Scratch Testing of Coatings

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The new ASTM standard for scratch adhesion testing, ASTM C1624-05, covers the determination of the adhesion strength and failure modes of hard thin ceramic coatings on metal or ceramic substrates.

The purpose of this article is to explain the principles and the terminology of the scratch test, along with the highlights of the RSX scratch tester.

Scratch testing

In scratch testing, a diamond stylus of defined geometry is drawn across the surface of a coated sample at a constant speed with a defined normal force over a defined distance. The normal force can be constant, may progressively increase, or may incrementally increase. The diamond stylus typically has a Rockwell C geometry with an angle of 120 degrees and a spherical tip radius of 200 µm (Fig. 1). Different tip radii can serve to change the contact pressure.

The tangential force, the penetration depth, and the acoustic emission signals are recorded as secondary test data, along with the normal force. After completion of the test, the scratch track is microscopically analyzed for specific, well-defined damage such as cracking, deformation, buckling, spallation, or delamination of the coating.

The critical load \( L_c \) is defined as the load that corresponds to the failure event. This load is related to the practical adhesion strength and the damage resistance of the coating/substrate system.

The critical load depends on the test parameters (stylus parameters...
and geometry, loading rate, scratch speed) as well as on the properties of the coated sample (coating thickness, surface roughness and microstructure, damage mechanism, hardness, modulus of elasticity, fracture strength). Figure 2 illustrates the scratch test method.

- In **constant-load** scratch testing, the normal force is maintained at a constant level while scratching the sample. By increasing the load for each subsequent scratch, a scratch map can be generated to determine the critical load corresponding to a specified damage.

- In **progressive-load** scratch testing, the stylus is drawn along the sample while the normal force is linearly increased to a maximum predetermined value. The critical load is recorded as the normal force at which the damage is first observed.

- **Incremental-load** scratch testing consists of incrementally increasing constant load scratch segments, and is very useful if space is limited on the sample. These three different scratch test modes are shown in Fig. 3.

The tangential force $F_t$ is defined as the force that opposes the relative motion between the stylus and the sample. During the scratch test, the tangential force (or frictional force) may change in amplitude as different types of damage develop with increasing load.

The acoustic emission sensor can detect any high-frequency elastic waves generated in the coating/substrate system by brittle damage events such as cracking, delamination, chipping, or buckling.

**The test instrument**

Appropriate test instruments are designed for ease of use for industrial quality control and production lines. Comprehensive scratch test software enables the operator to predefine the measurement protocol. For example, in the Revetest RSX instrument, the protocol is exported by means of a USB memory stick, which is inserted in the front panel of the RSX. After the test is completed, the scratch track can be investigated with the optical microscope attached to the RSX.

With optional tangential force and acoustic emission sensors, it becomes a complete coating characterization station for research facilities. The modular design makes it a good tool for both inexperienced operators and more experienced research personnel. Scratch test protocols can be easily sent to different production plants to maintain an internal test standard within the company.

Test instruments should also provide active feedback of the applied load. This active feedback control allows the measurement of both flat and curved surfaces. The indentation mode should allow conventional Rockwell and Vickers indentation tests to determine the hardness and the Young’s modulus. The available pre-scan and post-scan options enable profiling of the surface before and after the scratch. With pre-scan profiling, the surface roughness and topography are taken into account to get the true penetration depth ($P_{12}$) during scratching. The post-scan profiling provides the residual depth ($R_{12}$) data, which is important for certain materials subjected to viscoelastic relaxation.

**ASTM Standard**

The scratch test is now accepted as the primary quantitative analysis method in coating production, and this
has resulted in a high demand for scratch test equipment. Therefore, a new ASTM standard has been recently developed, based on the RSX machine. This ASTM standard for scratch testing is designated as ASTM C1624, and is entitled Standard Test Method for Adhesion Strength and Mechanical Failure Modes of Ceramic Coatings by Quantitative Single Point Scratch Testing.

This standard explains the principles of the scratch test in detail, along with the limitations of the test, applicability to different coatings, terminology, test methodology, specimen requirements, apparatus requirements, calibration, test procedure, calculations, and requirements for repeatability and reproducibility. A comprehensive bibliography and a scratch atlas describing and illustrating the common damage types are included in the standard. The standard provides a complete and accurate document for helping coating manufacturers develop an in-house quality control scratch test that can screen coated components and evaluate their adhesion.

DLC coating on steel

A typical scratch test example is shown on Fig. 4, along with the sample information and the data set. The sample is a 5 μm-thick diamondlike coating (DLC) on a steel substrate. A progressive load scratch test with a maximum load of 35 N was applied to the sample. The scratch length was 1 mm, and the loading rate was 35 N/min.

The first critical load (Lc1) is 12 N, and it corresponds to the point at which first damage is observed. This first damage has the shape of an interfacial shell-shaped spallation. Note that Lc1 corresponds to the first small jump on the acoustic emission signal, as well as on the friction force curve.

The second critical load (Lc2) is 18 N, and it is the point at which the damage becomes continuous and complete delamination of the coating starts. After this point, all of the acoustic emission, friction force, and penetration depth signals become noisier. These critical points are shown on the scratch track with arrows.

This test method is applicable to a wide range of hard ceramic coating compositions, including carbides, nitrides, oxides, diamond, and diamondlike carbon on ceramic and metal substrates.

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