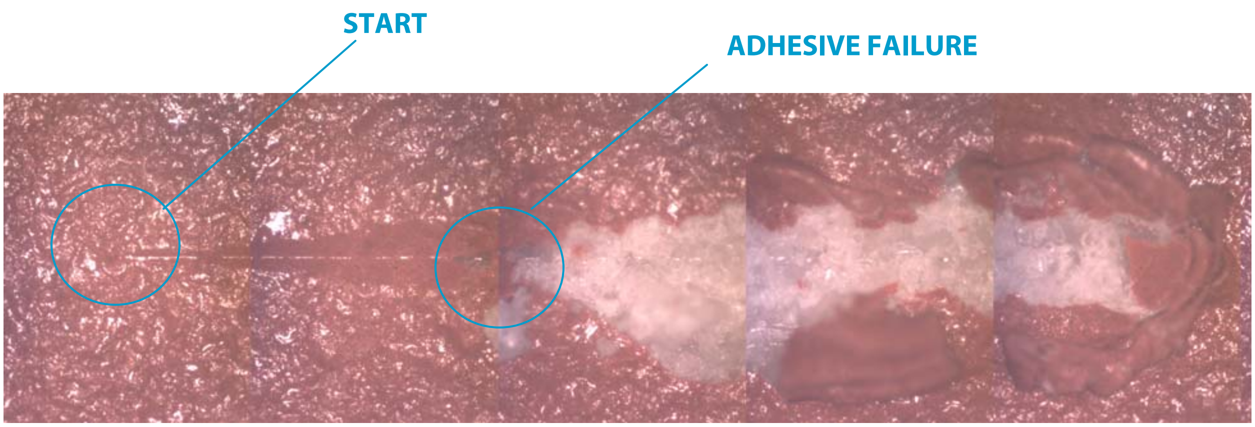
Test parameters-Hardness

Approach Speed(µm/min)	1
Contact Load(mN)	0.03
Initial Load (mN)	0.03
Final Load (mN)	7
Loading rate ((mN/min)	14
Indenter geometry	Berkovich
Indenter material (tip)	Diamond

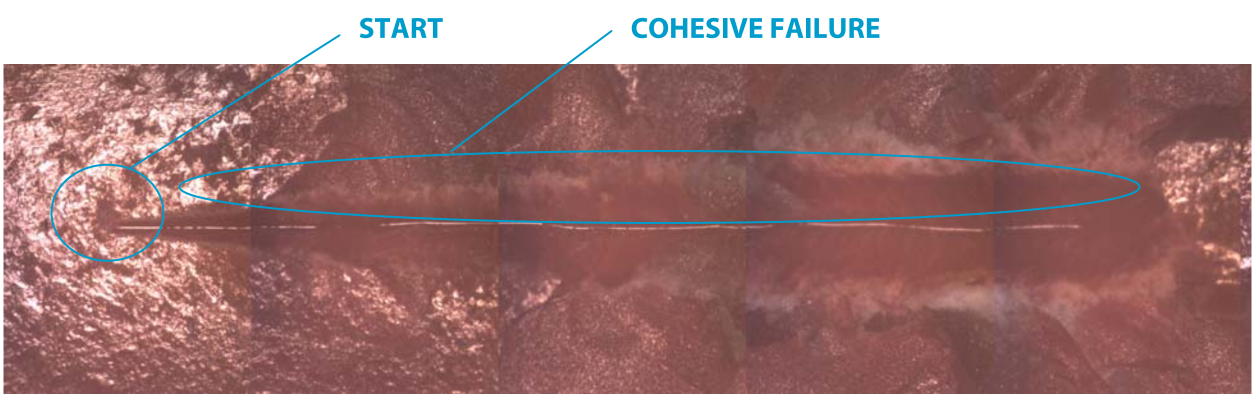
Results- Scratch

Scratch Graph



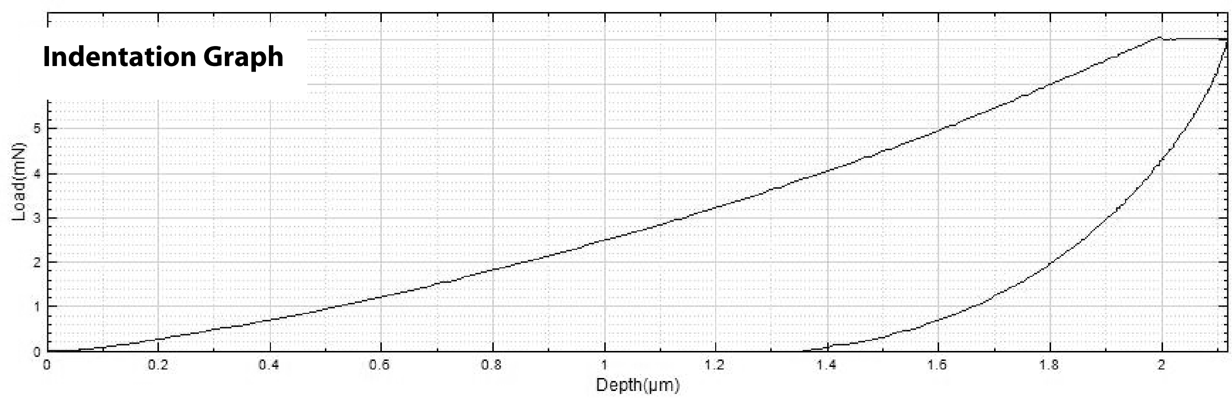


Generic- The critical load at which failure (delamination) occurred was 2.07N. Here we see the substrate exposed from delamination.



Brand- The critical load at which failure (cracking) occurred was 0.64N. Here we see the substrate exposed from cracking on the sides of the scratch.

Results- Hardness



GENERIC					
Test	**Max Load**	**Max Depth**	**Hardness(Gpa)**	**Hardness(HV)**	**Elastic Modulus**
Test 1	7.037	1990	0.1036	9.79	1.664
Test 2	7.014	1969	0.1086	10.27	1.595
Test 3	7.021	2024	0.1078	10.19	1.386
Average	**7.024**	**1994**	**0.107**	**10.08**	**1.548**

BRAND					
Test	**Max Load**	**Max Depth**	**Hardness (Gpa)**	**Hardness (HV)**	**Elastic Modulus**
Test 1	7.027	780.2	0.6303	59.56	10.06
Test 2	7.027	792.9	0.5187	49.01	11.30
Test 3	7.014	824.0	0.4815	45.49	10.64
Average	**7.023**	**799.0**	**0.543**	**51.36**	**10.67**

Conclusion

From the hardness test we can see that the Generic tablet is covered by a softer and more elastic coating. During scratch testing the failure seen on the Generic tablet show how the more elastic coating fully delaminates by ripping open at 2N. The coating of the Brand tablet is much harder and brittle. During scratch testing the failure of the Brand coating fails with cracks and chipping at 0.6N. Here the harder coating is pushed into the soft substrate indicated by the exposure of substrate on each side of the scratch. Visually the failure will be more apparent at over 2N on the Generic sample than the Brand samples. This is a typical scenario when comparing soft vs. brittle coatings on soft substrate.

As shown, the value of micro scratch testing for tablet coatings is the ability to quantify with superior repeatability the adhesion (adhesive failure) and cracking (cohesive failure) of the tablet coatings. There are many indenter tips that can be used to simulate various levels of scratches. Additionally, the Nanovea Mechanical Tester could have also been used to measure wear, friction coefficient, compression, fracture toughness, roughness and many others. It is a complete and powerful tool for tablet coating research and control. To learn more: Nanovea Mechanical Tester.