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MACRO SCRATCH TESTING OF ANODIZED COATINGS

Prepared by

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INTRO:

Anodizing is an electrolytic passivation process commonly applied to convert aluminum to aluminum oxide. It can modify the texture of the surface and changes the microstructure of the metal near the surface. The formation of an anodized aluminum oxide layer can enhance corrosion and wear resistance, improve cosmetic effects, and provide better adhesion for paint primers. Anodized aluminum is widely used on electronic devices, such as mp3 players, flashlights, cameras, & many other products.

IMPORTANCE OF MACRO SCRATCH TESTING FOR QUALITY CONTROL

Anodized aluminum surfaces have a higher hardness than aluminum; however, their wear resistance is moderate due to their porous feature. Anodic films are generally much stronger and more adherent than most types of paint and metal plating, but quality control of such films still possess great significance. For example, when one of the most popular cell phones with a dyed aluminum frame was first introduced in 2012, the anodizing process didn't create sufficient layers, leaving the aluminum frame vulnerable to chip, dent, and scratch. The scratch test allows us to detect premature adhesive/cohesive failure of the anodized aluminum layer in real-life applications.

MEASUREMENT OBJECTIVE

The process of scratching is simulated in a controlled and monitored manner to observe adhesive or cohesive failures. In this application, the Nanovea Mechanical Tester in its macro scratch testing mode (see Fig. 1) is used to measure the load required to cause failure to the anodized aluminum films produced by different processes.

Fig. 1: Image of the indenter tip over the scratches of the tested sample

SCRATCH TESTING PRINCIPLE:

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 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 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.

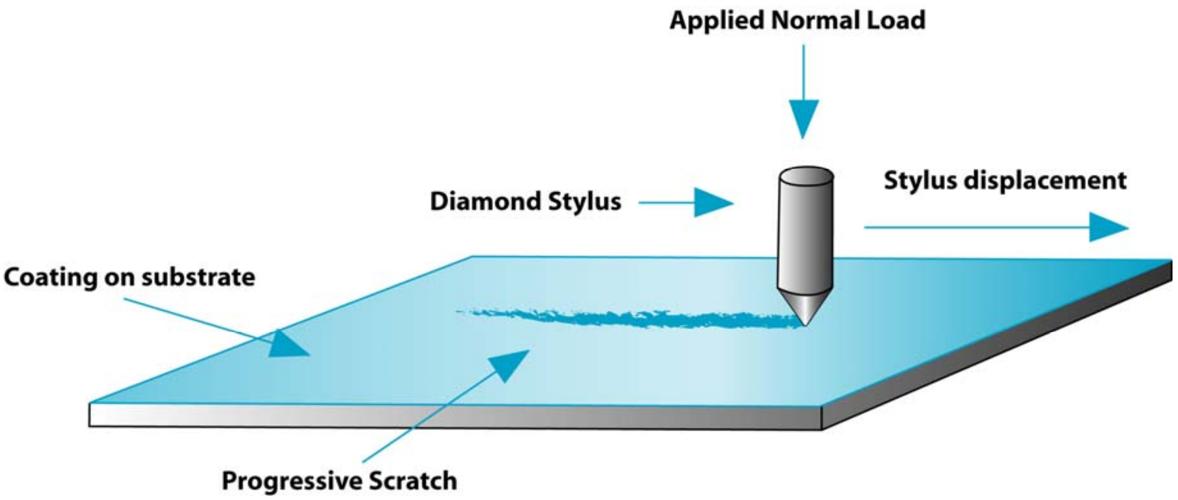

Fig. 2: Principle of scratch testing

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 parameters that determine the critical loads are summarized in Table 1 below.

Test specific parameters	Sample specific parameters
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 & roughness for coating-substrate systems 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.

TEST PARAMETERS:

Nanovea Mechanical Tester equipped with a Rockwell C diamond stylus (200 μm radius) was used to perform progressive load scratch tests on three anodized aluminum samples using Macro Scratch Tester Mode. Three tests were repeated at the same testing conditions on each sample to ensure reproducibility of the results. The test parameters and the tip geometry are shown below.

Load type	Progressive
Initial Load	0.03 N
Final Load	100N & 150N
Loading rate	200 N/min
Scratch Length	10 mm
Scratching speed, dx/dt	20 mm/min
Indenter geometry	120° cone
Indenter material (tip)	Diamond
Indenter tip radius	200 µm

Table 2: Test Parameters

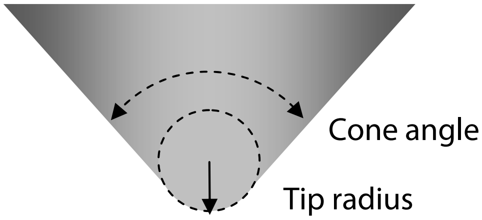

Fig. 3: Sphero-conical indenter.

TEST RESULTS:

Fig. 4, shows the plot of normal force, frictional force and penetration as a function of scratch length for Sample A as an example. An optional acoustic emission module can be installed to provide more information. It is clearly seen that as the normal load progressively increases, the indention tip gradually sinks into the tested sample as reflected by the nearly-linear change of Penetration at the beginning of the test. At a scratch length of ~3.8 mm, which corresponds to a normal load of ~37 N, a sudden change in the slopes of Frictional Force and Penetration curves takes place, followed by fluctuation of these two values. Such a behavior can be used as one of the implications that coating failure starts to occur.

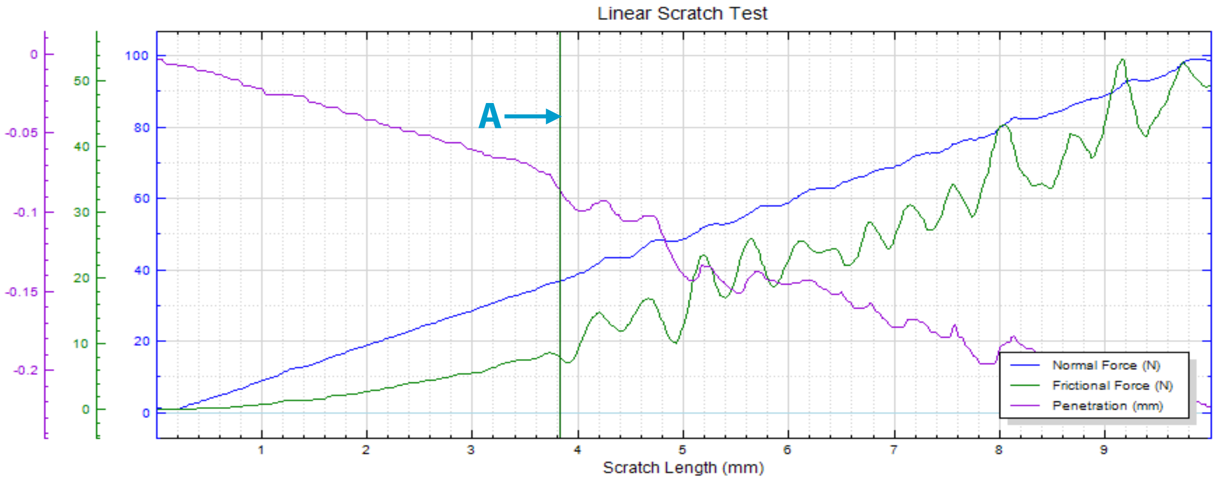

Fig. 4: Normal force, frictional force and penetration as a function of scratch length

The above finding is in agreement with our further optical observation under the microscope as displayed in Fig 5., where clear exposure of the substrate is shown at the correspondent load of ~37 N. Nanovea Mechanical Tester software includes the function that can automatically generate an image of the whole scratch (panorama) and correlate the different areas of the scratch with the load applied. This facilitates the user to perform analysis any time, rather than having to determine the critical load under the microscope immediately after the scratch tests.

Fig. 5: Micrograph of full scratch of Sample A

Table 2, summarizes the scratch test results of all three anodized aluminum samples (Samples A, B and C) produced using different process parameters. It is evident that optimization of the anodization process is critical to the mechanical properties, or more specifically, scratch resistance of the anodized aluminum oxide layer in this case. Sample C possesses substantially enhanced scratch resistance, exhibiting a critical load of 118.9 N, compared with only 37.3 N for Sample A. Moreover, it is demonstrated that Nanovea Mechanical Tester can provide reproducible quantitative analysis on the scratch resistance of anodized coatings: The scratch tests on all three different anodized aluminum oxide coatings show low standard deviations.

Scratch	Critical Load (N)		
	Sample A	Sample B	Sample C
1st	37.2	67.5	118.7
2nd	37.0	68.1	119.8
3rd	37.7	68.1	118.3
Average	37.3	67.9	118.9
Standard Deviation	0.34	0.34	0.78

Table 2: Summary of critical load of the tested samples

CONCLUSION:

Nanovea Mechanical Tester at Macro Scratch Tester Mode is a superior tool for quality control of anodized aluminum oxide coatings. Scratch testing can detect adhesion/cohesion problems in the coating system before parts are actually put to use. By applying loads in a controlled and closely monitored fashion, the tool allows users to identify quantitative and reproducible critical load failures. This type of information can help manufacturers improve and control coating quality. 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 on a single module. 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. Learn More about the Nanovea Mechanical Testers or Lab Services