ADHESION PROPERTIES OF GOLD COATING
ON QUARTZ CRYSTAL SUBSTRATE

Prepared by
Duanjie Li, PhD

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

INTRODUCTION

The Quartz Crystal Microbalance (QCM) is an extremely sensitive mass sensor capable of making precise measurements of small mass in the nanogram range. QCM measures the mass change on the surface through detecting variations in resonance frequency of the quartz crystal with two electrodes affixed to each side of the plate. The capacity of measuring extreme small weight makes it a key component in a variety of research and industrial instruments to detect and monitor the variation of mass, adsorption, density, and corrosion, etc.

IMPORTANCE OF SCRATCH TEST FOR QCM

As an extremely accurate device, the QCM measures the mass change down to 0.1 nanogram. Any mass loss or delamination of the electrodes on the quartz plate will be detected by the quartz crystal and cause significant measurement errors. As a result, the intrinsic quality of the electrode coating and the interfacial integrity of the coating/substrate system play an essential role in performing accurate and repeatable mass measurement. The Micro scratch test is a widely used comparative measurement to evaluate the relative cohesion or adhesion properties of coatings based on comparison of the critical loads at which failures appear. It is a superior tool for reliable quality control of QCMs.

MEASUREMENT OBJECTIVE

In this application, the Nanovea Mechanical Tester, in Micro scratch Mode, is used to evaluate the cohesive & adhesive strength of the gold coating on the quartz substrate of a QCM sample. We would like to showcase the capacity of Nanovea Mechanical Tester in performing micro scratch tests on a delicate sample with high precision and repeatability.

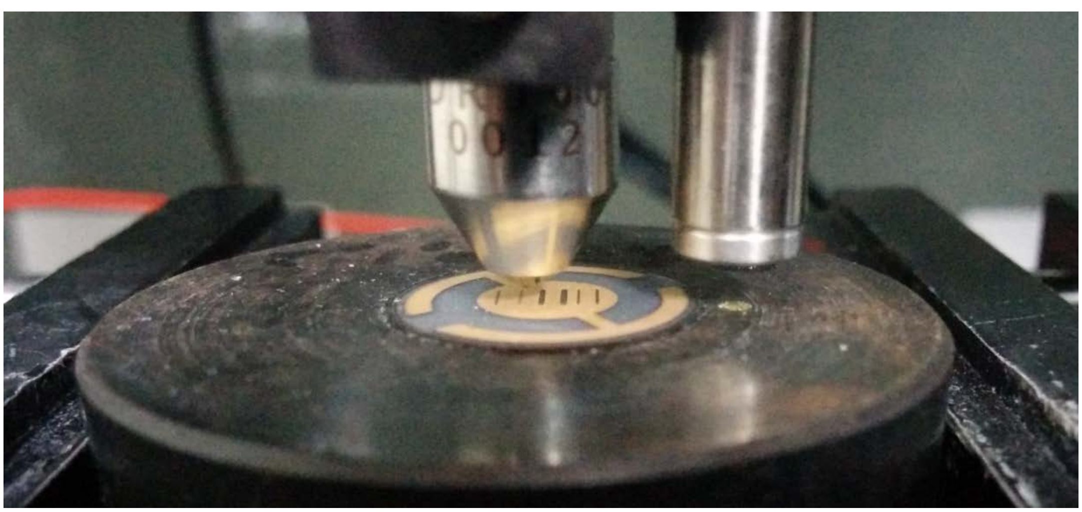


Fig. 1: Micro scratch tip and depth sensor on the QCM sample.

TEST CONDITIONS

The Nanovea Mechanical Tester was used to perform the micro scratch tests on a QCM sample using the test parameters summarized in Table 1. Three scratches were performed to ensure reproducibility of the results.

Test parameters	**Value**
Load type	Progressive
Initial load	0.01 N
Final load	30 N
Sliding speed	2 mm/min
Sliding distance	2 mm
Indenter geometry	Rockwell (120° cone)
Indenter material (tip)	Diamond
Indenter tip radius	100 µm
Atmosphere	Air
Temperature	24°C (room)

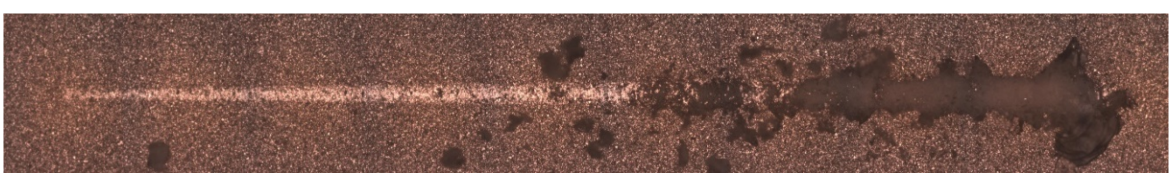
Table 1: Test parameters of the micro scratch measurements.

RESULTS AND DISCUSSION

The full micro scratch track on the QCM sample is shown in Fig. 2a. The failure behaviors at different critical loads are displayed in Fig. 2b, c and d, where critical load, L_{c1} is defined as the load at which the first sign of adhesive failure occurs in the scratch track, L_{c2} is the load after which repetitive adhesive failures take place, and L_{c3} is the load at which the coating is completely removed from the substrate.

It can be observed that at little chipping takes place at L_{c1} of 11.15 N as the first sign of coating failure. As the normal load continues to increase during the micro scratch test, repetitive adhesive failures occurs after L_{c2} of 16.29 N. When L_{c3} of 19.09 N is reached, the coating completely delaminates from the quartz substrate. Such critical loads can be used to quantitatively compare the cohesive and adhesive strength of the coating to select the best candidate for targeted applications.

(a)



(b) L_{C1} = 11.15 N: (c) L_{C2} = 16.29 N: (d) L_{C3} = 19.09 N:

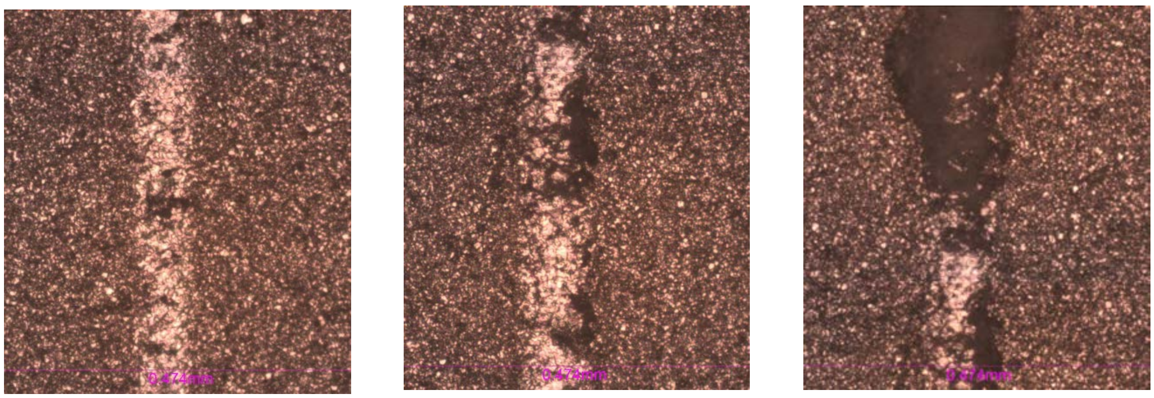


Fig. 2: The micro scratch track at different critical loads.

Fig. 3 plots the evolution of friction coefficient and depth that may provide more insight in the progression of coating failures during the micro scratch test.

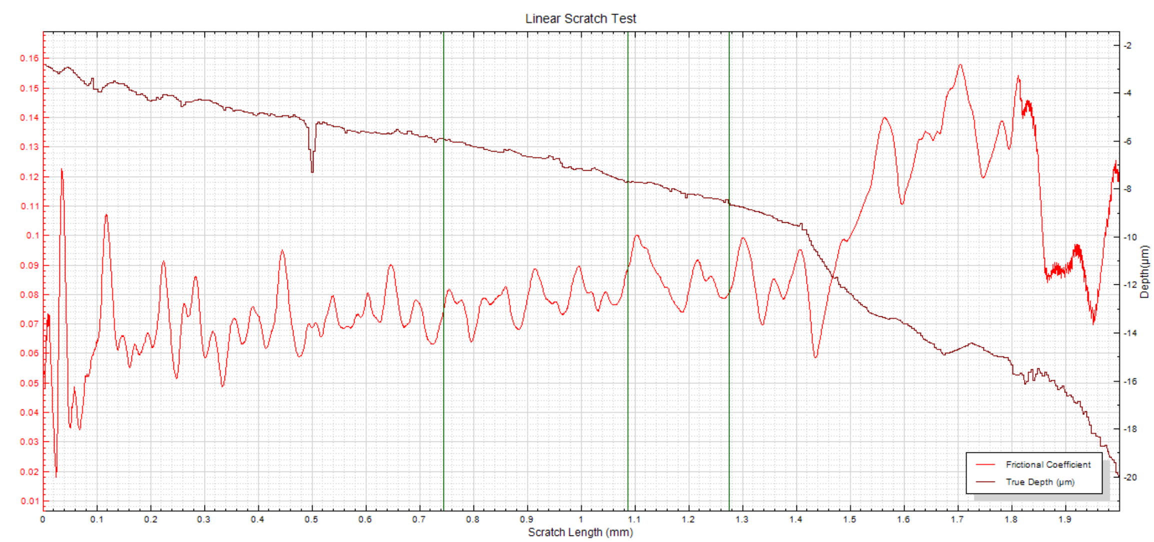


Fig. 3: Evolution of friction coefficient and depth during the micro scratch test.

CONCLUSION

In this study, we showcased that Nanovea Mechanical Tester performs reliable and accurate micro scratch tests on a QCM sample. By applying linearly increased loads in a controlled and closely monitored fashion, the scratch measurement allows users to identify the critical load at which typical cohesive and adhesive coating failure occurs. It provides a superior tool to quantitatively evaluate and compare the intrinsic quality of the coating and the interfacial integrity of the coating/substrate system for QCM.

The Nano, Micro or Macro modules of the Nanovea Mechanical Tester all include ISO and ASTM compliant indentation, scratch and wear tester modes, providing the widest and most user friendly range of testing available in a single system. Nanovea's unmatched range is an ideal solution for determining the full range of mechanical properties of thin or thick, soft or hard coatings, films and substrates, including hardness, Young's modulus, fracture toughness, adhesion, wear resistance and many others.

In addition, optional 3D non-contact profiler and AFM Module are available for high resolution 3D imaging of indentation, scratch and wear track in addition to other surface measurements such as roughness.

To learn more about Nanovea Mechanical Tester or Lab Services.

APPENDIX: MEASUREMENT PRINCIPLE

Principle of scratch test

The scratch testing method is a very reproducible quantitative technique. 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 200 μm) 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 200 μm for micro/macro scratch tests, and from 1 to 20 μm 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 of 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. Fig. 4 illustrates the principle of scratch testing.

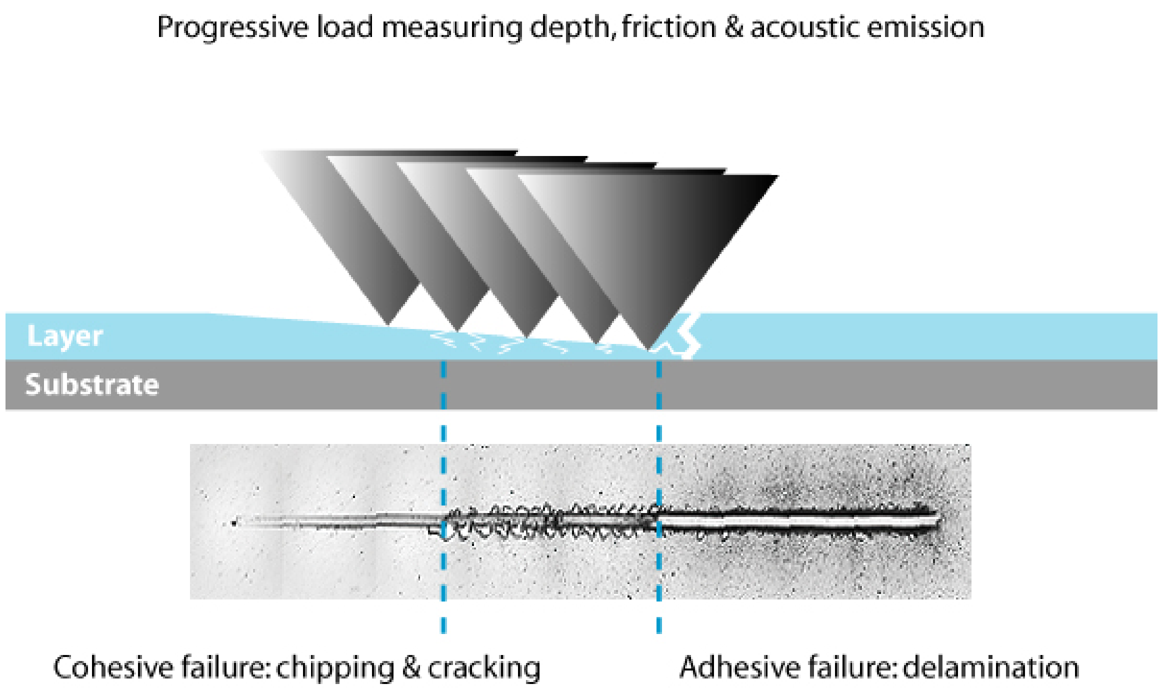


Fig. 1: Principle of scratch testing.

Comments on the critical load

The scratch test gives 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 parameters that determine the critical loads are summarized in Table 2.

Test specific parameters	Sample specific parameters
Loading rate	Friction coefficient between surface and indenter
Scratching speed	Internal stresses in the material for bulk materials
Indenter tip radius	Material hardness & roughness for coating-substrate systems
Indenter material	Substrate hardness and roughness
	Coating hardness and roughness
	Coating thickness

Table 1: List of parameters that determine the critical loads.

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 AFMs can be useful to measure exact depth of scratch after the test.

Other possible measurements by Nanovea Mechanical Tester:
Hardness and Young's Modulus, Stress-Strain & Yield Stress, Fracture Toughness, Compression strength, Fatigue testing and many others.