Microstructure Scratch Resistance
With Nano Scratch Testing

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INTRO

A Microstructure can be made up of a prepared surface or thin layer of material whether metallic, polymeric, ceramic or composite etc. These surfaces are often confined in limited areas which are extremely sensitive. The analysis of their resistance is often a crucial factor. The interest in the quality control of these surfaces is of great interest with applications found in optics, electronics and other micro manufactured parts in which surface properties are essential.

IMPORTANCE OF NANO SCRATCH TESTING FOR QUALITY CONTROL

The physical properties of materials are often affected by the changes related to the micro scale applications. For example, changes in stresses in the materials can affect the final resistance to scratches or dents. This is why the study of the resistance to scratch on microstructures is very critical to many applications. Nano scratch testing is an ideal tool to measure the scratch resistance of microstructures. It is often important for the manufacture to understand and monitor at what level marring can occur.

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 mode is used to measure the load required to cause failure to a microstructure. A 10μm diamond tipped stylus is used at a progressive load ranging from 10 mN to 20 mN to scratch the microstructure. The point where the coating fails by cracking is taken as the point of failure.

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 spherico-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.

<table>
<thead>
<tr>
<th>The test specific parameters include:</th>
<th>The sample specific parameters include:</th>
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</thead>
<tbody>
<tr>
<td>Loading rate</td>
<td>Friction coefficient between surface and indenter</td>
</tr>
<tr>
<td>Scratching speed</td>
<td>Internal stresses in the material</td>
</tr>
<tr>
<td>Indenter tip radius</td>
<td>For bulk materials</td>
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<tr>
<td>Indenter material</td>
<td>Material hardness and roughness</td>
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<tr>
<td></td>
<td>For coating-substrate systems</td>
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<tr>
<td></td>
<td>Substrate hardness and roughness</td>
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<tr>
<td></td>
<td>Coating hardness and roughness</td>
</tr>
<tr>
<td></td>
<td>Coating thickness</td>
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</table>

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

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
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<tbody>
<tr>
<td>Load type</td>
<td>Progressive</td>
</tr>
<tr>
<td>Initial Load</td>
<td>10mN</td>
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<tr>
<td>Final Load</td>
<td>20.00mN</td>
</tr>
<tr>
<td>Loading rate</td>
<td>20.00mN/min</td>
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<tr>
<td>Scratch Length</td>
<td>100microns</td>
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<tr>
<td>Indenter geometry</td>
<td>90° conical</td>
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<tr>
<td>Indenter tip radius</td>
<td>2 μm</td>
</tr>
<tr>
<td>Indenter material (tip)</td>
<td>Diamond</td>
</tr>
</tbody>
</table>

Results

![Graph showing linear scratch test results]
Conclusion

As seen in our analysis, the scratch resistance level of the microstructure has been identified. The coating is prone to cracking and should be further investigated as to how it will relate to intended resistance level and performance. If the adhesion were not strong enough, full delamination or partial delamination would occur at the coating substrate interface. The value of nano scratch testing for this application is the ability to quantify with superior repeatability the scratch resistance of the microstructure. This test simulates either handling of the microstructure or quantifies fracture toughness of the microstructure. The Nanovea Mechanical Tester, during Nano Scratch Tester Mode, is an excellent tool for the quality control of nano coatings on a wide range of substrates. The Failure points obtained can be quantitatively identified to insure a quality control and performance. The same instrument can be used in the indentation and wear modes to study other properties in very limited area of a few microns.