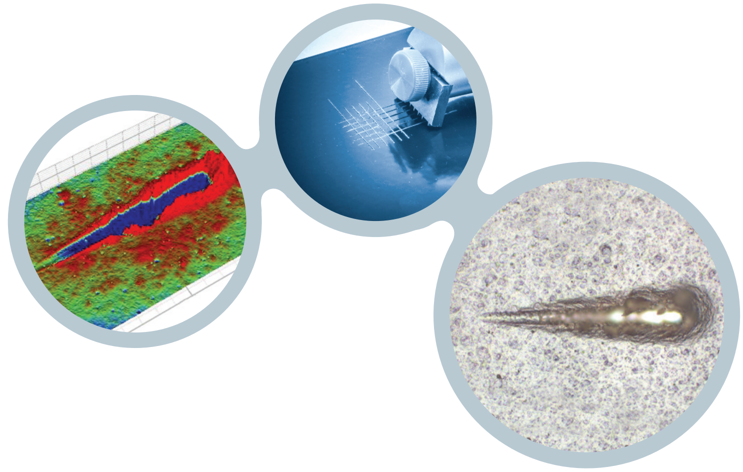

Application Note #1537

Tape Test versus Nanoindentation for Thin Films and Coatings

Depending upon the application, thin films and coatings have many potential testing needs; however, a high level of adhesion to the substrate is probably the most general requirement. Since adhesion is unique to each coating and substrate pairing, and can be further affected by processing routes and introduction of adhesion layers, quantitative and repeatable testing is necessary. A cost-effective technique, called the tape test, has been utilized for a long time. This application note discusses how this method, though, is not very quantitative or repeatable due to uncontrollable errors, and how, in comparison, nanoindenter-based approaches provide a much more reliable solution.

Testing Adhesion

In the tape test for adhesion (according to ASTM standard D3359-09, test method A), a piece of pressure sensitive tape is placed on x-shaped cuts made into the film, and is removed by pulling it off, back on itself at an angle of 180°. This area is then inspected, and its adhesion is rated on a 5-point scale according to the amount of peeled or removed coating material.[1] This method has its place as a cheap, field-performable test, but it produces only qualitative results with potential for significant operator errors. Typical errors might involve specified angles for load application, loading rates, accurate cutting of the film, and even variability in the tape itself.

By comparison, nanoindentation-based scratch adhesion tests can be used to quantitatively evaluate coating adhesion with high load and displacement resolution applied in closed-loop control.[2] Since both normal and lateral axis loads and displacements can be detected, identification of critical events and onset of deformation of the substrate are enabled, for instance. Using these methods, coating adhesion is evaluated based upon critical debonding loads and a corresponding scratch model.

Experimental Setup and Procedure

A Hysitron® TI 980 TriboIndenter® with a Performech® II controller (equipped with 3D OmniProbe® transducer and a 10 µm radius conical diamond probe) was used to measure the coating adhesion of paint samples. The paint samples came from anonymous industry providers, with different surface treatments, and are known to have large variations in real-world performance. All the samples had a similar surface roughness of approximately 0.35 µm.

For the adhesion measurements, ramped load scratch tests reaching a maximum load of 600 mN over a 500 µm scratch length were used to determine the critical load. The critical load is marked by the onset of failure in the film. The Laugier scratch model was used to determine the shear stress at the critical load, which is a measure of coating adhesion to its substrate.[3] It is approximated that the shear stress (τ) at the interface of a coating and its substrate is:

$$\tau = \frac{(\sigma_x a)}{R}$$

where a is half of the scratch width at the critical point, σ_x is the critical stress, R is the radius of the probe used, and $a \approx R$. The critical load from the scratch tests was converted to critical stress via elastic Hertz analysis.

Testing Comparison Results

The onset of failure during the scratch can be identified by a change in the slope of the normal displacement versus the lateral displacement, see Figure 1. The scratch width at the critical point was measured from postmortem imaging using a Bruker ContourGT® optical profiler. This was identified by correlating to the lateral displacement measured during the scratch test. The width was measured at the original surface height as the distance between the inner flanks of the material pile-up. The 3D images from the ContourGT also clearly show material pile-up along the corners of the scratch, see Figure 2.

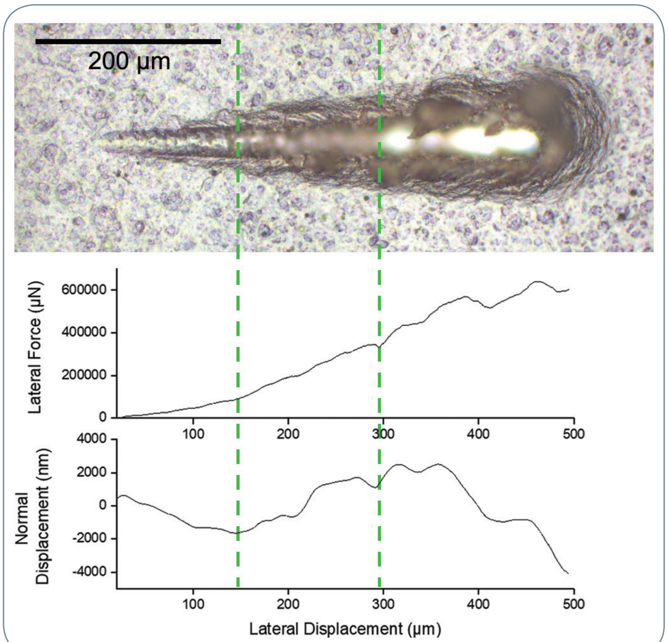

Figure 1. A comparison of optical (top), lateral force (middle) and normal displacement (bottom) ramped load scratch profiles versus the scratch length. The first vertical dashed line shows the critical point for failure, at which the slope of normal displacement changes. The second dashed line denotes the ejection of material from the scratch track and the beginning of deformation in the substrate.

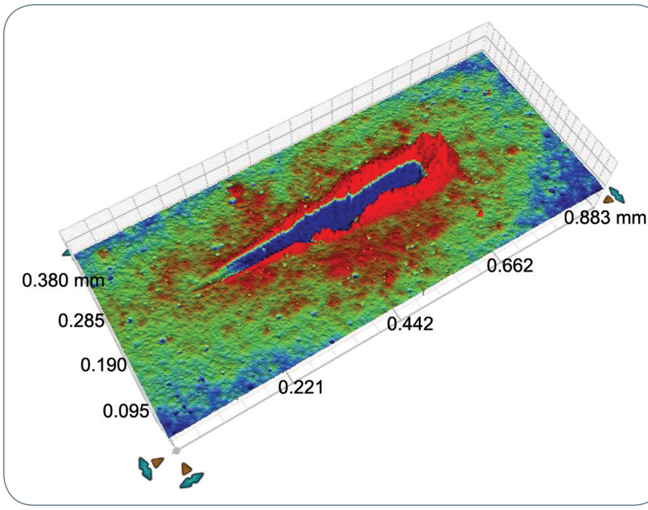

Figure 2. 3D image of the ramped load scratch, showing pile-up in the corners of material ejected from the scratch track.

Using the Laugier scratch model[3], the critical shear stress for delamination was determined for each sample and was used as a measure of coating adhesion. Table 1 summarizes these results and compares them with the tape test results, which show no adhesion loss across all the samples. The crosshatch adhesion loss for all samples was zero, meaning that ASTM crosshatch could not differentiate the process differences.

Sample ID	**% Crosshatch Adhesion Loss** *Tape Test*	**Coating Adhesion (Pa)** *Hysitron TI 980*
1	0	372.7±14.7
2	0	387.2±42.7
3	0	406.4±41.1
1w	0	402.9±21.7
2w	0	435.5±73.6
3w	0	328.0±47.1

Table 1. Tape test adhesion loss versus coating adhesion measurements as determined by ramping load scratch tests.

Conclusions

Scratch tests using Hysitron TI 980 give a clearly quantifiable measure of coating adhesion. This is in comparison to the tape test, which shows the same adhesion measurement for all three surface preparations. Measurements of the quantifiable scratch adhesion can be used for product and process improvement. It is of further interest to know at which interface the initial failure occurs, which is of importance for multilayer coatings, such as the multi-step prime-paint-clearcoat shown here.

References

1. ASTM, *Standard Test Methods for Measuring Adhesion by Tape Test*, ASTM International, 2005.
2. A. G171-03, *Standard Test Method for Scratch hardness of Materials Using a Diamond Stylus*, 2017.
3. M. T. Laugier, *J. Mater. Sci.*, vol. 21, p. 2269, 1986.

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