Designation: C1624 – 05 (Reapproved 2015)

Standard Test Method for Adhesion Strength and Mechanical Failure Modes of Ceramic Coatings by Quantitative Single Point Scratch Testing[1]

This standard is issued under the fixed designation C1624; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the practical adhesion strength and mechanical failure modes of hard (Vickers Hardness HV = 5 GPa or higher), thin (≤30 µm) ceramic coatings on metal and ceramic substrates at ambient temperatures. These ceramic coatings are commonly used for wear/abrasion resistance, oxidation protection, and functional (optical, magnetic, electronic, biological) performance improvement.

1.2 In the test method, a diamond stylus of defined geometry (Rockwell C, a conical diamond indenter with an included angle of 120° and a spherical tip radius of 200 µm) is drawn across the flat surface of a coated test specimen at a constant speed and a defined normal force (constant or progressively increasing) for a defined distance. The damage along the scratch track is microscopically assessed as a function of the applied force. Specific levels of progressive damage are associated with increasing normal stylus forces. The force level(s) which produce a specific type/level of damage in the coating are defined as a critical scratch load(s). The test method also describes the use of tangential force and acoustic emission signals as secondary test data to identify different coating damage levels.

1.3 *Applicability to Coatings*—This test method is applicable to a wide range of hard ceramic coating compositions: carbides, nitrides, oxides, diamond, and diamond-like carbon on ceramic and metal substrates. The test method, as defined with the 200 µm radius diamond stylus, is commonly used for coating thicknesses in the range of 0.1 to 30 µm. Test specimens generally have a planar surface for testing, but cylinder geometries can also be tested with an appropriate fixture.

1.4 *Principal Limitations:*

1.4.1 The test method does not measure the fundamental adhesion strength of the bond between the coating and the substrate. Rather, the test method gives an engineering measurement of the practical (extrinsic) adhesion strength of a coating-substrate system, which depends on the complex interaction of the test parameters (stylus properties and geometry, loading rate, displacement rate, and so forth) and the coating/substrate properties (hardness, fracture strength, modulus of elasticity, damage mechanisms, microstructure, flaw population, surface roughness, and so forth).

1.4.2 The defined test method is not directly applicable to metal or polymeric coatings which fail in a ductile, plastic manner, because plastic deformation mechanisms are very different than the brittle damage modes and features observed in hard ceramic coatings. The test method may be applicable to hard metal coatings which fail in a brittle mode with appropriate changes in test parameters and damage analysis procedures and criteria.

1.4.3 The test method, as defined with the Rockwell C diamond stylus and specific normal force and rate parameters, is not recommended for very thin (<0.1 µm) or thicker coatings (>30 µm). Such coatings may require different stylus geometries, loading rates, and ranges of applied normal force for usable, accurate, repeatable results.

1.4.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard. Test data values in SI units (newtons (N) for force and millimetres (mm) for displacement) are to be considered as standard and are in accordance with IEEE/ASTM SI 10.

1.4.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

1.5 *Organization*—The test method is organized into the following sections:

[1] This test method is under the jurisdiction of ASTM Committee C28 on Advanced Ceramics and is the direct responsibility of Subcommittee C28.04 on Applications.

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2. Referenced Documents

2.1 *ASTM Standards:*[2]

B659 Guide for Measuring Thickness of Metallic and Inorganic Coatings

E4 Practices for Force Verification of Testing Machines

E18 Test Methods for Rockwell Hardness of Metallic Materials

E750 Practice for Characterizing Acoustic Emission Instrumentation

E1316 Terminology for Nondestructive Examinations

E1932 Guide for Acoustic Emission Examination of Small Parts

IEEE/ASTM SI 10 Standard for Use of the International System of Units (SI) (The Modern Metric System)

2.2 *ASME Standard:*[3]

ASME B46.1 Surface Texture (Surface Roughness, Waviness, and Lay)

2.3 *CEN Standard:*[4]

CEN prEN 1071-3 Advanced Technical Ceramics—Methods of Test for Ceramic Coatings—Part 3: Determination Of Adhesive And Other Mechanical Failure Modes By A Scratch Test

3. Terminology

3.1 *Definitions:*

3.1.1 *acoustic emission, n*—class of phenomenon in which elastic waves are generated by the rapid release of energy from localized sources within a material, or the transient waves so generated. **E1316**

3.1.2 *adhesive failure, n*—detachment and separation of a coating from the substrate with cracking and debonding at the coating-substrate interface.

3.1.3 *cohesive failure, n*—material damage and cracking in the coating or in the substrate, separate and distinct from detachment and adhesive debonding at the coating-substrate interface.

3.1.4 *critical scratch load (L_{CN}), n*—applied normal force at which a specific, well-defined, recognizable damage/failure event occurs or is observed in the scratch test of a specific coating on a specific substrate.

3.1.4.1 *Discussion*—The subscript N is used to identify progressive failure events. For example, L_{C1} is often used to identify the first level of cohesive failure in the coating itself; L_{C2} is often used to identify first adhesive failure between the coating and the substrate. Multiple subscripts can be used for progressive levels of distinct damage in a specific coating-substrate systems.

3.1.5 *fundamental adhesion, n*—summation of all interfacial intermolecular interactions between a film or coating and its substrate.

3.1.6 *normal force (L_N), n*—in a scratch test, the force exerted by the stylus, perpendicular to the test surface of the test specimen.

3.1.7 *practical adhesion, n*—force or work required to remove or detach a film or coating from its substrate irrespective of the locus of failure.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American Society of Mechanical Engineers (ASME), ASME International Headquarters, Three Park Ave., New York, NY 10016-5990, www.asme.org.

[4] Available from European Committee for Standardization (CEN), 36 rue de Stassart, B–1050 Brussels, www.cenorm.be.

3.1.7.1 *Discussion*—"Practical adhesion" is a test concept which uses various engineering coating adhesion test methods to obtain a quantitative, reproducible adhesion measurement which can be related to the functional performance of the coating. The practical adhesion is an extrinsic property which depends on the complex interaction of coating/substrate properties and characteristics with the specific test parameters.

3.1.8 *stylus drag coefficient, n*—in scratch testing, the dimensionless ratio of the tangential force to the normal force applied to the stylus at a specific point in the scratch test.

3.1.8.1 *Discussion*—The term stylus drag coefficient is preferred to the more common term scratch coefficient of friction (SCF). The tangential force is primarily a measure of the perpendicular force required to plow the indenter through the coating, rather than to slide it on the surface (sliding friction is a relatively minor contribution to the measured tangential force unless penetration is very small and surface properties dominate). Thus the term friction coefficient is not appropriate for these stylus scratch tests. The SCF term is too easily misunderstood or misused as a measurement of sliding friction.

3.1.9 *tangential force* (L_T)*, n*—force that opposes the relative motion between a moving stylus and the surface that is being scratched by the stylus and which is perpendicular to the normal force exerted by the stylus (also called the friction force, drag force, or the scratching force).

4. Summary of Test Method

4.1 This test consists of producing and assessing controlled damage in a hard ceramic coating by single point scratch action (see Fig. 1). The scratch is developed on a coated test specimen by drawing a diamond stylus of defined geometry and tip size (Rockwell C, 200 µm radius) across the flat surface of the specimen at a constant speed and a controlled and measured normal force (constant or progressively increasing). With increasing applied normal force, the stylus produces progressive mechanical damage in the coating and the substrate through the complex combination of elastic/plastic indentation stresses, frictional forces, and residual internal stresses in the coating/substrate system (Fig. 2).

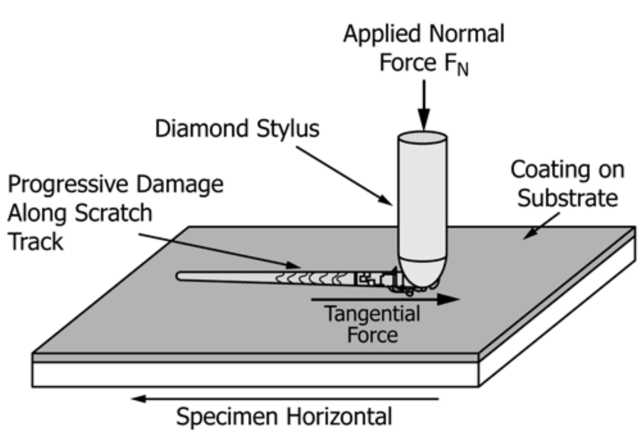


FIG. 1 Test Method Schematic

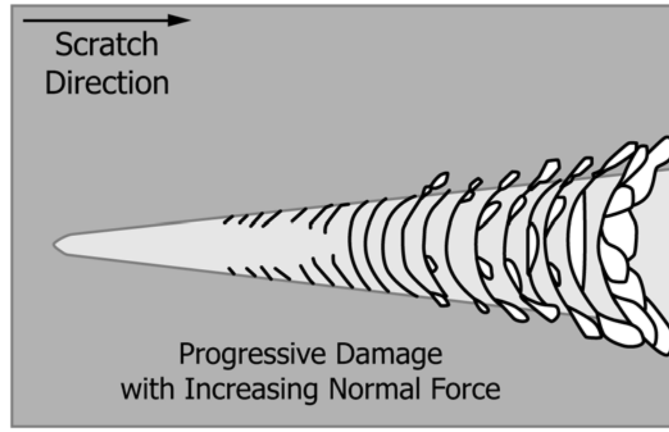


FIG. 2 Schematic Example of Progressive Damage in Scratch Track in a Progressive Load Scratch Test

4.2 The specific levels and types of progressive damage in the scratch track are assessed and associated with the applied normal stylus forces. The normal force which produces a specific, defined, reproducible type/level of damage is defined as a critical scratch load (L_C). For a given coating-substrate system, one or more different critical scratch loads (L_{CN}) can be defined for progressive levels of defined coating damage.

4.3 Coating damage is assessed by optical microscopy or scanning electron microscopy, or both, during or after the scratch test is done. The tangential force and acoustic emission signals can also be measured and recorded during the scratch test process and used as supplementary test data to identify different coating damage levels. In commercial instruments, computerized electronic systems are commonly used to apply, control, measure, and record the force signals and acoustic emission signals and to control the stylus-specimen movement.

4.4 The two primary modes of scratch adhesion testing are constant load and progressive load. In constant load (CL) scratch testing, the normal force on the stylus is maintained at a constant level as the stylus moves in relation to the test specimen surface. Sequential scratch tests are done at increasing force increments to determine the critical scratch load for a given damage level.

NOTE 1—Test systems may have either a movable stage or a movable stylus with the alternate component in a fixed position.

4.5 In progressive load (PL) scratch tests, the applied stylus force is linearly increased to a defined maximum force as the stylus moves in relation to the test specimen surface.

4.6 The critical scratch loads at which a defined coating failure event occurs depend on a complex interaction of coating-substrate properties and test parameters/conditions. It is the purpose of this test standard to: *(1)* describe and define the test equipment and procedures and the major and minor coating-substrate properties which have to be controlled, measured, and understood to produce reliable, comparable coating adhesion test data, and *(2)* define a report format that will provide complete and accurate test data.

5. Significance and Use

5.1 This test is intended to assess the mechanical integrity, failure modes, and practical adhesion strength of a specific hard ceramic coating on a given metal or ceramic substrate. The test method does not measure the fundamental "adhesion strength" of the bond between the coating and the substrate. Rather, the test method gives a quantitative engineering measurement of the practical (extrinsic) adhesion strength and damage resistance of the coating-substrate system as a function of applied normal force. The adhesion strength and damage modes depend on the complex interaction of the coating/substrate properties (hardness, fracture strength, modulus of elasticity, damage mechanisms, microstructure, flaw population, surface roughness, and so forth) and the test parameters (stylus properties and geometry, loading rate, displacement rate, and so forth).

5.2 The quantitative coating adhesion scratch test is a simple, practical, and rapid test. However, reliable and reproducible test results require careful control of the test system configuration and testing parameters, detailed analysis of the coating damage features, and appropriate characterization of the properties and morphology of the coating and the substrate of the test specimens.

5.3 The coating adhesion test has direct application across the full range of coating development, engineering, and production efforts. Measurements of the damage mechanisms in a coating as a function of applied normal forces are useful to understand material-process-property relations; quantify and qualify the mechanical response of coating-substrate systems; assess coating durability; measure production quality; and support failure analysis.

5.4 This test method is applicable to a wide range of hard ceramic coating compositions—carbides, nitrides, oxides, diamond, and diamond like carbon—applied by physical vapor deposition, chemical vapor deposition, and direct oxidation methods to metal and ceramic substrates.

NOTE 2—Under narrow circumstances, the test may be used for ceramic coatings on polymer substrates with due consideration of the differences in elastic modulus, ductility, and strength between the two types of materials. Commonly, the low comparative modulus of the polymer substrate means that the ceramic coating will generally tend to fail in bending (through-thickness adhesive failure) before cohesive failure in the coating itself.

5.5 Ceramic coatings can be crystalline or amorphous, but commonly have high relative density with limited porosity (<5 %). Porous coatings can be tested, but the effects of porosity on the damage mechanisms in the coating must be carefully considered.

5.6 The test method, as defined with the 200 µm radius Rockwell diamond stylus, is commonly used for ceramic coating thicknesses in the range of 0.10 to 30 µm. Thinner coatings may require a smaller diameter stylus and lower normal forces for reliable results. Thicker coatings may require larger diameter stylus and higher normal forces. Any variations in stylus size and geometry and designated normal force ranges shall be reported.

5.7 Specimens commonly have a flat planar surface for testing, but cylinder geometries can also be tested if they are properly fixtured and aligned and the scratch direction is along the long axis of the specimen. The physical size of the test specimen is determined primarily by the capabilities and limits of the test equipment stage and fixturing.

5.8 The test is commonly conducted under unlubricated conditions and at room temperature. However, it is feasible and possible to modify the test equipment and test conditions to conduct the test with lubrication or at elevated temperatures.

5.9 Coated specimens can be tested *after* high temperature, oxidative, or corrosive exposure to assess the retained properties and durability (short-term and long-term) of the coating. Any specimen conditioning or environmental exposure shall be fully documented in the test report, describing in detail the exposure conditions (temperature, atmosphere, pressures, chemistry, humidity, and so forth), the length of time, and resulting changes in coating morphology, composition, and microstructure.

5.10 The test method as described herein is not appropriate for polymer coatings, ductile metal coatings, very thin (<0.1 µm) ceramic coatings, or very thick (>30 µm) ceramic coatings.

6. Test Methodology and Experimental Control

6.1 *Test Overview:*

6.1.1 Coating adhesion is a challenging property to quantify, because the material response to a scratch force is "not a basic property but a response of a system to an applied test condition" (from Blau's *Lab Handbook of Scratch Testing*); but, quantified data are still needed, and the instrumented single point scratch test is the most widely-used test for determining quantitative practical adhesion of coatings.

NOTE 3—Practical adhesion is the force or work required to remove or detach a film or coating from its substrate irrespective of the locus of failure. "Practical adhesion" is a test concept which uses direct engineering test methods to obtain a quantitative, reproducible adhesion measurement which can be related to the functional performance of the coating.

6.1.2 The instrumented single point scratch adhesion test is simple and rapid when performed properly, but it requires a detailed understanding and careful measurement and control of a wide range of specimen characteristics and test parameters for the test is to produce valid, repeatable, and reproducible data (Blau, Bull, Meneve, Mittal, Ichimura, etc.).

6.2 *Test Modes:*

6.2.1 The scratch adhesion test can be done in either of two test modes—constant load (CL) and progressive load (PL). In the CL mode, the normal force on the stylus is maintained at a constant level as the stylus moves at a constant displacement rate in relation to the test specimen surface. Multiple scratch tests are done at increasing force increments (and the same displacement rate) to determine the critical scratch load for a given damage level (Fig. 3). In progressive load (PL) scratch tests, the normal stylus force is linearly increased as the stylus moves at constant displacement rate with respect to the test specimen surface (Fig. 4). [Figs. 3 and 4 plot normal force (constant loads and progressive load) and scratch distance (stylus horizontal movement) against time.]

6.2.2 Table 1 shows relative advantages, disadvantages, and appropriate applications for the two test modes.

6.2.3 The user should choose the test mode which best meets the requirements for data completeness and confidence, specimen characteristics, material supply, and available time.

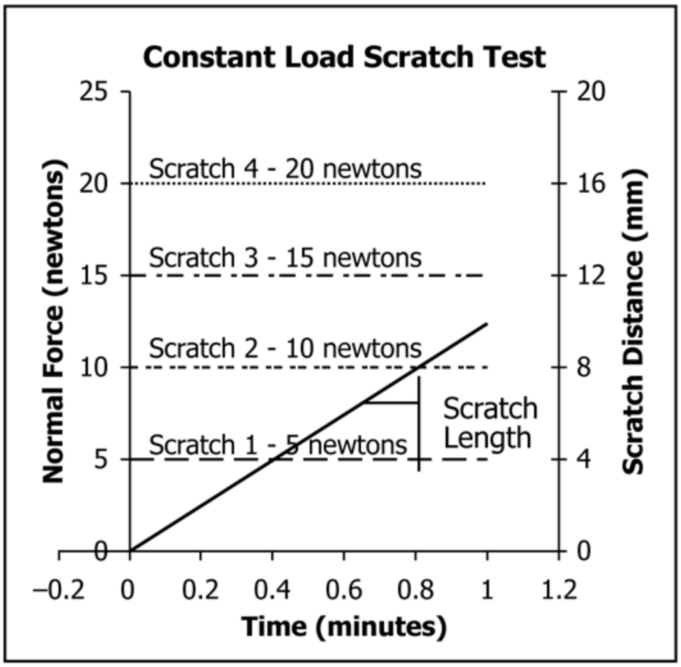


FIG. 3 Constant Load Graph

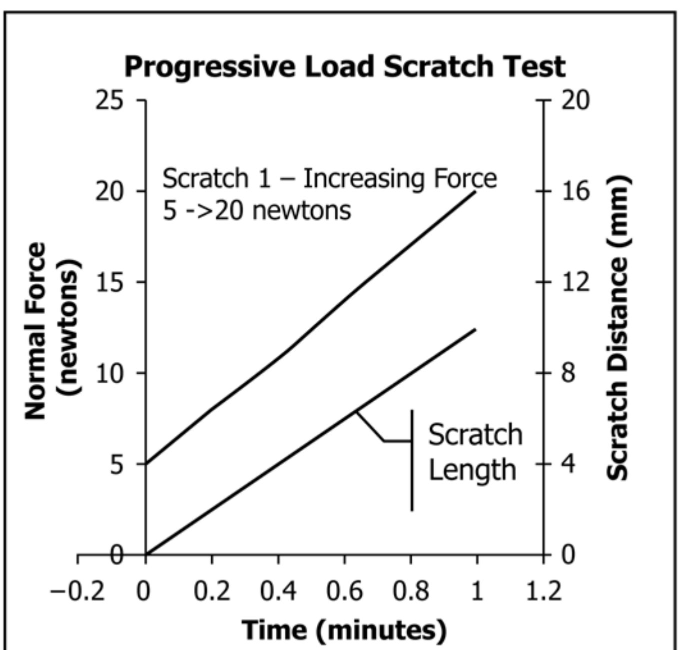


FIG. 4 Progressive Load Graph

In some cases, both test modes may be used for more complete assessment of the coating properties.

6.3 *Primary and Supplemental Measurements:*

6.3.1 *Normal Force and Optical Analysis:*

6.3.1.1 The primary experimental measurements in the scratch adhesion test are the applied normal stylus force and the optical identification/analysis of the damage features in the scratch track. The applied normal force (under constant load or progressive load test modes) is independently controlled and measured during stylus movement. The specific levels and types of progressive damage in the scratch track are optically assessed and directly correlated with the applied normal forces. The force level which produces a specific, defined, reproducible type/level of damage is defined as a critical scratch load (L_C). For a given coating-substrate system, several different critical scratch loads (L_{CN}) can be defined for progressive levels of coating damage (see Fig. 2).

6.3.1.2 Two other experimental measurements are also used as dependent variables in scratch adhesion tests—tangential force and acoustic emission analysis. They can serve as supplemental indicators of coating damage events.

6.3.2 *Tangential Force:*

6.3.2.1 The tangential force on the stylus is the force that opposes the relative motion between a moving stylus and the surface that is being scratched by the stylus and which is perpendicular to the normal force exerted by the stylus (see Fig. 1). That force (L_T) is an indicator of how the stylus and the specimen are interacting through in-plane forces developed by the applied normal force, indenter penetration, and scratch path features. Tangential force generally increases with increasing normal force. (The ratio of tangential force to normal force is the stylus drag coefficient and serves to normalize the tangential force against the applied normal force.)

6.3.2.2 In scratch testing, the tangential force may change in amplitude and shift into a stick-slip character (with more frequent and higher amplitude signal spikes) as different types of damage events occur in the scratch track. The tangential force data are plotted against the applied normal force (Fig. 5) The tangential force may also change through tip damage, from contamination (grease, debris, and so forth) between the stylus and the coating, or from changes in surface roughness along the scratch track.

6.3.2.3 Calculating the stylus drag coefficient for different normal stylus force levels permits the direct comparison of tangential force data done at different normal force levels. Stylus drag coefficient data can be graphed versus time, distance, and normal force and analyzed for the same type of signal variations; stepwise changes in average signal value and significant increases in the frequency and amplitude of signal spikes.

6.3.2.4 Distinct changes in tangential forces and stylus drag coefficient are indications of changes in stylus drag and stress or damage events in the scratch test. However, these changes cannot be associated *a priori* with specific coating damage-failure events without optical analysis to correlate the damage features with the changes in tangential force signals and calculated stylus drag coefficients.

6.3.3 *Acoustic Emission:*

6.3.3.1 Brittle damage events (cracking, delamination, chipping, spalling, buckling, and so forth) can produce high frequency elastic waves in the coating and substrate which can be detected by acoustic emission (AE) systems. As the applied normal force increases in the scratch test, coating damage events occur with increasing frequency and severity and the resulting elastic waves are detected, measured, and recorded by the acoustic emission equipment. The AE data record for each scratch test is analyzed for significant changes in AE signal

TABLE 1 Comparison of Constant Load and Progressive Load Test Modes

	Constant Load (CL) for Each Scratch	Progressive Load (PL) for One Scratch
Advantages	Better discrimination of different damage levels for each incremental loading level. Greater statistical confidence in damage events for a given loading level. Constant load discriminates for coating non-uniformity along the scratch path.	More rapid testing and better specimen utilization, with a single scratch covering a full load range. Progressive force application covers the full range of force without gaps.
Disadvantages	Multiple increment testing requires more specimen area and test time. Incremental loads can miss damage events at intermediate load levels.	Two experimental variables (load and location) changing at the same time. Limited statistical analysis of scratch damage features.
Application	Detailed load specific assessment of coatings (for research, process development, and durability studies) Single value tests are suitable for ″pass-fail″ QA and for assessing coating uniformity.	Screening assessment and QA tests of coatings (for research, process development, and durability studies)

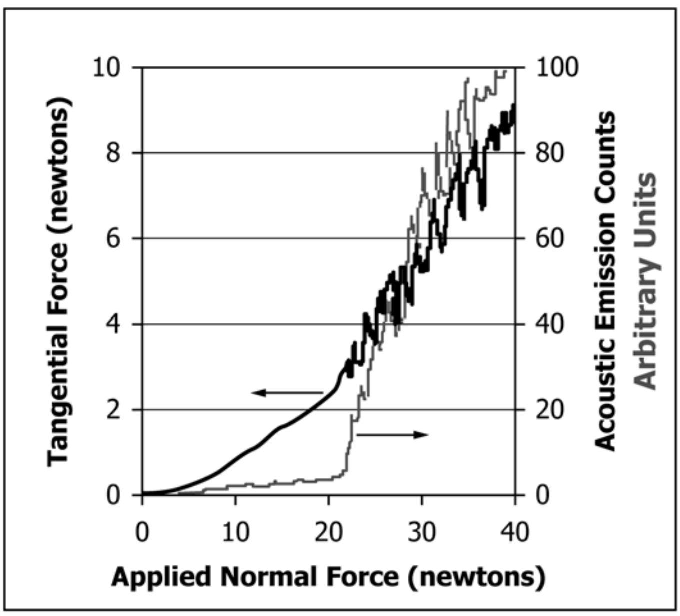


FIG. 5 Tangential Force and Acoustic Emission Versus Applied Normal Force in Progressive Load Test

characteristics (peak amplitude, frequency, event counts, rise-time, signal duration, and energy intensity) that correlate with a given normal stylus force. AE data can be plotted against time, horizontal displacement distance, or normal stylus force (Fig. 5).

6.3.3.2 It should be noted that changes in acoustic emission events at given normal force levels cannot discriminate *a priori* between the different damage events and coating failure modes. Acoustic emission event/signal identification with specific coating failure events requires extensive testing of a given coating system and correlation with the optical analysis of the damage events for that specific coating system.

6.4 *Critical Scratch Load Damage Criteria and Scratch Atlas:*

6.4.1 A primary requirement in using the scratch adhesion test is to clearly identify and categorize the specific coating damage features which are used to define the critical scratch load(s). Since different coating systems can fail with different types of damage, there is no universal set of "critical scratch damage features" that can be applied to all types of coatings.

6.4.2 Appendix X1 gives an overview of typical types of ceramic coating damage mechanisms and a scratch atlas which lists a set of descriptive terms for different types of scratch damage supported by sketches and micrographs. The scratch atlas is not totally comprehensive, but it provides a baseline and framework for users to assess and describe crack damage with a set of generally accepted and understood terms.

6.4.3 Each test user will select the particular levels and classes of coating damage features for a specific coating/substrate system that best meets the coating performance requirements and testing needs. For example, the simplest critical scratch load criteria may be a single level (L_{C1}) at which the first cohesive failure occurs in the coating. A two-level critical scratch load (L_{C1} and L_{C2}) might be defined for cohesive cracking/failure (L_{C1}) in the coating and for subsequent adhesive failure/spalling (L_{C2}) between the coating and the substrate at a higher applied normal force. If necessary, for complete damage mechanism mapping (for research, failure analysis, or durability assessment), multiple (>2 levels) critical scratch loads may be defined to identify each distinctive type of damage feature.

6.4.4 It is critically important to the validity and reproducibility of the scratch test for a given coating-substrate system that the damage events for a given critical scratch load be well defined and described in the test report. This is best done with micrographs and sketches to show the typical damage features of interest. Alternatively, the damage features may be verbally described in the report. Valid comparisons between different test specimens require that they have the same failure/damage mechanisms, which can only be confirmed by optical analysis.

6.5 *Experimental Factors and Variables:*

6.5.1 Appendix X2 provides an overview of the full range of experimental and material variables which have varying degrees of impact in a scratch adhesion test. The different factors can be categorized into six sets of variables: coating variables, substrate variables, interface variables, equipment and procedure variables, specimen variables, and environment variables.

6.5.2 The required depth and detail of specimen characterization and test parameter control will depend on the purpose, scope, and level of confidence and detail required by the user. The experimenter needs to understand and carefully consider how each of these variables can impact a particular test and to what degree each needs to be controlled and measured. This is necessary for the scratch adhesion test is to be used with an acceptable degree of confidence, accuracy, and reliability.

6.5.3 Table 2 lists the test parameters and specimen characteristics that have the *top* priority for control and measurement to ensure acceptable scratch adhesion test results.

6.5.4 Additional test parameters and specimen characteristics may need to be measured and controlled for full analysis and understanding; but, at a minimum, the characteristics and parameters in Table 2 shall be well-controlled and documented to ensure valid and reproducible scratch adhesion test results.

7. Interferences

7.1 The repeatability, reproducibility, and precision in the scratch adhesion test requires that variations in test parameters and specimen characteristics are minimized. As described in Appendix X2, there are many variables that *may* have an impact on the test data and need to be considered to varying degrees. However, the following material and test parameters are the primary source of test interference and need to be understood and controlled.

7.2 *Material and Specimen Related:*

7.2.1 Variations (in individual specimens and between specimens) in the coating thickness and in the surface roughness of the coating are a major source of variability in the critical scratch load values.

7.2.2 Major variations (in specimens and between specimens) in the microstructure, morphology, mechanical properties, and flaw population of the coating may change the damage mechanisms and modes of failure and modify the critical scratch load values.

7.2.3 Contamination and debris on the surface of the coating may interfere with the stylus and increase data variability.

7.3 *Test Method Related:*

7.3.1 Test data are not comparable between specimens and specimen sets unless the scratch adhesion tests are conducted under directly comparable conditions using:

7.3.1.1 Identical styluses (composition, geometry, size, and orientation), and

7.3.1.2 Identical force application rates and horizontal displacement rates.

7.3.2 Stylus damage and contamination will modify the stylus-surface interaction and increase data variability.

7.3.3 The definitions and documentation of the damage criteria for each critical scratch load level for a given coating-substrate shall be clearly defined in complete detail to minimize subjective analysis and improve reproducibility between operators and laboratories.

TABLE 2 Top Priority for Control and Measurement of Specimen Characteristics and Test Parameters

Factor	Details
Diamond Stylus	Verified geometry, size, condition (damage free and clean)
Force and Displacement Control	Accurate calibration, precise and accurate control, measurement, and data recording
Damage Assessment	Optical analysis with well-defined damage criteria and complete documentation with photos/ sketches.
Coating Characterization	Detailed information (by analysis or from coating supplier) on composition, thickness, pedigree, and surface roughness.

8. Apparatus

8.1 *General Description:*

8.1.1 The quantitative scratch adhesion test system commonly consists of six equipment subsystems: *(1)* stylus and stylus mounting, *(2)* mechanical stage and displacement control, *(3)* test frame and force application system, *(4)* force sensors, *(5)* optical measurement, and *(6)* data acquisition/ recording. The test system may also include additional measurement systems, such as acoustic emission and displacement sensors (Fig. 6).

8.1.2 Commercial scratch adhesion test systems are widely available and extensively used. They commonly include computer feedback control of normal force and horizontal displacement, computer data acquisition, and video microscope recording systems.

8.2 *Stylus and Stylus Mounting:*

8.2.1 The stylus shall be a diamond indenter that meets the specifications for a Rockwell sphericonical diamond indenter, as described in 13.1.2.1 of Test Methods E18 and commonly called a Rockwell C diamond indenter. The Rockwell diamond indenter has an apex angle of 120° and terminates in a hemispherical tip with a mean radius of 200 µm (400 µm diameter). Full specifications for the Rockwell C diamond indenter from Test Methods E18 are included in Annex A1. The use of the Rockwell C diamond indenter is specified for this test to ensure comparability and reproducibility of test results within and between laboratories.

NOTE 4—It is recommended that the Rockwell C diamond stylus geometry be definitively checked, verified (SEM, interferometry, profilometry, interference microscopy, and so forth) and documented against specifications by the supplier or by the end user. Significant variations can occur between nominally identical styluses and will have a significant effect on test results.

NOTE 5—If a diamond stylus with smaller or larger tip radius is required and used (for thinner or thicker coatings), the test report shall indicate that a modified version of the standard was used, and the size of the tip radius shall be reported. Scratch test data produced with different stylus geometries, tip radii, or compositions are not directly comparable.

8.2.2 The stylus mounting system shall be designed and constructed to rigidly and securely hold the diamond stylus with a minimum of vertical and horizontal compliance or backlash, given the applied normal and tangential forces.

8.2.3 The diamond stylus shall be secured in a consistent orientation in the mounting holder, either by index marks or alignment flats. This is necessary to eliminate variation between tests caused by spatial variations in the condition, orientation, or shape of the diamond stylus, or a combination thereof, found either in the as-received condition or after accumulated wear from testing.

8.2.4 The diamond stylus shall be microscopically inspected for tip wear and damage and contamination at the beginning of each test series or after ten scratch tests. See 11.4 for a detailed discussion and description of the stylus inspection procedure.

8.3 *Mechanical Stage and Displacement Control System:*

8.3.1 The mechanical stage serves to rigidly secure and accurately align and position the test specimen. Relative

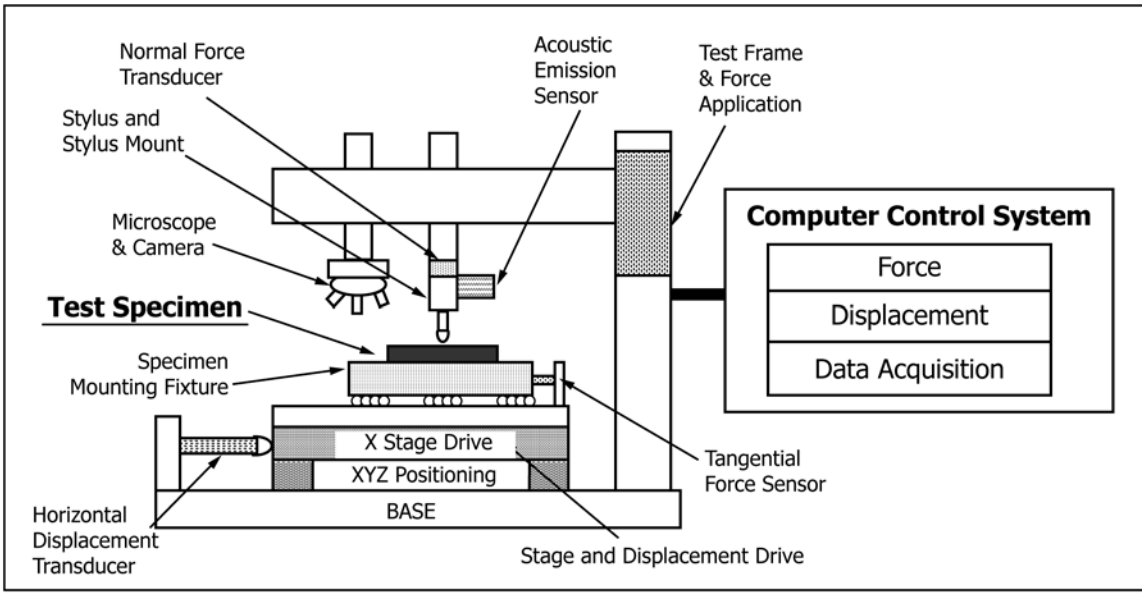


FIG. 6 Scratch Adhesion Test System Schematic

movement between the diamond stylus and the specimen can be produced by either of two methods: *(1)* movement of the mechanical stage with respect to a fixed stylus, or *(2)* movement of the stylus with respect to a fixed stage.

8.3.2 The mounting stage fixture shall be designed and constructed of hard metal (tool steel, stainless steel) to be sufficiently rigid to withstand the normal and lateral forces associated with the scratching action without undue elastic or plastic deflection. The fixture must secure the test specimen so that there is no lateral movement, rocking, or backlash of the specimen during the scratch test. The fixture shall have alignment mechanisms to ensure that the test specimen surface plane (or long axis/test direction for cylinder specimens) can be aligned orthogonal and level with respect to the loading direction of the stylus along the length of a given scratch track (see Section 10 and Annex A2 on stage alignment).

8.3.3 The stage should have 2-axis (*X* and *Y*) manual horizontal adjustment (to position the specimen for scratch testing). Horizontal accuracy (straight-line position) should be 10 µm or better in both the *X* and *Y* directions. The test specimen stage must have vertical axis (*Z*) adjustment (manual or motorized) to raise and lower the specimen (or the stylus) into the test position.

8.3.4 The scratch adhesion test is commonly conducted under unlubricated conditions and at room temperature. However, it is feasible and possible to modify the test equipment and test conditions to conduct the test with lubrication or at cryogenic or elevated temperatures. For elevated temperature (>100°C) testing, test equipment will have to be specially modified to develop and maintain specimen temperature, minimize oxidation and thermal degradation of the test specimens and test equipment, and maintain precise control and accurate measurement of the experimental parameters. Any modifications of the test system or test procedure shall be fully documented in the test report.

NOTE 6—Some commercial test systems now offer temperature-controlled stages for testing specimens across a range of cryogenic and elevated temperatures.

8.3.5 The movement control system shall produce straight-line horizontal movement between the stylus and the specimen at a constant, controlled, and repeatable speed. This controlled horizontal displacement is most easily produced with an electromechanical stage. The range of translation/displacement (scratch length) shall be at least 10 mm. Translational accuracy and repeatability shall be 0.5 % of the minimum displacement range or 50 µm, whichever is smaller. The system shall be capable of a specimen displacement speed of 10 mm/min with an accuracy of ±0.1 mm/min (higher or lower translation speeds, or both, may be necessary for modified tests).

NOTE 7—Current test systems (commercial and in-house built) commonly have a range of displacement motion of 20 to 150 mm and a range of displacement speeds of 10 to 100 mm/min. It is also common in commercial systems for the specimen positioning and stage movement to be feedback controlled by displacement sensors and computer controlled translation motors.

8.3.6 The movement control system shall be calibrated for accuracy and precision in accordance with Annex A2.

8.3.7 The test system may also be instrumented with an independent horizontal displacement sensor to independently measure the specimen horizontal translation as a function of time. The horizontal displacement sensor shall have a resolution and accuracy of 10 µm or 1 % (or better) of the maximum measured translation, whichever is smaller. Current commercial systems commonly have horizontal positioning precisions of 1 µm or better (see Section 10 and Annex A2 for calibration).

8.4 *Test Frame and Force Application System:*

8.4.1 The test frame system (specimen stage, stylus mounting system, and load frame) shall be sufficiently rigid so that the vertical compliance (µm/N) of the system does not significantly affect the application of force to the specimen or the determination of stylus indent depth. A recommended system compliance value is 5 % or less of the compliance of the test specimen.

8.4.2 The force application system shall be designed to apply the desired normal force to the stylus in a controlled and repeatable manner across the full range of stylus vertical and horizontal displacement. The maximum force required will depend on the properties of the specific coating-substrate system being tested, but a force range of 0 to 150 N will be sufficient for most hard coatings tested with the Rockwell C indenter. Force control shall be precise and repeatable to an accuracy of at least 0.5 N or better. Depending on the type of test (constant load or progressive load), the applied force is either held constant or linearly increased during the specimen/stylus translation. For progressive loading, the minimum force application rate shall be 5 N/min.

NOTE 8—Current commercial test systems commonly use a spring loaded cantilever beam load train with a servo motor compressing the spring to control the force. Such systems commonly have a maximum force of 200 N and a range of force application speeds of 0 to 500 N/min. It is also increasingly common for normal force application to be programmed, controlled, and recorded by a computer controlled system with active feedback and control based on force sensors, force-actuators, and electric motors. Specimen and stage translation is also controlled through the same computer system with displacement sensors and electronic motors.

8.5 *Force and Displacement Sensors:*

8.5.1 The unit of force measurement shall be the newton. The test system shall be instrumented with a force sensor to measure and record the normal force on the stylus as a function of time through the full range of applied force with a resolution and accuracy of at least 0.5 % or better of the maximum expected normal force for the coating specimens of interest.

NOTE 9—Current test systems (commercial and in-house built) commonly have force sensors with accuracies of 50 mN or better.

8.5.2 The normal force sensor shall be calibrated in accordance with Section 10 and Annex A2.

8.5.3 The test system may also be instrumented with a tangential force sensor on the stylus or the stage to measure and record the tangential/drag force on the stylus or specimen as a function of time, normal force, or displacement. If so equipped, the tangential force sensor shall have a resolution and accuracy of 1 % or better of the maximum expected tangential force. The sensor shall be calibrated in accordance with Section 10 and Annex A2. If the tangential force is measured, the stylus drag coefficient (tangential force/normal force) can also be calculated.

8.5.4 The unit of displacement measurement shall be the millimetre. It is recommended that the test system be instrumented with an independent horizontal displacement sensor to record the displacement of the specimen relative to the stylus with a resolution and accuracy of 50 µm or better. The horizontal displacement sensor shall be calibrated in accordance with Section 10 and Annex A2.

8.5.5 The test system may also be instrumented with a vertical displacement sensor to measure the vertical movement of the stylus as a function of time or normal force. If the specimen is flat and level, the vertical stylus movement will directly related to stylus penetration into the coating. Stylus penetration may be related to different damage levels. The vertical displacement sensor shall have a resolution and accuracy of 1 % or better of the maximum measured displacement. Current commercial systems commonly have a vertical displacement range of 1 mm and a precision of 10 nm or better. The vertical displacement sensor shall be calibrated in a similar manner as the horizontal displacement sensor.

8.6 *Optical Analysis and Measurement:*

8.6.1 The scratch test method requires a means of optically analyzing the condition of the coating and the damage events along the scratch track. This is commonly done with a reflected light optical microscope having an objective lens with magnification of 5 to 20× and total magnification of 100 to 500×. The actual magnification required will depend on the scale and morphology of the damage features of interest in the scratch track. The optical system shall have sufficient resolution and depth of focus to clearly observe and identify crack damage features on the scale of 5 µm and greater.

NOTE 10—Microscopic examination of the scratch track is mandatory for determining critical scratch load values, because it is the only reliable method of associating a specific damage/failure event with a measured normal force.

NOTE 11—Special optical microscope techniques (oblique illumination, polarized light, differential interference contrast, dark field illumination, in-focus/out-of-focus, and so forth) may be of value in identifying and evaluating smaller, more detailed damage features.

8.6.2 The optical system must be capable of accurately measuring the position of the defined damage along the length of the scratch track in the progressive load test mode. This is most commonly done with a traveling microscope, instrumented so that the distance along the scratch track can be measured to within ±50 µm or better. This optical evaluation is commonly done after the scratch test with a microscope system that is an integral in-line component of the test system. It can also be done on a stand-alone microscope system.

NOTE 12—Many current commercial scratch test systems are instrumented with in-line optical microscopes. The position of the microscope is calibrated with respect to the stylus, so that horizontal position and damage events can be directly correlated with the associated normal force at those event locations. With the in-line optics, the specimen does not have to be removed from the instrument for optical examination. Such microscopes may also have video cameras to display (and record) a real-time image of the scratch features as they are formed.

8.6.3 The optical system shall be calibrated in accordance with Section 10 and Annex A2.

8.6.4 It is strongly recommended that the microscope be fitted with a camera (video or film) to take micrographs of the defined damage features in the scratch track. This is very useful in accurately documenting the type, scope, and degree of coating damage at the different applied loads. The micrographs should be included in the test report. If micrographs are not available, damage shall be described in the test report by reference to Appendix X1 or by drawing representative sketches of the observed damage.

8.6.5 Scanning electron microscopy (post test) may also be used as an imaging tool to characterize the damage events along the scratch path. SEM micrographs should be included in the test report.

8.7 *Data Acquisition and Recording:*

8.7.1 As a minimum, the applied normal force shall be recorded as a function of time and correlated with the displacement distance, either measured directly against time or by calculation from the displacement speed and time. The force data can be recorded by analog chart recorder, but it is preferred to record the data with a digital data acquisition system for ease of later analysis. Recording devices shall be accurate to within 1 % for the total testing system, including readout unit as specified in Practices E4, and shall have a minimum data acquisition rate of 10 Hz with a response of 50 Hz deemed more than sufficient. All data shall be recorded to a precision of at least three significant figures or 0.1 % of the maximum measured value, whichever is more precise.

8.7.2 If the test system has sensors for tangential force and horizontal and vertical displacement, the data should be recorded at the same acquisition rate and comparable accuracy used for the normal force data.

8.7.3 Optical images recorded digitally or photographically shall have sufficient image resolution to accurately show the damage features of interest in the scratch path.

8.8 *Acoustic Emission System (Optional):*

8.8.1 The test system may also be instrumented with an acoustic emission (AE) system to record the elastic waves generated in the coating as a result of the formation and propagation of damage events in the coating under the stylus normal force. These acoustic events commonly occur at frequencies of 10 kHz to 1 MHz.

8.8.2 The acoustic emission system (piezoelectric sensors, preamplifiers, signal processors/filters, counting/recording devices) measures and records the acoustic events (peak amplitude, frequency, rise-time, signal duration, event counts, and energy intensity) that occur during the scratch test procedure. The acoustic system signal conditioning parameters (sensitivity, amplification, bandwidth, amplitude thresholds, frequency gates, and so forth) have to be designed and adjusted to accurately detect and record the high frequency acoustic events associated with scratch testing of a given coating-substrate system. (As background, Appendix X1 of Practice E750 describes the components of an acoustic emission system.)

8.8.3 General guidance on the use of acoustic emission can be found in Guide E1932. Specific instructions on the set-up, calibration, and use of a given acoustic emission system will be found in the manufacturer's operation instructions.

8.9 *Coating Adhesion Reference Specimens (Optional):*

8.9.1 It is useful to use a coating adhesion reference standard to evaluate the accuracy and repeatability of the scratch adhesion test system and assess accumulated wear and damage on a particular diamond stylus. Such a reference standard should be used to check the test system on a regular scheduled basis, depending on the level of usage and the degree of confidence required for the test (see Section 10 and Annex A2).

8.10 *Coating Surface Profilometery (Optional):*

8.10.1 A surface profilometer is useful for measuring the surface roughness and directional character of the coated specimen surface prior to the scratch adhesion test. Quantitative measurement of the surface roughness, waviness, and lay will provide important (but not essential) information for interpreting variations in force data along scratch tracks, between repeated scratch tests, and among different specimens. ASME B46.1 gives detailed guidance on suitable techniques, procedures, and reporting requirements for the measurement of surface texture and geometric irregularities.

8.11 *Data Analysis and Output Software (Optional):*

8.11.1 Commercial test system suppliers are supplementing the scratch adhesion test system with rapid computer data collection capabilities and appropriate software for comprehensive data conditioning, display, analysis, and export. The complete range of experimental data (normal force, tangential force, horizontal displacement, stylus depth penetration, acoustic emission, digital video data, and so forth) can be fully displayed in real time. In addition, the dependent and calculated experimental data can be plotted versus time, distance, and normal force and then statistically analyzed for subtle changes in data amplitude, standard deviation, frequency, and first and second derivatives. The mathematical analysis of the data provides a statistical tool for quantitatively measuring subtle changes in output data as a function of time, distance, and applied normal force.

9. Test Specimens

9.1 *Specimen Requirements:*

9.1.1 The coated test specimens must be representative of the desired coating-substrate configuration and application, considering the full range of coating, substrate, and process variables (see Appendix X2).

9.1.2 The identification and pedigree (source, lot identification, date of production, and so forth) of the test specimens shall be fully described and reported

9.1.3 It is important that the coating be uniform across the surface area of the test specimens. Variations in coating thickness, composition, microstructure, adhesion, and residual stress along the scratch track (or between different scratch tracks) will produce variations in the stress fields and damage progression, and may produce anomalous test results.

9.1.4 The surface morphology of the coating must be suitable for smooth force application along the scratch track and for clear optical identification of the scratch damage features. The coating surface may be unsuitable for scratch testing if its roughness, surface porosity, or surface features are large enough to cause the stylus to skip, bounce, or catch during displacement. The surface will also be unsuitable if the surface features or porosity, or both, mask or confuse the clear optical identification of the progressive critical damage events (cracks, chipping, spalling, and so forth) in the scratch track.

NOTE 13—Surface roughnesses of 1 µm RMS or better are typical in scratch adhesion testing of hard ceramic coatings.

9.1.5 If the as-received surface condition of the specimen is unsuitable for scratch adhesion testing, the coating surface may be ground or polished, or both, in such a way to produce a suitable test surface condition (see 9.6).

9.2 *Specimen Characterization:*

9.2.1 As a minimum, the composition, thickness, and deposition method of the coating and the composition of the substrate must be known and reported, either from producer/supplier information or by independent characterization. Coating thickness can be measured by a range of different techniques (Guide B659), depending on the coating thickness and the physical properties of the coating and the substrate. Different methods include cross-section microscopy (optical or SEM), X-ray fluorescence, magnetic induction, eddy current, ball cratering, and beta backscatter.

9.2.2 The surface roughness of the coating is an important experimental variable that has a direct effect on the stylus drag and the stresses developed within the coating under the applied normal force. For a full and comprehensive understanding of scratch data results, it is recommended that surface roughness be measured for all specimens to determine the character and uniformity of the surface texture on individual specimens and between specimens. See 8.10 on surface profilometry.

9.2.3 It is also recommended to microscopically analyze and photograph the surface of the specimen prior to testing. This documents the morphology and character of the unscratched surface to identify the size, character, distribution, and uniformity of surface features of the coating. Use a magnification level that will show features on the scale of the scratch width. Report pretest characterization results in the final report.

9.2.4 Depending on the purpose of the test (material development, system assessment, quality control, life prediction, failure analysis, and so forth), it may be of value to obtain information from the producer or to independently measure one or more of the following coating-substrate properties:

9.2.4.1 *Coating*—Microhardness, grain size and microstructure, porosity, density, residual stress, anisotropy, spatial uniformity, through-thickness uniformity, batch-lot uniformity, grinding/polishing features.

9.2.4.2 *Substrate*—Surface morphology and roughness, microhardness, grain size and microstructure, porosity, residual stress, anisotropy, spatial uniformity, through-thickness uniformity, batch-lot uniformity, grinding/polishing features.

9.2.4.3 *Coating Variability*—Microstructure and property anisotropy, spatial and through thickness variation in microstructure and properties, batch-to-batch variation.

9.2.4.4 *Coating Process*—Specimen cleaning and preparation, processing time, temperatures, reactant atmospheres, process materials and conditions.

9.2.5 All available coating, substrate, processing, and analysis information shall be included in the test report.

9.3 *Specimen Size*—The test specimen shall be cut to fit the specimen stage and fixturing. Any cutting/sizing operations should avoid or minimize damage to the coating surface to be tested. Cutting procedures shall be documented in the test report.

9.4 *Specimen Flatness and Level:*

9.4.1 The scratch adhesion test requires that the normal force be applied in a controlled and measured manner. Major geometric irregularities (bumps, waviness, pitch, and so forth) along a given scratch track in the coating surface can cause local force anomalies. The actual flatness required for the specimen will depend on the type of force control mechanism used in the test system. Systems with a force feedback control loop can handle moderate surface variations, but the specimen should still be mounted so that the test surface is level and orthogonal to the vertical motion of the stylus.

9.4.2 Test systems without a force feedback control loop will require a flat and level test specimen that will not produce significant force variations during stylus traverse across surface variations. The specimen must be sufficiently flat and level, so that a scratch test with a fixed 10 N force does not produce variations greater than ±0.5 N during stylus traverse. Annex A2 describes the procedures for assessing the level and flatness of the mounted test specimen.

9.5 *Polishing (Optional):*

9.5.1 If the specimen surface is excessively rough or wavy, and prevents the smooth and controlled application of force or confuses the examination and interpretation of the coating damage, it may be necessary to polish the surface to remove the surface roughness and develop an appropriate surface texture. Any polishing procedure should be tailored to the coating composition and microstructure to avoid excessive force, abrasion, or wear to prevent excessive coating loss and anomalous damage (grinding-induced cracks, gross-grinding marks, grain pull-out, anisotropic features, residual stresses, and so forth) to the coating, the interface, or the substrate. Any grinding/polishing steps shall be fully documented for method and means in the test report. Any additional polishing steps shall be fully documented in the test report.

9.6 *Specimen Exposure Conditioning (Optional):*

9.6.1 High temperatures (static and cyclic), oxidation, corrosion, thermal shock, and ambient humidity can/may affect the composition, microstructure, flaw population, and residual stresses in the coating/substrate system with resulting changes in the mechanical properties and failure modes of the coating. The scratch adhesion test can be used to assess the coated test specimens after environmental and exposure conditioning.

9.6.2 The specific environmental and exposure conditioning (time, temperature, conditions, and so forth) will depend on the desired performance environment for the coating-substrate composition of interest. Any conditioning test must be suitably controlled for all the critical experimental factors. After exposure testing, the specimen should also be examined and analyzed for changes in composition, morphology, coating thickness, scale-build up, erosive wear, etc., which may affect the mechanical properties. Any exposure conditioning steps and subsequent analysis methods and results shall be fully documented in the test report.

9.7 *Specimen Cleaning:*

9.7.1 The test specimen shall be clean and free of surface contamination (oil, grease, fingerprints, debris, dust, and so forth) which could affect the applied force, horizontal traverse, and stylus drag coefficient of the stylus on the specimen. Wiping with acetone or other solvent is generally insufficient for removing contamination in a complete and reproducible manner. Ultrasonic cleaning is recommended unless it will produce coating or substrate damage or degradation. Ultrasonically clean the specimens in acetone or ether for 5 min followed by a 5 min ambient temperature air dry equilibration. Document the cleaning procedure in the test report.

9.8 *Specimen Handling, Storage and Protection (Pre- and Post-Test):*

9.8.1 After cleaning, the specimen(s) should be handled with tweezers or gloved hands to prevent contamination. Prior to and after testing, the specimens should be stored in protective envelopes in a dessicator to prevent contamination, surface damage, and moisture exposure. Environmentally sensitive or fragile coatings may require more stringent storage conditions.

10. Calibration

10.1 *System Calibration:*

10.1.1 The accuracy and repeatability of the force, displacement, optical, and acoustic emission measurements in the adhesion test depend upon the regular and accurate alignment and calibration of the test system and the different sensors. Calibration intervals shall be defined for the laboratory and will depend on the frequency of use and the required level of confidence. As a rule of thumb for a regularly used system, the test system should be calibrated monthly, upon anomalous test results, or with any component replacement or major adjustment. Calibration shall be done against independent transducers, traceable against national reference standards where appropriate.

10.1.2 Calibration instructions are included in Annex A2 for specimen-stage alignment and leveling, horizontal displacement sensor and stage calibration, force sensors (normal and tangential, referencing Practices E4), optical system, and the acoustic emission system.

10.2 *Coating Reference Specimens—Internal and Certified:*

10.2.1 Periodic scratch adhesion testing of a reference specimen is a useful technique for verifying the repeatability and accuracy of the scratch test system and assessing diamond stylus condition. This can be done with an internally-produced reference specimen or with a certified reference specimen. The reference specimen shall be tested for scratch adhesion with the standard stylus using a well-defined, consistent test procedure (tip cleaning, preload, loading rate, displacement rate), specified for the certified specimen or defined for the internal reference. A reference test shall consist of five scratches on the specimen to establish a basis for repeatability and data variation.

10.2.2 If available, reference specimens should be tested as part of the scheduled calibration procedure. Reference specimens can also be tested at regular intervals between calibrations as a check of system reproducibility and diamond stylus condition. For example, for a high-usage system, three reference scratch tests could be done at the start of each daily test series.

10.2.3 Annex A2 describes the characteristics, properties, test methods, and calibration procedures for the reference specimens.

10.2.4 As of 2004, there is no U.S.-certified standard reference specimen for scratch adhesion testing of hard ceramic coatings. The European Institute for Reference Materials and Measurements offers a certified reference material for scratch adhesion testing—BCR-692 (diamond-like carbon coating on steel).[5]

11. Test Procedure

11.1 *System Calibration:*

11.1.1 Check the calibration records to ensure that system calibration is current (per laboratory procedures and rules) for all the components of the test system. If calibration is not current for one or more components, run and record the appropriate calibration procedures.

11.2 *Test Mode Selection:*

11.2.1 Two test modes are commonly used for scratch adhesion testing of coatings—progressive loading (PL) and constant loading (CL). Section 6.2 discussed the two test modes and their relative advantages and disadvantages. Choose the test mode that best meets the experimental objectives and assesses the coating-substrate properties within the constraints of time and materials.

NOTE 14—In some cases, both types of test modes may be necessary and useful in fully determining the coating adhesion and mechanical properties of a particular coating-substrate system.

11.3 *Test Planning:*

11.3.1 Based on the properties and geometry of the coating-substrate test coupons, plan and define the specific test parameters that will be used in the scratch adhesion. The first test parameters to be defined are: maximum load (L_{max}) for both PL and CL tests, the preload (L_{min}) for PL tests, and the load increments for CL tests.

NOTE 15—Preliminary PL scratch adhesion tests may be necessary to determine the preload and maximum load levels for new specimens whose coating damage properties are unknown.

11.3.2 The maximum load (L_{max}) should be selected to produce the desired maximum level of coating damage, but

[5] Available from European Commission–Directorate-General Joint Research Centre, Institute for Reference Materials and Measurements, Retiesweg 111, B-2440 Geel, Belgium, www.irmm.jrc.be.

TABLE 3 Standard Scratch Adhesion Test Parameter Values

Test Parameter	Standard Values		Alternate Range
	For $L_{max} < 20$ N	For $L_{max} > 20$ N	
Loading Rate (Progressive Load)	10 N/min	100 N/min	10 to 200 N/min
Loading Increment (Constant Load)	⅕ of L_{max}	⅕ of L_{max}	1 to 100 N
Horizontal Displacement Rate	10 mm/min	10 mm/min	2 to 25 mm/min
Total Scratch Length	≤10 mm	≤10 mm	At least 2 mm, up to 20 mm
Scratch Spacing	At least 1 mm	At least 1 mm	>5× scratch widths

without markedly exceeding that load, which could produce excessive stylus wear. (For example, if the maximum coating damage occurs at approximately 30 N force, then the maximum load (L_{max}) might be set at 40 N. A maximum load greater than 40 N could unnecessarily wear or damage the stylus.)

11.3.3 A minimum preload (L_{min}) for PL tests should be selected to produce a readily identifiable initial indentation point in the coating without visible local damage. A common preload is 5 N for harder (L_{max} >20 N) coatings; a preload of 1 N might be used for softer (L_{max} < 10 N) coatings.

11.3.4 For CL tests, an initial test increment of 20 % of L_{max} is suggested; this gives five scratches (20 %, 40 %, 60 %, and 100 %) to cover the full load range. Smaller or larger increments may also be used, if appropriate and necessary data are needed/generated.

11.3.5 The test parameter values shown in Table 3 should be used as standard values, unless coating-substrate properties or specimen geometry require a change.

11.3.6 For comparability, it is recommended that the standard loading rates (10 or 100 N/min) and the standard displacement rate (10 mm/min) be used as a standard practice and that the scratch length be shortened or lengthened to adjust for the maximum load.

NOTE 16—Different loading and displacement rates can produce different stress application rates within the coating, which may modify the damage mechanisms and affect the critical scratch load levels. For comparison between different coating systems and between batches, it is recommended that the standard loading and displacement rates be used for all specimens.

11.3.7 Alternate loading and displacement rates may be used to assess the effect of different stress application rates, but these experimental variables shall be clearly noted in the test report.

11.3.8 Repeat scratch tests on the same specimen should be offset from the preceding scratch track by at least 1 mm (scratch spacing) to prevent damage and deformation interference from the prior track with the next scratch test. All scratch tracks should be at least 2 mm from the edge of the specimen to avoid edge effects.

11.4 *Stylus Inspection and Cleaning:*

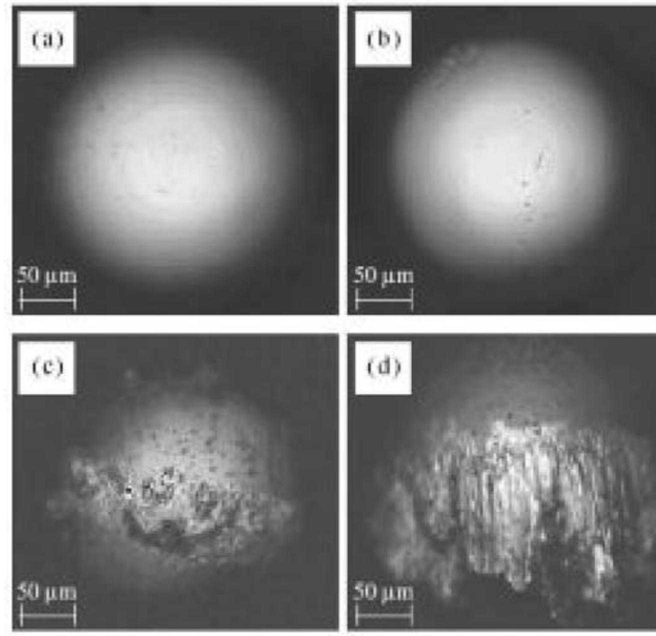
11.4.1 The diamond stylus shall be microscopically inspected for tip wear and damage and contamination at the beginning of each test series or after ten scratch tests.

NOTE 17—If during a scratch test, the tangential force precipitously changes (+ or −) in an anomalous manner or is markedly different from the tangential force in a previous, similar scratch test, it is a presumption of tip contamination or damage. The tip shall be inspected and cleaned/replaced, as necessary.

11.4.2 Remove the stylus from the mount and examine it under the microscope at 200×.

11.4.3 Tip damage is defined as changes in tip geometry (such as chipping, rounding, crater wear, or ring cracking; see Fig. 7) that is observable at 200× or lower magnification by reflected light microscopic examination. If distinct and wide spread tip damage (pitting, cracking, cavitation, and so forth) is observed, the stylus must be replaced.

NOTE 18—In accordance with CEN prEN 1071-3, "uncertainties in the Rockwell C stylus tip shape and manufacturing defects are a major source of error for the scratch test method. The use of an imperfect stylus may result in different values of critical scratch load when the stylus is rotated in the holder. Control of stylus shape is imperative, in the as-received condition, as well as during usage to detect wear at the tip."



NOTE 1—A new undamaged indenter is shown in (a) and a slightly worn (but acceptable) indenter in (b). Note the ring crack damage in (c) and the catastrophically worn tip shown in (d) (from CSM Instruments).

FIG. 7 A Selection of 200 µm Radius Diamond Indenters Viewed Through an Optical Microscope at 200× Magnification

NOTE 19—Many stylus suppliers include an SEM or optical interferometer scan of the stylus tip as a baseline record and confirmation of the as-received condition and integrity of the stylus tip.

11.4.4 Contamination of the tip is defined as a residual debris or film (oil, grease) build-up on the stylus tip from previous tests.

11.4.5 If there is residual debris or film (oil, grease) on the stylus tip, the tip shall be wiped with a soft tissue soaked in ether or acetone. Allow the tip to air dry. If microscopic examination still shows adhered debris, #1200 and #2400 grit SiC abrasive paper can be used to gently remove the debris, followed by the wiping the tip with the ether or acetone tissue. (Ultrasonic cleaning of the stylus should not be used, because of possible cavitation damage to the tip.)

11.4.6 Optical inspection results shall be documented in the test report with a statement of the observed tip condition and geometry, the date and time of inspection, and the type and number of scratch tests performed since the last inspection. Micrographs are also useful for recording tip condition. An alternative technique for recording tip condition is to periodically imprint the stylus into a soft copper coupon with a 2 N load followed by optical examination of the imprint. This is done prior to each test sequence and records the progressive state of tip wear. This type of record is useful in tracking tip condition, explaining anomalous adhesion test results, and understanding tip durability as a function of coating test specimens and test parameters.

11.4.7 After inspection, reinstall, align, and secure the diamond stylus in the mounting system.

11.5 *Environmental Conditions:*

11.5.1 Measure, record, and report the ambient temperature and humidity at the time of the test. An ambient temperature of 20 ± 5°C and relative humidity of 50 % RH ±10 % are recommended, but not required.

11.5.2 If the coated specimens are environmentally sensitive and the test is conducted under modified or specially controlled environmental conditions (temperature, controlled humidity, controlled atmosphere, surface chemistry additions, and so forth), record and report those conditions in full.

11.6 *System Set-Up and Check:*

11.6.1 Turn on the power to the test system and allow all components to equilibrate in accordance with the manufacturer's operating instructions. Check the total system operation and adjust the electronic and mechanical controls for the different test components: force control, displacement control, force and displacement sensors, data recording, optical system, and acoustic emission system.

NOTE 20—Acoustic emission systems require detailed test set up for a full range of sensor signal conditioning factors: amplification, frequency range and response, signal thresholds, frequency filtering and cut-offs, signal-to-noise ratios, and so forth. Refer to the manufacturer's operating manual and to Practice E750 and Guide E1932 for instructions and guidance on acoustic emission set up and operational adjustment.

11.6.2 Set/program the test control system for the defined test variables: horizontal displacement speed and scratch length; load values (L_{min}, L_{max}, and L increments), loading rate (for PL testing), and data collection parameters.

11.7 *Test Specimen Mounting:*

11.7.1 Visually examine the test specimen for cleanliness and, if necessary, reclean and dry in accordance with 9.8. Align and secure the clean test specimen on the specimen stage. If there are directional grinding or polishing marks on the specimen, it is common procedure to orient the stylus displacement direction parallel to the grinding direction on the specimen. Record the orientation (parallel, perpendicular, off-set) of the stylus displacement direction relative to any directional grinding marks.

11.7.2 Check the alignment and level of the mounted specimen in accordance with Appendix X2 and ensure that the stylus is perpendicular to the plane of the specimen.

11.8 *Conducting the Test:*

11.8.1 Select the specific area of the specimen to be scratch tested and position the stage so that the stylus is properly located over the area of interest. Position the stylus at least 2 mm away from the specimen edge to reduce edge effects on the scratch.

11.8.2 *Progressive Load (PL) Test*—Raise the stage (or lower the stylus) to contact the stylus to the specimen. Preload the specimen to L_{min}. Simultaneously start the horizontal displacement, the load progression, and the data collection (including optical recording and acoustic emission, if used). When the maximum load (L_{max}) is reached, stop the stage motion, load progression, and data collection. Unload the stylus. Check that data collection was successful. Prepare for the next scratch test, by moving the stylus at least 1 mm away from the prior scratch.

11.8.3 *Constant Load (CL) Test*—Raise the stage (or lower the stylus) to contact the stylus to the specimen. Load the specimen to the first load increment (L_1). Start the horizontal displacement and the data collection (including optical recording and acoustic emission, if used). When the defined final scratch length is reached, stop the horizontal stage motion and data collection. Unload the stylus. Check that data collection was successful.

11.8.4 Lift the stylus and reposition the specimen for the next scratch test, moving the stylus at least 1 mm away from the previous scratch. Lower the stylus and load the specimen to the next load increment (L_2). Start the horizontal displacement and data collection and complete the scratch test. Repeat the scratch test procedure for each load increment, until the maximum load level is used for the final scratch track.

11.9 *Specimen Count:*

11.9.1 A minimum of five PL tests or five CL test sets is required for calculating a critical scratch load L_{CN} value/s for statistical purposes for each specimen set or condition. Because of the complexity, statistical variation, and qualitative evaluation of damage features in hard coatings, a single PL scratch test or a single test set of CL scratches will not give L_{CN} values with acceptable statistical significance.

11.9.2 However, for PASS-FAIL quality assurance or for simple, qualitative comparisons of coating systems or batches, a single PL test or CL test set may be suitable, depending on coating damage mechanisms and the required confidence and accuracy levels.

11.9.3 Consecutive scratches should be spaced at least 1 mm apart to prevent damage interference between scratches.

11.10 *Invalid and Censored Data:*

11.10.1 Scratch tests are invalid and data shall be discarded if a scratch test is disrupted for any of the following reasons: stylus damage or debris which affects the stylus drag coefficient and geometry of the tip; extraneous contamination or debris on the specimen surface which affects the stylus drag coefficient between the stylus and coating; system malfunctions which change the loading rate or horizontal displacement rate during a test.

11.10.2 Scratch test data should be censored as outliers (but still included in the test report) if individual critical scratch load data are anomalous and correlate with: *(1)* clearly observable isolated variations (pitting, cracking, delaminations, spalling, excessive roughness, and so forth) in the surface texture or the morphology of the coating; and *(2)* severe coating damage (massive chipping, spalling, cracking) at unreasonably low load levels which indicate low cohesive and adhesive strength in a localized area of the coating.

11.11 *Scratch Damage Assessment:*

11.11.1 Scratch damage assessment is commonly done by three methods: optical examination (by microscope or SEM) of the scratch track, monitoring changes in tangential/drag force during the test, and monitoring changes in acoustic emission during test.

11.11.2 Since different coating systems can fail in different manners and at different stresses, a critical requirement in using the scratch adhesion test is to clearly describe the specific, progressive coating damage events/features which are used to define the critical scratch load/s for the coating-substrate system of interest.

11.11.3 The only direct and reliable method of assessing scratch damage remains microscopic and SEM analysis, particularly for low level damage. Changes in tangential force and acoustic emission signals do not easily distinguish between different levels of damage and are not independently reliable indicators. Acoustic emission and tangential force data are supplemental signals which have direct value, if reliable correlations have been established for the specific types of damage/failure events in particular coating system after extensive scratch testing and optical characterization.

11.11.4 *Microscopic Examination of the Scratch Tracks:*

11.11.4.1 Optical examination of the scratch tracks is the primary technique for determining critical scratch load values for progressive damage features in the coating. Analyze the scratch track(s) using a reflected light microscope at a suitable magnification (commonly 100 to 500×) to observe the scratch features of interest. During examination, loosely adhering debris that obscures the scratch track can be removed with a puff of compressed air (from a spray can filled with air for dusting) or with a soft brush, taking care to avoid further damage to the scratch track.

11.11.4.2 Starting at the scratch start point, microscopically examine each scratch track for different damage-failure features and correlate those features with the applied force. Different types of coating-substrate systems will have different modes of failure and different types of damage in the scratch track. Use the terms, descriptions, graphics, and photos in the scratch atlas (Appendix X1) as a tool for identifying and describing the different levels of observed damage. There is an element of subjectivity in describing the damage features, but the use of the scratch atlas will assist the experimenter in using commonly accepted terms to describe the features.

NOTE 21—A stylus indent mark, offset to the side of the start point of the scratch, can be used as a distinct zero marker for scratch tracks that start at very light loads and minimal deformation.

11.11.4.3 The specific progressive failure modes and damage characteristics have to be examined, characterized, and documented for each system and then assigned a specific critical scratch load L_{CN} where N is based on the observed damage features and the degree of detail required, assign a series of sequential critical normal loads to the different damage levels of interest. This may require screening tests on coating-substrate systems whose damage mechanisms have not been characterized.

11.11.4.4 As an example, a PL scratch track schematic is shown in Fig. 8, illustrating different progressive damage events. Based on that schematic, the following critical scratch loads can be defined:

(1) L_{C1} is associated with the start of chevron cracking, indicating cohesive failure in the coating.

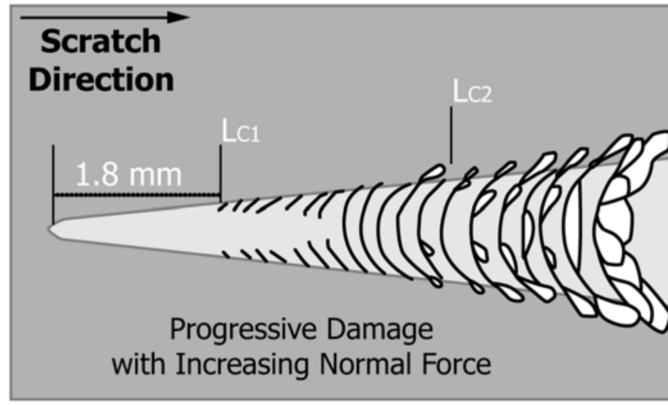


FIG. 8 Critical Scratch Load Damage Features in Progressive Load Test

(2) L_{C2} is associated with the start of chipping failure extending from the arc tensile cracks, indicating adhesive failure between the coating and the substrate.

11.11.5 *Progressive Load Tests:*

11.11.5.1 For PL tests, assess and define the progressive levels of damage in the scratch track, describing and reporting the character, size, shape, and frequency of the progressive damage events. Use the traveling microscope to measure the distance along the scratch axis from the scratch start point to the start point of each cluster of damage features of interest.

NOTE 22—It is commonly accepted procedure to ignore single, isolated damage features and to consider clusters of specific failure and damage events as the valid indicators of force-induced damage.

11.11.5.2 Knowing the rate of force application and the horizontal displacement rate, correlate the specific damage location/cluster with the applied normal stylus force at that point. For example (see Fig. 8), if the start point of the first cluster of chevron cracks (L_{C1}) occurs 1.8 mm from the scratch start point and the applied normal stylus force increased at a rate of 100 N/min with a preload of 5 N and the displacement rate is 10 mm/min, then the critical scratch load L_{C1}:

$$L_{C1} = 100\ \text{N/min} \cdot 1.8\ \text{mm}/(10\ \text{mm/min}) + 5\ \text{N} = 23\ \text{N}$$

11.11.6 *Continuous Load Tests:*

11.11.6.1 For CL tests, each scratch track is associated with a specific constant stylus normal force. Examine the scratch track and describe and report the damage features (character, size, shape, frequency) associated with the specific applied normal force for that scratch track. Since the applied force is constant over the length of the scratch, it is not necessary to measure the start point of the damage cluster and calculate a force for a specific position on the scratch length.

11.11.7 *SEM:*

11.11.7.1 Scanning electron microscope analysis of the scratch tracks is useful to evaluate the damage features at a much finer level of detail. The SEM analysis can also support the microstructure analysis with tools such as energy dispersive X-ray (EDX) analysis or back-scattered mode.

11.11.7.2 Clear and complete descriptions of the different damage features associated with each critical scratch load shall be included in the test report. It is strongly recommended that micrographs be taken of the different scratch damage features and also be included in the report to support the written description.

11.11.8 *Tangential Force and Stylus Drag Coefficient Analysis:*

11.11.8.1 See 6.3 for a discussion of the significance and use of tangential force and stylus drag coefficient data in the scratch adhesion test. Tangential force and stylus drag coefficient can be plotted against time, horizontal displacement distance, or normal stylus force.

11.11.9 *CL Scratch Tests:*

11.11.9.1 In CL tests, examine the tangential force signal data for each incremental scratch track, looking for two types of features in the record/graph of tangential force versus time/distance for the tests done at different normal stylus force levels: *(1)* the average value of the tangential force during the scratch test versus the normal force, and *(2)* the character (frequency and amplitude) of the signal noise or spikes versus the normal force.

11.11.9.2 Calculating the stylus drag coefficient for different normal force levels permits the direct comparison of data for CL scratch tracks done at different force levels (however, there is no normalization for higher bearing surface area based on deeper stylus penetration). CL test stylus drag coefficient data are graphed versus time or distance and are analyzed for the same type of features described for the tangential force data: average signal value against normal force and the character (frequency and amplitude) of the signal noise or spikes versus the normal force.

11.11.10 *PL Scratch Tests:*

11.11.10.1 In PL tests, tangential force data are commonly graphed against the normal stylus force. The analysis of the graph is complicated by the linear increase in tangential force, as the normal force is increased and the stylus digs into the coating. However, the upward sloping data line may still show changes in the slope of tangential force data and changes in the character (frequency and amplitude) of the signal noise or spikes (see Fig. 5).

NOTE 23—In both CL and PL tests, marked step changes in amplitude or signal noise in a single scratch record can also indicate sudden tip damage or contamination build-up.

11.11.10.2 Calculating the stylus drag coefficient for PL tests reduces to some degree the effect of increasing stylus normal force during the PL test; however, there is no normalization for higher bearing surface area based on deeper stylus penetration. PL test stylus drag coefficient data are graphed against applied normal force and are analyzed for the same type of features described for the tangential force data: average signal value against normal force and the character (frequency and amplitude) of the signal noise or spikes versus the normal force.

11.11.10.3 Describe and record significant changes in the tangential force data record and stylus drag coefficient data record for each scratch test as the changes correlate with the specific damage classes for the coating-substrate system and the associated normal stylus force. Those correlations shall be established by direct optical analysis of the scratch tracks or by demonstrated historical correlation with scratch data on the same system using the same test parameters.

11.11.11 *Acoustic Emission:*

11.11.11.1 As the applied normal force increases in the scratch adhesion test, failure events occur in the coating with increasing frequency and severity. These discrete events produce the elastic waves which are measured and recorded by the acoustic emission equipment. AE data can be plotted against time, horizontal displacement distance, or normal stylus force for CL and PL tests (see 6.3).

11.11.11.2 The analysis of AE data for scratch adhesion is similar to the analysis of the tangential force data record. The AE data record for each scratch test is studied looking for significant changes in AE signal characteristics (peak amplitude, frequency, event counts, rise-time, signal duration and energy intensity) correlated with a specific normal stylus force.

11.11.11.3 Describe and record significant changes in the acoustic emission data record for each scratch test as they correlate with the specific damage classes for the coating-substrate system and the normal stylus force. As with the analysis of tangential force and stylus drag coefficient data, those AE data correlations shall be established by direct optical analysis of the scratch tracks or by demonstrated historical correlation with scratch data on the same coating/substrate system using the same scratch adhesion test parameters.

12. Calculations

12.1 The Critical Scratch Load (L_{CN}) for a given type of damage (Level N) is determined by correlation of a specific type of defined damage event with the normal stylus force that produced those damage events.

12.2 For a constant load test, the critical scratch load for is defined by the constant normal force used in that particular scratch test.

12.3 For a progressive load test, the critical scratch load is calculated by correlating the location of the defined damage with the normal stylus force at that point.

$$L_{CN} = [L_{rate} \cdot (l_N / X_{rate})] + L_{start} \tag{1}$$

where:

L_{CN} = the critical scratch load in N for a defined type of damage (N = number sequence),

L_{rate} = the rate of force application (N/min) in the specific scratch test,

l_N = the distance in mm between the start of the scratch track and the start point of the defined type of damage in the scratch track,

X_{rate} = the rate of horizontal displacement (mm/min) in the specific scratch test, and

L_{start} = the preload stylus force in newtons established at the start of the scratch test.

12.4 Stylus drag coefficient (DSC) is the nondimensional normalized ratio of the tangential force to the normal force applied to the stylus at a specific point in the scratch test.

$$D_{SC} = L_T / L_N \tag{2}$$

where:

D_{SC} = the stylus drag coefficient,
L_T = the tangential force at a given point in the scratch test, and
L_N = the normal stylus force at a given point in the scratch test.

12.5 The statistical mean, standard deviation, and coefficient of variation of the critical scratch load are calculated using valid data and standard statistical formulas.

$$\text{Mean} = \bar{x} = \frac{\sum_{i=1}^{n} x_i}{n} \quad (3)$$

$$\text{Standard deviation} = s.d. = \sqrt{\frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1}} \quad (4)$$

$$\text{Percent coefficient of variation} = \%C.V. = \frac{100(s.d.)}{\bar{x}} \quad (5)$$

where:

x_i = the valid measured value, and
n = the number of valid tests.

13. Report

13.1 Report the following information in the written test report:

13.2 *Test Identification:*

13.2.1 Report identification number, test date, location/address, operator, test identification number.

13.2.2 A reference to this ASTM test method "Determined in accordance with ASTM C1624."

13.3 *Specimen Information:*

13.3.1 *Pedigree of the Test Specimen*—Coating identification, supplier fabrication source, material and lot ID, date of manufacture.

13.3.2 *The Coating-Substrate System Description:*

13.3.2.1 Coating composition, thickness, and method of manufacture.

13.3.2.2 Substrate composition and identification.

13.3.3 Report, if available, coating surface roughness and flatness/planarity and method of measurement, coating fabrication parameters, coating grain structure, coating porosity, observed anisotropy and residual stresses, visible surface flaws and defects and their uniformity, substrate roughness.

13.3.4 Report, if available, any other microstructure, mechanical, or thermal property data for the coating or the substrate obtained by specimen analysis and testing or from manufacturer's specifications.

13.3.5 Report, if available, micrographs of the as-prepared coating surface and the coating-substrate cross-section to show microstructure, interface structure and grain size.

13.3.6 *Specimen Conditioning and Environmental Exposure*—Purpose, time, temperature, atmosphere, chemistry, and so forth, supported by analytical or testing results after exposure.

13.3.7 *Specimen Preparation Description*—Cutting, grinding, polishing, cleaning, and storage methods for the specimens.

13.4 *Test Equipment and Procedure Information:*

13.4.1 *Test System Description*—Manufacturer, model identification, and a description of major components and utilized capabilities. Report any modifications of the test system.

13.4.2 *Description of the Stylus*—Type (Standard = Rockwell C diamond), composition, source, tip radius (micrometres), geometry.

13.4.3 *Stylus Inspection*—Method of inspection, inspection results by physical description or micrographs, or both, the date and time of inspection, and the type and number of scratch tests performed since the last inspection.

13.4.4 Date, method, and results of last calibration of all system components.

13.4.5 The equipment, method, and magnification used for optical assessment of the damage.

13.4.6 The equipment, method, and parameters used for data collection.

13.4.7 Report, if measured and recorded, how tangential force was measured and recorded and if stylus drag coefficient was calculated and recorded.

13.4.8 Report, if measured and recorded, description of the AE equipment, the signal processing and conditioning factors, and the AE data characteristics that were analyzed.

13.4.9 *Test Environment Conditions*—Ambient temperature, relative humidity, and any lubrication or exposure liquid on the specimen. If the stage is environment controlled, report the temperature, relative humidity, and atmosphere composition.

13.4.10 The test mode: constant load or progressive load.

13.4.11 For constant load tests, report the maximum normal force/load and force/load increments for each scratch, the horizontal displacement rate, and the scratch length.

13.4.12 For progressive load tests, report the preload force, the maximum normal force/load, the rate of force application, the horizontal traverse rate and the scratch length.

13.4.13 The nominal distance between individual scratch tests on a given specimen.

13.4.14 If there are directional grinding or polishing marks on the specimen, report the orientation of the stylus displacement direction to the grinding direction.

13.4.15 Describe any significant changes or deviations in equipment or test procedure from the standard test method.

13.5 *Test Data and Statistics:*

13.5.1 The number (N) of defined critical scratch load levels (L_{C1}, L_{C2}, … L_{CN}) and a description of the failure/damage features defined for each critical scratch load level. Provide sketches or micrographs to illustrate the distinctive shape, character, and size of the damage features at each level.

13.5.2 The total number of specimens tested and the total number of scratch tests for each specimen, used for statistical calculations.

13.5.3 The critical scratch load (L_{CN}) data (to 3 significant figures) for each valid PL scratch test (or each valid CL scratch test set) on a test specimen, based on optical evaluation of the scratch tracks.

13.5.4 Calculate and report the mean, standard deviation, coefficient of variation, and number of valid tests the L_{CN} data in each test set.

13.5.5 The individual *censored* critical scratch load (L_{CN}) and a description of what specific experimental or specimen factors produced the censored result.

13.5.6 The number of *invalid* scratch tests and the factors that produced the invalid data.

13.5.7 If available, the analysis of the tangential force data and the stylus drag coefficient data as it correlates with the defined damage features and the corresponding critical scratch loads. Provide representative graphs showing the tangential force and stylus drag coefficient data against the normal stylus force.

13.5.8 If available, the analysis of the acoustic emission (AE) data as it correlates with the defined damage features and the corresponding critical scratch loads. Provide representative graphs showing the AE data and data features of interest against the normal stylus force.

13.5.9 Document any observations, interpretation, or conclusions on the test methods, specimens, damage features, data results, or data analysis.

14. Precision and Bias

14.1 *Precision*—The precision of scratch adhesion critical scratch load values is dependent on the mechanical properties, physical characteristics, and uniformity of the specific coating/substrate system under test and the specific test parameters (stylus composition, geometry, and condition; applied force range and loading rate, displacement rate, and so forth). The damage features produced in the scratch adhesion test must be optically analyzed and classified, and there is a subjective factor in that analysis between specimens and between different operators. For these reasons, it is not possible to state in absolute terms the precision for this test method.

14.2 *Bias*—Since there is currently no widely accepted reference material for scratch adhesion tests, bias cannot be currently determined.

14.3 *Repeatability and Reproducibility*—The repeatability and reproducibility of scratch adhesion testing are dependent on the same variables (uniformity of specimens, repeatability of test procedures and parameters, analytical subjectivity) that affect the absolute precision of this test method.

14.3.1 There have been studies done on repeatability and reproducibility of scratch adhesion testing of hard ceramic coatings. Annex A3 describes the results of three published studies on repeatability and reproducibility of scratch adhesion tests on different coatings by different laboratories, and the results are representative of the current level of repeatability and reproducibility in scratch adhesion testing.

15. Keywords

15.1 adhesion test; ceramic coating; critical scratch load; hard coatings; scratch adhesion; scratch test

ANNEXES

(Mandatory Information)

A1. ROCKWELL DIAMOND INDENTER SPECIFICATIONS

NOTE A1.1—The following information are the specifications for the Rockwell Diamond Indenter (used for Rockwell C Hardness testing) in accordance with Section 13.1.2.1 of Test Methods E18.

A1.1 Diamond Indenter

A1.1.1 The diamond indenter shall be free from surface defects (cracks, chips, pits, and so forth) and polished to such an extent that no unpolished part of its surface makes contact with the test piece when the indenter penetrates to a depth of 0.3 mm and 0.2 mm for Rockwell superficial hardness testing.

A1.1.2 The verification of the shape of the indenter can be made by direct measurement or by measurement of its projection on a screen. The verification shall be made at not less than four approximately equally spaced sections.

A1.1.3 The diamond indenter shall have an included angle of 120 ± 0.35°.

A1.1.4 The angle between the axis of the indenter holder (normal to the seating surface) shall not exceed 0.5°.

A1.1.5 The spherical tip of the diamond shall have a mean radius of 0.200 ± 0.010 mm. In each measured section, the radius shall not exceed 0.200 ± 0.0010 mm and local deviations from a true radius shall not exceed 0.002 mm. The surfaces of the cone and spherical tip shall blend in a truly tangential manner.

A1.1.6 Requirement for hardness test verification by means of a performance test. See 13.1.2.1.6.

A2. ALIGNMENT AND CALIBRATION

A2.1 Introduction

A2.1.1 The precision, repeatability, and reproducibility of the scratch adhesion test require that the force, displacement, and analysis measurements are accurate and correct. This annex describes the mandatory procedures for calibration of the apparatus stage, force sensors, displacement sensors, optical instrumentation, and acoustic instrumentation. It also provides instructions on the use of reference specimens for instrument verification.

A2.1.2 The calibration procedures for sensors and measurement devices compare the outputs of the experimental transducers, controllers, and recording devices to certified instruments, which are traceably calibrated to national standards.

A2.1.3 Sensors and controllers shall be calibrated as installed in the test system, not by removing them and calibrating them independently.

A2.1.4 Calibration of the system shall be checked on a regular schedule. Calibration intervals shall be defined for the laboratory and will depend on the frequency of use and the required level of confidence. As a rule of thumb for a regularly used system, the test system should be calibrated monthly, upon anomalous test results, or with any component replacement or major adjustment.

A2.1.5 Calibration results shall be fully documented as a historical record of system operation, accuracy, and repeatability.

A2.2 Specimen-Stage Alignment and Leveling

A2.2.1 For every test, the mounted test specimen must be level and orthogonal to the diamond stylus for each scratch track (~10 mm long), in particular if the force application system does not have feedback control.

A2.2.2 This requires that the specimen is flat and level on the scale of the scratch track and that the mounting stage can be adjusted for *z*-height and for *x-y* tilt to be level and orthogonal with respect to the diamond stylus.

A2.2.3 Mount the test specimen securely and adjust the stage so that the specimen area of interest is level with respect to the stylus. Check the level by one of two methods:

A2.2.3.1 Perform a scratch test at a constant normal force of 10 N at a traverse rate of 10 mm/min with a scratch length of 10 mm. Record the normal force for the full length of the scratch test. The test surface is considered sufficiently flat and level, if the measured normal force is uniform along the length of the scratch and does not deviate (increase, decrease, or spike) by more the ±0.5 N from the initial 10 N applied force.

A2.2.3.2 Alternatively, the specimen level can be checked with the in-line optical system, by examining the specimen area of interest (at least 10 mm in length) at a magnification of at least 200×. If the 10 mm+ area remains in focus at 200× (or greater), the specimen is considered sufficiently flat and level.

A2.3 Horizontal Displacement Sensors and Stage Calibration

A2.3.1 The horizontal displacement control of the stage shall be checked and calibrated for the positional accuracy and traverse speed. This is done by two possible methods:

A2.3.2 *Independent Displacement Transducer:*

A2.3.2.1 Mount an independent calibrated displacement transducer in line and co-linear with the system horizontal transducer. Establish a zero displacement position, and use the horizontal traverse control to incrementally displace the stage over the full range of measurement of the system transducer. A minimum of twenty increments along the maximum displacement shall be made. Record the output of the calibrated transducer and the system transducer at each incremental step to an resolution of 25 µm or better. Repeat the calibration cycle four times and discard the first set of measurements.

A2.3.2.2 Convert the data from the two transducers into horizontal displacement data and perform a linear least squares regression on the system data versus the calibrated data for the three sets of data. Calculate and record the scaling factor and the offset factor for each data set. Determine the repeatability among the three data sets and determine a final scaling factor and offset factor for the horizontal displacement transducer. Use that scaling factor and offset factor for signal conditioning or for direct calculation to provide accurate horizontal displacement data.

A2.3.2.3 Displacement rate/speed is calibrated by measuring controlled stage displacement at the test displacement rate of interest (commonly 10 mm/min) over a defined and measured period of time (1 min) with the independent calibrated displacement transducer and comparing it to the data from the already calibrated system transducer. Calculate the displacement velocity from the 1 minute displacement measured from the independent transducer and compare that to the nominal displacement rate. The measured rate shall be accurate to ±0.10 mm/min. Adjust the displacement control system to produce the desired speed. The displacement speed shall be checked with four calibration runs to check for repeatability.

A2.3.3 *Optical Scratch Measurement:*

A2.3.3.1 If an independent displacement transducer is not available, the displacement system can be calibrated by measuring scratch lengths. Make a series of four scratches at a constant normal force (10 N) at four scratch lengths (2.5 mm, 5 mm, 7.5 mm, and 10 mm) on a hard steel or ceramic specimen (properly leveled) while recording the displacement with the system transducer. Optically measure the total length of each scratch and calculate an actual scratch length (measured length minus the scratch width; see Fig. A2.1). Compare the actual scratch length to the displacement recorded from the system transducer. If necessary, calculate and use a scaling factor and offset factor for signal conditioning or for direct calculation to provide accurate horizontal displacement data.

A2.3.4 The same scratch analysis technique can be used for verification of the displacement rate. Make four scratches at a

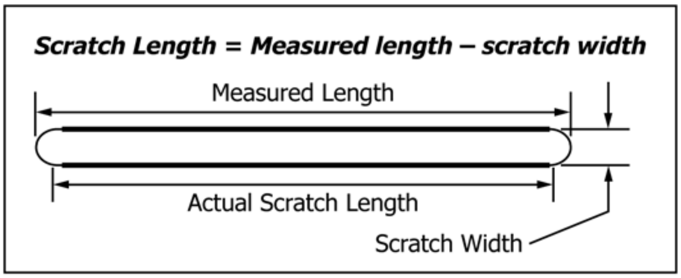


FIG. A2.1 Schematic for Scratch Length Measurement

fixed normal force (commonly 10 N) at the selected test displacement rate (commonly 10 mm/min) over a defined and measured period of time (1 min). Then optically measure each scratch length and calculate an actual scratch length (measured scratch length minus the scratch width; see Fig. A2.1) to establish a displacement distance and calculate a true displacement rate/speed. Adjust the displacement control system to produce the desired rate/speed. The displacement speed shall be checked with four calibration runs to check for repeatability.

A2.4 Force Sensor Calibration (Normal and Tangential)

A2.4.1 The force sensors (normal and tangential) shall be checked and calibrated for accuracy using independent transducers (traceably calibrated against national reference standards where appropriate) and the procedures in accordance with Practices E4.

A2.4.2 *Normal Force Sensor:*

A2.4.2.1 Mount an independent calibrated force transducer (load cell) in line and co-axial with the normal force system transducer (load cell). Mount a hard steel plate as a calibration plate. Establish a zero force level, and use the force control to incrementally apply the normal force over the full range of applied force (commonly 100 N). A minimum of twenty increments up to the maximum load shall be made. Record the output of the calibrated transducer and the system transducer at each incremental step to a resolution of 0.5 N or better. Repeat the calibration cycle four times and discard the first set of measurements.

A2.4.2.2 Convert the data from the two transducers into force data and perform a linear least squares regression on the system data versus the calibrated data for the three sets of data. Calculate and record the scaling factor and the offset factor for each data set. Determine the repeatability among the three data sets and determine a final scaling factor and offset factor for the normal force transducer. Use that scaling factor and offset factor for signal conditioning or for direct calculation to provide accurate normal force data.

A2.4.3 *Tangential Force Sensor:*

A2.4.3.1 The tangential force sensor is also calibrated with an independent calibrated force transducer. Mount the calibrated transducer in line and co-axial with the tangential force system transducer (load cell).

A2.4.3.2 The preferred method for calibration is to use an independent force application system. The mechanical stage is blocked to prevent movement and a tangential force is applied to the stage by an independent mechanism (thumb screw pushrod, pulley system, and so forth). The tangential force is directly applied to the stage in increments and the outputs of the calibrated transducer and the system transducer are recorded. This method gives the most controlled application of tangential force.

A2.4.3.3 Alternatively, the tangential force can be developed by mounting a steel specimen on the stage, applying the stylus with a constant normal load and horizontally displacing the stage at a fixed rate, while monitoring the output of the calibrated and the system transducers. Higher tangential forces are produced by running the calibration at successively higher normal forces. However, this method produces the tangential forces indirectly with limited control of the developed tangential force.

A2.4.3.4 In both calibration methods, the outputs of the two transducers are recorded at each force increment at a resolution of 0.5 N. The tangential force is calibrated and recorded at a minimum of 20 increments of the maximum applied force. Repeat the calibration cycle four times and discard the first set of measurements.

A2.4.3.5 The tangential force calibration data are converted and analyzed in the same manner used for the normal force calibration, producing and using signal scaling and offset factors for data accuracy.

A2.5 Optical System Calibration

A2.5.1 The optical system commonly uses a traveling microscope to measure the distance along the scratch track from the scratch start point to the selected damage feature. The traveling microscope is calibrated by two possible methods. In the first method, a given amount of travel produced by the micrometer mechanism is measured and checked by an independent calibrated micrometer mounted in the system. In the second method, the system microscope is used to measure the known length of calibrated feature (for example, a series of different length grooves) on a calibrated optical reference specimen. The optical measurement system for scratch length shall be accurate to 1 % of the scratch length or ±10 µm, whichever is better.

A2.6 Acoustic Emission Calibration

A2.6.1 The acoustical emission system shall be checked and calibrated in accordance with the manufacturer's operational instructions to ensure that the system components (sensors, preamplifiers, amplifiers, signal processors, data recorders, and so forth) are properly operating.

A2.7 Scratch Adhesion Reference Specimens—Internal and Certified

A2.7.1 If a reference specimen (internal or certified) is available, it shall be tested as a regular part of the scheduled calibration procedure in accordance with a defined and appropriate scratch test method. An internally-produced reference specimen shall consist of an appropriate ceramic coating deposited on a well-defined, carefully prepared substrate by a well-controlled, repeatable deposition method that produces a uniform and reproducible composition, thickness, microstructure, and adherence. If possible, the reference specimen should be representative of the type of coatings commonly tested in the laboratory (such as a TiN coating on tool steel). The results of the reference specimen calibration tests shall be documented as a historical record of system operation and precision.

A2.7.2 The reference specimen shall be tested for scratch adhesion with the standard stylus using a well-defined, consistent test procedure (tip cleaning, preload, loading rate, displacement rate, analysis procedure, and so forth) specified for the certified specimen or defined for the internal reference. A reference specimen calibration test shall consist of five scratches on the reference specimen.

A2.7.3 The internal reference specimen shall be initially tested with ten scratches in accordance with the defined testing procedure to establish baseline critical scratch load values (L_{Cn}) for the different coating damage levels. The reference specimen tests shall be documented with a full description of the test specimens, the defined testing procedure, the baseline critical scratch load values, and damage level descriptions. It is also useful to document the damage levels/features of the internal reference specimen by micrographs.

A2.7.4 Alternatively, a certified reference specimen can also be used to assess system accuracy and reproducibility. In that case, perform the scratch adhesion test in the manner defined for the certified reference specimen.

A2.7.5 The results of the scratch adhesion tests on reference specimens shall be documented in the calibration record.

A3. REPEATABILITY AND REPRODUCIBILITY STUDIES

A3.1 The following three studies give a current picture of the repeatability and reproducibility levels which have been achieved in scratch adhesion tests of different ceramic coatings on different substrates. References are listed in detail in the document bibliography.

A3.2 Blau (*Lab Handbook of Scratch Testing*) cites a 1990 paper by Ronkainen, et. al. where three hard coatings (3 to 8 µm thick) were tested in three laboratories with a Rockwell C indenter scratch test, considering lower and upper critical scratch based on tangential force traces and optical examination. The number of scratches for each test set is not referenced by Blau.

A3.3 Meneve et.al. report on a European EC SMT 1995 interlaboratory round robin comparison on scratch adhesion testing of a TiN coating on AISI M2 steel. The test used detailed calibration and measurement guidelines, instructions for critical scratch determination, and a detailed report format. One coated specimen was sent to each of 12 laboratories. Each laboratory performed at least ten scratches on the submitted specimen. The L_{C2} adhesive failure critical scratch loads are listed in Table A3.2 with mean and 95 % confidence ranges. After the completion of the round robin, all 12 specimens were scratch tested at one laboratory. The mean L_{C2} value for all 12 specimens was 19 N, as compared to the intralaboratory value of 26 N. The variability within and between laboratory data sets was attributed primarily to imperfect scratch styli among different laboratories. The critical conclusions from the study were:

A3.3.1 Stylus contamination and stylus wear/damage must be minimized.

A3.3.2 "Acoustic emission data and stylus drag force data should not be used as stand-alone criteria for critical load values. Microscopic observation remains the most reliable means of associating a failure event with a measured normal load."

A3.4 Aldritch-Smith et al reported on a 2001-2002 VAMAS round robin evaluation of adhesion testing of a chromium nitride coating on AISI 304 stainless steel. Three different critical scratch values were defined and measured. The critical scratch values were defined as forward chevron cracks (L_{C1}), cohesive spalling on the edges (L_{C2}), and interfacial spalling (L_{C3}). Adhesion was measured at nine different laboratories with extensive instructions on calibration with a certified reference material, test procedures, and damage criteria. The coating adhesion was varied by depositing a gold-palladium interlayer at three thickness levels: 0, 25, and 50 nm. A review of the data confirms that there is significant variation between laboratories in terms of both the mean values and the standard deviations. Among the nine labs there were two laboratories that could be considered outliers. Removing those two outliers (1 high and 1 low) markedly improves the standard deviation of the intralaboratory data and brings it closer to the interlaboratory statistics, as shown in Table A3.3. The major conclusions of the study were:

A3.4.1 Coating thickness variation between specimens was a possible source of variability.

A3.4.2 Acoustic emission data and tangential force data should not be used as stand-alone criteria for critical load values. Microscopic observation remains the most reliable means of assessing clearly defined levels of coating damage.

TABLE A3.1 Lower and Upper Critical Scratch Loads (Ronkainen et al)—Mean and S.D.

NOTE 1—L_{C1} = cohesive failure; L_{C2} = adhesive failure

NOTE 2—From Blau, *Lab Handbook of Scratch Testing*, page 7.10.

Coating	Lab A—Mean and SD		Lab B—Mean and SD		Lab C—Mean and SD		Range among Labs	
	L_{C1} (N)	L_{C2} (N)	L_{C1} (N)	L_{C2} (N)	L_{C1} (N)	L_{C2} (N)	L_{C1} (N)	L_{C2} (N)
4.6 µm thick TiB2 by EB PVD	45 ± 3	91 ± 3	47 ± 3	95 ± 5	51 ± 9	>96	6	5
8.5 µm thick TiB2 by magnetron sputter	46 ± 5	60 ±10	57 ± 4	62 ± 2	59 ± 3	69 ± 9	13	9
2.7 µm Ti-Al-N by EB PVD	46 ± 2	57 ± 2	40 ± 10	75 ± 5	61 ± 2	78 ± 7	15	21

TABLE A3.2 L_{C2} Values for TiN on AISI M2 Steel (Ten scratches minimum)

NOTE 1—Mean and 95 % Confidence, from Meneve et. al.

NOTE 2—L_{C2} Mean Value for all Laboratories = 26 N.

Lab 1	Lab 2	Lab 3	Lab 4	Lab 5	Lab 6
17 ± 1.5	24 ± 2	30 ± 8	32 ± 3	32 ± 5	8 ± 2
Lab 7	Lab 8	Lab 9	Lab 10	Lab 11	Lab 12
38 ± 4	26 ± 3	17 ± 3	34 ± 3	24 ± 2	38 ± 3.5

TABLE A3.3 Aldrich Smith Report—CrN on AISA 304 Stainless Steel

	L_{C1} Data	L_{C2} Data	L_{C3} Data
Mean for All Labs	3.15 N	5.97 N	11.28 N
SD of Mean for All Labs	2.50 N	2.25 N	1.92 N
CV of Mean for All Labs (Intralab)	79 %	38 %	17 %
Mean CV for All Labs (Interlab)	22 %	10 %	10 %
Mean for 7 Labs w/o 2 Outliers	2.53 N	5.92 N	11.36 N
SD of Mean for 7 Labs w/o 2 Outliers	0.81 N	1.12 N	1.00 N
CV of Mean for 7 Labs w/o 2 Outliers (Intralab)	32 %	19 %	9 %
Mean CV for 7 Labs w/o 2 Outliers (Interlab)	26 %	9 %	8 %

APPENDIXES

(Nonmandatory Information)

X1. COATING DAMAGE CRITERIA AND SCRATCH ATLAS

X1.1 Coating Damage Definitions and Criteria

X1.1.1 In scratch adhesion testing, one of the most difficult experimental challenges is the clear definition and classification of the specific levels and features of the progressive damage. Since different coatings have different modes of damage and failure (which themselves can depend on thickness, interfacial bonding, test method, and test conditions), there is no universal, absolute definition/description of the coating damage modes. Rather, there is a subjective factor in describing and assessing coating damage. However, there is a need for a general framework that gives the community a common set of baseline terms to describe the different damage features.

X1.1.2 A number of coating researchers (Bull, Larsson, Hedenqvist) have laid out general descriptions and categories of coating damage which are useful for organizing the types of observed damage. Bull's classification system is clear and comprehensive. He defines four major categories of scratch damage and maps them as a function of the ratio of the quasi-static hardnesses of the coating and the substrate (see Fig. X1.1).

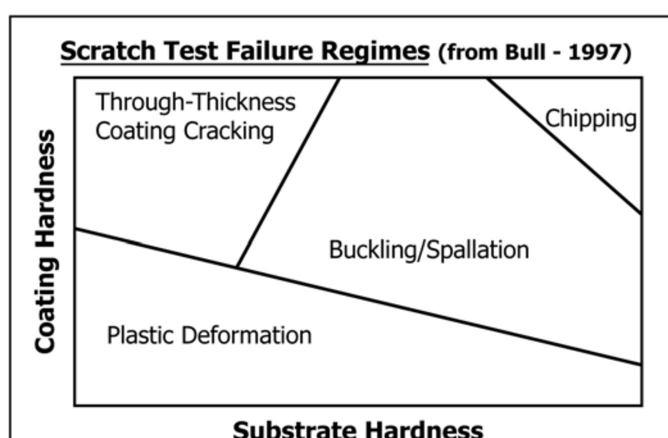


FIG. X1.1 Scratch Test Failure Regimes (from Bull, 1997)

X1.1.3 If coatings and substrates can be generalized into ductile and brittle categories, then the general failure mechanisms for the four combinations of coatings and substrates are described in Table X1.1.

TABLE X1.1 Failure Mechanisms in Different Coating-Substrate Combinations

	Brittle Substrate	Ductile Substrate
Brittle Coating	Tensile cracking in the coating followed by spalling and chipping of both the coating and the substrate.	Tensile and Hertzian cracks in the coating progressing to chipping and spallation of the coating as the substrate is deformed.
Ductile Coating	Coating plastic deformation and conformal cracking, followed by spalling and buckling failure in the coating as the substrate cracks.	Combined plastic deformation of the coating and the substrate producing tensile and conformal cracking with predominant buckling failure of the coating.

X1.1.4 Plastic deformation is seen in adhesion scratch testing of some thin, ceramic coatings, but it is not commonly used to define a critical scratch load for ceramic coatings. Rather, the three other modes (through thickness cracking, buckling/spallation, and chipping) are the defining phenomena for critical scratch load definition.

X1.1.5 Through-thickness cracking of the coating is a cohesive failure mode that normally occurs at lower applied forces than adhesive failures. It generally occurs by tensile stresses in the coating behind the stylus. In adhesive failures the coating separates from the substrate either by cracking and lifting (buckling) or by full separation (spalling and chipping). This commonly occurs under compressive stresses. The specific type of damage that occurs for a given coating-substrate system depends on the properties (thickness, hardness, etc) of the coating.

X1.1.6 Bull also describes different coating damage modes for brittle coatings based on stress state, substrate properties, and interface bonding strength as shown in Table X1.2.

X1.1.7 Bull's damage category system is referenced in many papers on scratch adhesion testing of thin, hard coatings. The Bull system provides a suitable frame work for organizing and categorizing damage criteria that are specific to a given coating-substrate system. Table X1.3 (derived from Bull and Blau) categorizes and describes the different damage features which are commonly seen in testing of hard ceramic coatings.

X1.1.8 Often these different damage features will be observed together at particular locations in the scratch track. The user should use these terms and definitions to identify and classify the damage features of interest in his/her scratch adhesion tests of particular coating-substrate systems.

X1.2 Scratch Atlas

X1.2.1 The scratch atlas shown in Table X1.4 provides a framework for analyzing and describing common crack damage features seen in the failure of hard coatings. It gives a set of terms based on Bull's table with illustrative drawings and representative micrographs.

X1.2.2 It is common in scratch adhesion testing to see multiple types of damage features occurring close together, reflecting local variations in coating properties and morphology. Mixed damage modes should be described to the fullest extent possible.

X1.2.3 This scratch atlas is not comprehensive enough to include every type of damage feature seen in scratch testing, but it does provide a basis for common terminology and interpretation. It will be expanded over time with input from the coating industry.

TABLE X1.2 Categories of Different Damage Modes in Brittle Coatings (from Bull, 1991)

Stress State	Coating	Substrate	Interface Bonding	Damage Modes—Failure Mechanisms
Tensile	Brittle	Ductile	Good	Coating Cracking (No Interface Failure)
			Poor	Coating Cracking With Interface Failure
Compressive	Brittle	Ductile	Good	Buckle Cracking In Coating
			Poor	Buckle Cracking With Interface Failure
Tensile	Brittle	Brittle	Good	Coating Cracking With Interface Failure
			Poor	Edge Failure At Interface
Compressive	Brittle	Brittle	Good	Substrate Cracking And Splitting
			Poor	Buckle Propagation At Interface

TABLE X1.3 Categories, Terms, and Description of Crack Damage Features (derived from Blau and Bull)

Main Category	Damage Term	Description
1. Through-thickness Cracking and Cohesive failure	Brittle tensile cracking	Series of nested micro-cracks, some of which are semicircular, arcs open toward the direction of scratching and form behind the stylus.
	Hertz cracking	Series of nested, nearly-circular micro-cracks within the scratch groove.
	Conformal cracking	Cracking due to the coating trying to conform to the shape of the scratch groove. Less sharp than tensile or hertz cracks; arcs open away from the direction of scratching.
2. Spallation and Adhesive Failure	Buckling	Coating buckles ahead of the tip, producing irregularly-spaced arcs opening away from the direction of scratching, Common for thinner coatings.
	Buckle spallation	Similar to buckling, but with wide, arc-shaped patches missing.
	Wedging spallation	Regularly-spaced and shaped, annular circular that extend beyond the edges of the groove, caused by a delaminated region wedging ahead to separate the coating. Commonly seen in thicker coatings.
	Recovery spallation	Regions of detached coating along one or both sides of the groove. Produced by elastic recovery behind the stylus and depends on plastic deformation in the substrate and cohesive cracking in the coating.
	Gross spallation	Large sections of detached coating within and extending beyond the groove. Common in coatings with low adhesion strength or high residual stresses.
3. Chipping		Rounded regions of coating removal extending laterally from the edges of the groove.

TABLE X1.4 Scratch Atlas

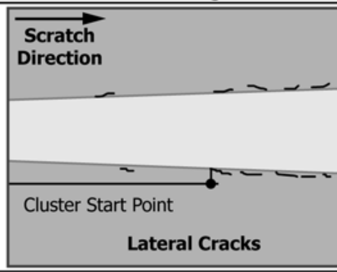
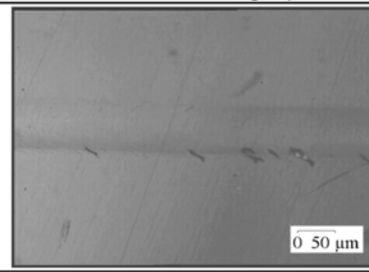
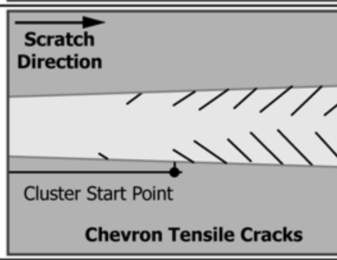
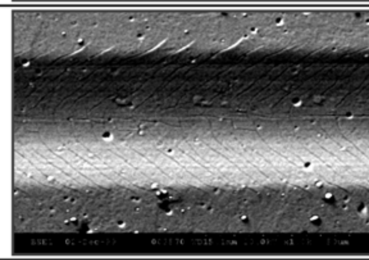
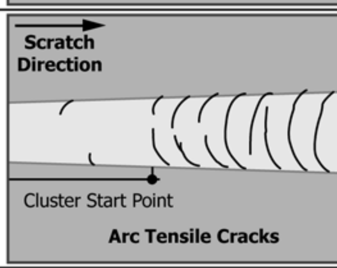
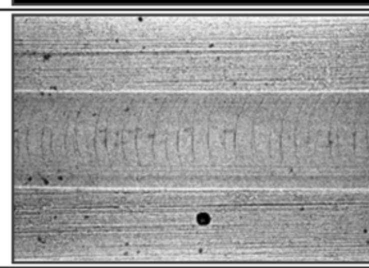
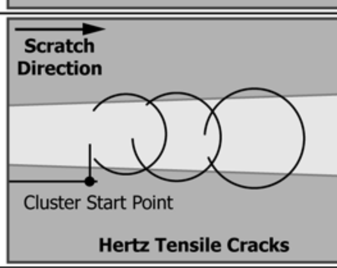
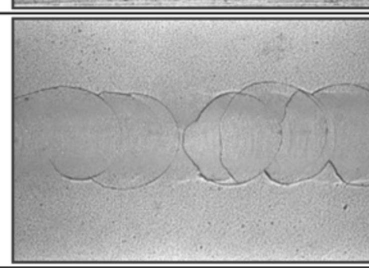
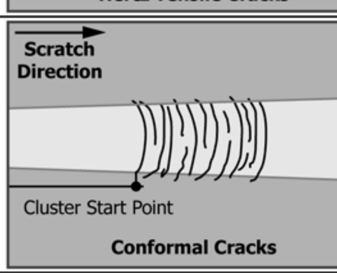
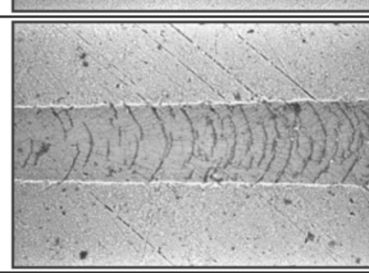
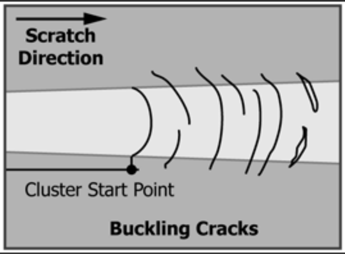
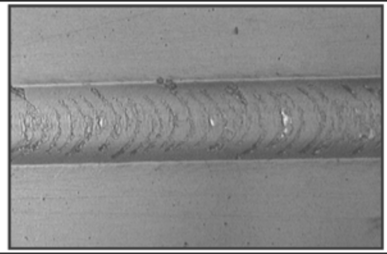
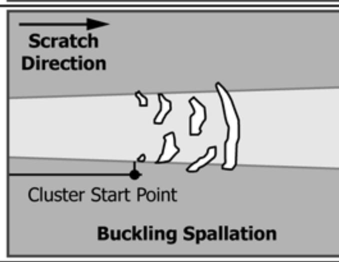
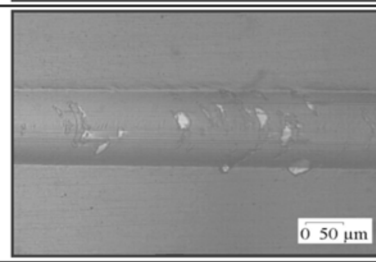
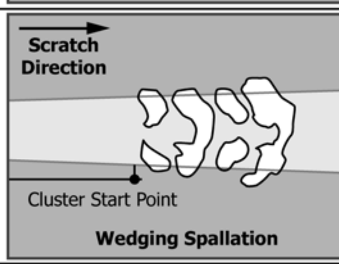
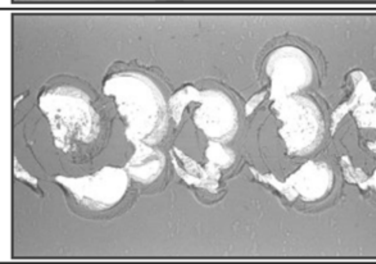
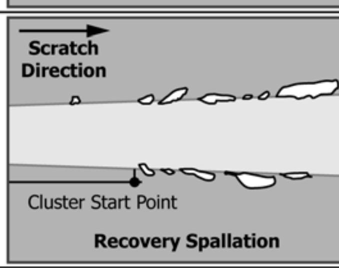
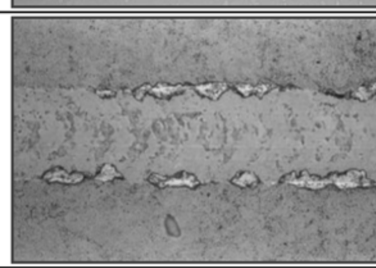
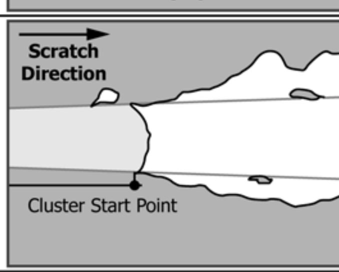
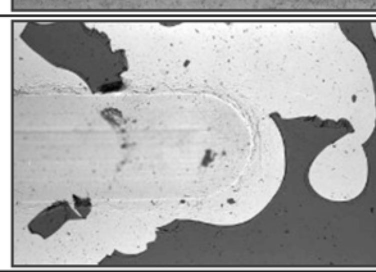
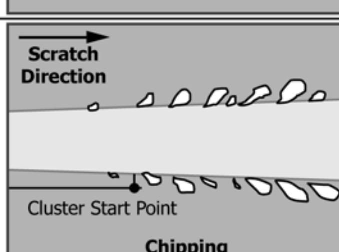
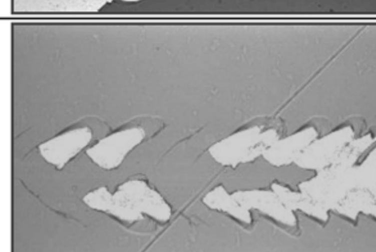
Term	Drawing	Micrograph and Description	
Lateral Cracks			**Lateral Cracks** PACVD DLC (2.3 μm) on hardened and polished M42 steel (66 HRC); Lc = 17 N (*Micrograph Source 1*)
Forward Chevron Tensile Cracks			**Forward Chevron Tensile Cracks** PVD CrN coating (1.9 μm thick) on polished tool steel (HV 720). Lc = 2 N (*Micrograph Source 2*)
Arc Tensile Cracks			**Arc Tensile Cracks** PVD Cr- 0.5% C (5 μm) on hardened and ground M2 Steel (64 HRC); Lc = 15 N (*Micrograph Source 3*)
Hertz Tensile Cracks			**Hertz Type Circular Cracks** PVD AISI 316-10% N (10 μm) on polished 316 steel (155 HB); Lc = 28 N (*Micrograph Source 3*)
Conformal Cracks			**Conformal Cracks** Arc-discharge DLC (0.4 μm) on annealed and polished 440B steel (260 HB); Lc = 8 N (*Micrograph Source 1*)

TABLE X1.4 Scratch Atlas *(continued)*

<table>
<tr><td>Buckling Cracks</td><td>
</td><td></td><td>Buckling Cracks
PVD DLC (2.0 μm) on hardened and polished M42 steel (66 HRC);
Lc = 54 N
(Micrograph Source 1)</td></tr>
<tr><td>Buckling Spallation</td><td>
</td><td>
</td><td>Buckling Spallation
PVD DLC (2.0 μm) on hardened and polished M42 steel (66 HRC);
Lc = 57 N
(Micrograph Source 1)</td></tr>
<tr><td>Wedging Spallation</td><td>
</td><td></td><td>Wedging Spallation Along the ScratchCrack
PACVD DLC (3.3 μm) on hardened and polished M2 steel (64 HRC);
Lc = 43 N
(Micrograph Source 3)</td></tr>
<tr><td>Recovery Spallation</td><td>
</td><td></td><td>Recovery Spallation at the Border of the ScratchCrack
PVD TiN (1.4 μm) on hardened and polished M2 steel (64 HRC);
Lc = 37 N
(Micrograph Source 3)</td></tr>
<tr><td>Gross Spallation</td><td>
</td><td></td><td>Large Area Interfacial Spallation
PACVD DLC (2 μm) on hardened and polished M2 steel (64 HRC);
Lc = 10 N
(Micrograph Source 3)</td></tr>
<tr><td>Chipping</td><td>
</td><td></td><td>Chipping from Forward Chevron Cracks
PACVD DLC (3.3 μm) on hardened and polished M2 steel (64 HRC);
Lc = 29 N.
(Micrograph Source 3)</td></tr>
</table>

Micrographs are provided courtesy and with the permission of the following sources:

Source #	Micrograph Source	Test Parameters
1	Ms. Joanne Stallard, Teer Coatings LTD, Droitwich, Worcestershire, England; Contact joanne.stallard@teercoatings.co.uk	NA
2	Dr. Yong Song Xie, National Research Council Canada Institute for Fuel Cell Innovation, Vancouver, BC, Canada; Contact: yongsong.xie@nrc-cnrc.gc.ca	NA
3	Scratch Test Atlas of Failure Modes, in poster format, issued by Vito Flemish Institute for Technological Research, Boeretang 200, B-2400 Mol, Belgium; Contact: karel.vanacker@vito.be	Progressive load of 100 N/min and a displacement rate of 10 mm/min

X2. EXPERIMENTAL VARIABLES IN SCRATCH ADHESION TESTING

X2.1 For both the coating scientist and the coating engineer, the measurement of coating adhesion is a critical requirement in the development, evaluation, production, and use of functional coatings. But coating adhesion is one of the most difficult physical properties to quantify for both experimental and fundamental reasons, because the response to a scratch load is "Not a basic property but a response of a system to an applied test condition" (from Blau's *Lab Handbook of Scratch Testing*).

X2.2 One aspect of adhesion testing that is generally accepted is that adhesion can be considered from two different perspectives: "fundamental adhesion" and "practical adhesion" (Mittal; Buchwalter; Meneve). Mittal defines the two terms as:

X2.2.1 *Fundamental Adhesion*—The summation of all interfacial intermolecular interactions between the contacting materials.

X2.2.2 *Practical Adhesion*—The force or work required to remove or detach a film or coating from its substrate irrespective of the locus of failure.

X2.3 "Fundamental adhesion" is the term used to define the binding force and energy at the interface between two layers, determined by the chemical bond between the layers. It requires the quantitative measurement of the force necessary to break the chemical bonds or the specific energy needed to separate the two materials. As Evans points out, the energy measurement is analogous to fracture toughness and could be used explicitly in design codes and durability models in a methodology similar to fracture mechanics models. However, direct measurement of the fundamental adhesion is very challenging, because of geometric, material, and experimental difficulties.

X2.4 "Practical adhesion" is a test concept which uses various engineering coating adhesion test methods to obtain a quantitative, reproducible adhesion measurement which can be related to the functional performance of the coating. "Practical adhesion" can also be described as the force or work required to detach or disrupt the coating from the substrate. The practical adhesion measurement is a function of the fundamental adhesion and the different experimental factors related to the coating-substrate pair and the specific experimental method.

X2.5 The stresses during the dynamic scratch test are not simple or straightforward. The linear motion of the hard diamond stylus under the applied normal force produces a complex, changing stress state (compression, shear, and tensile) in the coating. The applied stresses also combine with any residual stresses in the coating. All these stresses interact with the coating-substrate system (in all its complexity of geometry, microstructure, and mechanical properties) to give a wide range of possible mechanical responses—plastic deformation, cracking, spallation, buckling, chipping, etc.

X2.6 In considering the mechanism of coating damage in a scratch test, it should be understood that two general failure modes can be observed (Blau):

X2.6.1 *Cohesive Failure*—Separation of or significant damage in one portion of the coating system, either the coating or the substrate.

X2.6.2 *Adhesive Failure*—A separation between the coating and substrate

X2.7 In a hard coating system, both adhesive and cohesive failure can occur, but at different locations and loads within the coating system. As Blau describes: "if a crack grows within a coating and that process results in the delamination of a layer from the surface, that would be a cohesive failure. This occurs when the bond between the coating and the substrate is stronger than either the coating material or the substrate or when the point of maximum contact stress lies within the coating or substrate and not at a strong interface."

X2.8 As Meneve et al state: "Only some of the observed failure modes, however, are related to detachment at the coating/substrate interface and are thus relevant as a measure of adhesion. Other failure events, such as cracking or cohesive failure within the coating or substrate may occur but clearly cannot be used to assess the coating/substrate adhesion strength. The latter, however, can be equally important to determine the behavior of a coated component in a particular application. Indeed, the scratch test is increasingly being regarded as a tribological test to assess the mechanical integrity of a coated surface."

X2.9 Since different coating systems can fail in different manners, a primary requirement in using the scratch adhesion test is to clearly describe the coating damage events which are used to define the critical scratch load(s).

X2.10 *Critical Experimental Test Variables*—The scratch adhesion test is by its very nature a complex experimental system with a wide range of specimen and test method variables. The variables can be organized into five groups as shown in Table X2.1.

X2.10.1 Under ideal conditions, all of the experimental (specimens, equipment, procedures) variables would be measured, controlled, and reported. However, time, cost and experimental difficulties limit the variables which can be practically managed. In the real world, resources must be focussed on those experimental variables which have the most impact and greatest effect on the accuracy, repeatability, and validity of the scratch adhesion test.

X2.10.2 A review of five technical documents on scratch adhesion test methodology gave a comprehensive list of critical experimental parameters which must be measured and controlled for data reproducibility and repeatability.

X2.10.2.1 *Coating Thickness*—Thicker coatings have a "general" tendency to produce lower critical scratch loads,

TABLE X2.1 Critical Experimental Test Variables

Coating Properties	Substrate Properties
Coating thickness	Substrate composition and phases
Coating composition and phases	Surface hardness and roughness at the interface
Coating hardness and surface roughness	Grain size/microstructure
Grain size/microstructure	Anisotropy in structure and properties
Density and porosity	Areal variation and batch-to-batch variation
Anisotropy in structure and properties	Elastic/plastic mechanical properties
Areal variation and batch-to-batch variation	Fracture mechanisms
Elastic/plastic mechanical properties	Flaw population and distribution
Fracture mechanisms	Strain rate effects
Flaw population and distribution	Exposure conditioning effects on microstructure and properties
Strain rate effects	
Exposure conditioning effects on microstructure and properties	
Interface and Material System Properties	**Test Equipment and Procedure Variables**
Adhesion energy at the interface	Stylus—material, geometry, tip size, cleanliness, damage condition
Residual stresses in the system as a function of coating thickness	Drag coefficient between the stylus and the coating
Elastic modulus mismatch	Loading and displacement rates
Interface failure mechanisms	System compliance–horizontal and vertical
Interface porosity, flaws, and damage	Sensor accuracy and precision
Areal variation and batch-to-batch variation	Equipment calibration
Exposure conditioning effects on microstructure and properties	Scratch length
	Proximity to prior scratches
	Optical/microscope quality
	Operator skill
Specimen and Test Environment Variables	
Surface cleanliness and contamination	Test temperature, humidity, contaminants
Specimen flatness, level, and orthogonality	Lubrication and tailored test environment

because there are higher residual stresses with thicker coatings. With higher residual stresses, lower applied loads will reach the practical adhesion stress levels and produce coating damage.

$$\text{Coating Thickness}\uparrow = L_C\downarrow \qquad [\text{Many References}]$$

NOTE X2.1—Coating thickness can interact with other factors in a complex manner to influence the critical load As describe above, thicker coatings commonly have lower critical loads, because of residual stresses. However, hard thicker films can also provide greater load-bearing capability, if residual stresses are not too high. This is particularly important for hard, brittle coatings on soft, ductile substrates where considerable substrate deformation can occur before delamination failure. Under these conditions the critical load for spallation may increase with coating thickness.

X2.10.2.2 *Substrate Roughness*—Higher substrate roughness decreased the critical scratch loads.

$$\text{Substrate Roughness}\uparrow = L_C\downarrow \qquad [\text{Blau}]$$

X2.10.2.3 *Loading and Traverse Rates*—Increasing the load rate (N/min) or decreasing the sliding speed (mm/min) increased the critical scratch load.

$$\text{N/min}\uparrow = L_C\uparrow \quad \rightarrow \quad \text{mm/min}\uparrow = L_C\downarrow \qquad [\text{Blau, Randall}]$$

X2.10.2.4 *Stylus Size*—Increasing the stylus tip radius increased the critical scratch loads (larger loading area = lower applied stress.

$$\text{Tip Radius}\uparrow = L_C\uparrow \qquad [\text{Blau, Randall, Xie, Ichimura}]$$

Xie also proposed that a larger tip radius will produce higher compressive stresses and induce the desired adhesive failures and suppress cohesive failure by reducing the bending-induced stresses.

X2.10.2.5 *Stylus Composition*—Stylus tip materials with lower coefficients of friction decreased the critical scratch loads. Diamond has the lowest coefficient of friction, compared to tungsten carbide and chromium steel.

$$\text{Coef. Friction}\downarrow = L_C\downarrow \qquad [\text{Blau}]$$

X2.10.2.6 *Stylus Wear and Damage*—Wear and flattening of the diamond stylus tip increased the critical scratch loads, while chipping of the diamond tip decreased the critical scratch loads through the introduction of stress concentrations.

$$\text{Tip Wear} = L_C\uparrow \quad \text{Tip Damage} = L_C\downarrow \qquad [\text{Meneve}]$$

X2.10.2.7 *Specimen and Tip Contamination*—Contamination on the stylus tip and/or the coating surface changes the friction coefficient and can increase or decrease the critical scratch loads, depending on the composition of the contamination.

$$\text{Contamination} = L_C\uparrow\text{or}\downarrow \qquad [\text{Meneve}]$$

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