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Designation: D7187 − 20

Standard Test Method for
Measuring Mechanistic Aspects of Scratch/Mar Behavior of Paint Coatings by Nanoscratching[1]

This standard is issued under the fixed designation D7187; 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 a nanoscratch method for determining the resistance of paint coatings on smooth flat surfaces to scratch/mar.

1.2 Other methods used in scratch/mar evaluation physically scratch or mar a sample's surface with single or multiple contact cutting, and then use visual inspection to assign a ranking. It has been recognized that loss of appearance is mainly due to surface damage created. This method quantitatively and objectively measures scratch/mar behavior by making the evaluation process two steps with emphasis on surface damage. Step one is to find the relationship between damage shape and size and external input (such as forces, contact geometry, and deformation). Step two is to relate damage shape and size to visual loss of luster. The first step is covered by this method; in addition, a survey in the appendix provides an example of an experiment to relate the damage to the change in luster.

1.3 There are three elementary deformation mechanisms: elastic deformation, plastic deformation and fracture; only the latter two contribute significantly to mar. This method evaluates scratch/mar based on the latter two damage mechanisms.

1.4 Although this standard was developed for paint coatings, it can also be applied to other types of similar polymer-based coatings, for example, lacquers, varnishes, glazes and other decorative and protective layers deposited on hard substrates.

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D609 Practice for Preparation of Cold-Rolled Steel Panels for Testing Paint, Varnish, Conversion Coatings, and Related Coating Products

D823 Practices for Producing Films of Uniform Thickness of Paint, Coatings and Related Products on Test Panels

D1005 Test Method for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers

D1044 Test Method for Resistance of Transparent Plastics to Surface Abrasion by the Taber Abraser

D3363 Test Method for Film Hardness by Pencil Test (Withdrawn 2020)[3]

D3924 Specification for Standard Environment for Conditioning and Testing Paint, Varnish, Lacquer, and Related Materials

D5178 Test Method for Mar Resistance of Organic Coatings

D6037 Test Methods for Dry Abrasion Mar Resistance of High Gloss Coatings

D6279 Test Method for Rub Abrasion Mar Resistance of High Gloss Coatings

D7027 Test Method for Evaluation of Scratch Resistance of Polymeric Coatings and Plastics Using an Instrumented Scratch Machine

D7091 Practice for Nondestructive Measurement of Dry Film Thickness of Nonmagnetic Coatings Applied to Ferrous Metals and Nonmagnetic, Nonconductive Coatings Applied to Non-Ferrous Metals

G171 Test Method for Scratch Hardness of Materials Using a Diamond Stylus

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[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Films.

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

[3] The last approved version of this historical standard is referenced on www.astm.org.

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3. Summary of Test Method

3.1 This test method is based on representative samples of the paint film being scratched using a nanoscratch instrument. From information generated during a scratch test, values for plastic resistance (PR) and fracture resistance (FR) can be determined. From these values, the mechanistic aspects of scratch/mar behavior of the coating can be compared.

4. Significance and Use

4.1 This test addresses two limitations in existing mar tests such as Test Methods D1044, D3363, D5178, D6037, and D6279, namely:

4.1.1 Measured damage is caused by hundreds of contacts with differing contact geometries making it difficult or impossible for mechanical quantities (force, displacement) at the contact points to be reliably determined.

4.1.2 The damage is evaluated using subjective visual assessments, which provide only a qualitative sense of wear with little information about mar mechanisms.

4.2 This test provides a quantitative assessment of a paint coating's mechanistic aspects of scratch/mar behavior in various conditions. The ability to control testing variables such as loading rate, speed, and temperature allow the study of the scratch/mar behavior in a variety of environments.

4.3 This test method is particularly suitable for measurement of paint coatings on laboratory test panels.

4.4 The accuracy and precision of scratch/mar performance may be significantly influenced by coating surface nonuniformity and irregularities.

4.5 A correlation has been observed between good mar resistance in field studies and a combination of high plastic resistance and high fracture resistance. When coatings have had either high plastic resistance and low fracture resistance, or low plastic resistance and high fracture resistance, there have been contradictory results in field studies.

4.6 Mar resistance characterizes the ability of the coating to resist light damage. The difference between mar and scratch resistance is that mar is related to only the relatively fine surface scratches which spoil the appearance of the coating. The mechanistic aspects of mar resistance depend on a complex interplay between visco-elastic and thermal recovery, yield or plastic flow, and micro-fracture. Polymers are challenging because they exhibit a range of mechanical properties from near liquid through rubber materials to brittle solids. The mechanical properties are rate and temperature dependent and visco-elastic recovery can cause scratches to change with time. One such test for evaluating polymeric coatings and plastics is Test Method D7027.

4.7 Since this method measures mechanical qualities, such as forces and displacements (deformations) during the damage making process, rate dependence, temperature dependence, and visco-elastic-plastic recovery can be further investigated and visual impacts of damage can be related to deformation mechanisms.

5. Apparatus

5.1 *Paint Application Equipment,* as described in Practices D609 and D823.

5.2 *Nanoscratch Instrument,* consisting of an instrument with a well-defined indenter, which translates perpendicular to the coating surface and has the capacity to produce an instrumented scratch of controlled and variable normal force and continuously measured displacement during testing. The normal force shall be feedback controlled, in order to quickly respond to variations in surface morphology. The force of the instrument should have a maximum normal force of at least 50 mN ± 0.1 mN. The maximum tangential force, if measured, should be at least 50 mN ± 0.5 mN. The range of the displacement sensors should be at least 200 µm with a resolution of at least 20 nm. Displacement and tangential force response of the coating should be measured with a high data acquisition rate, such as a maximum of 5 µm between data points.

5.3 *Suggested Range for Testing Parameters:*

5.3.1 Indenter size should range from 1 µm to 100 µm and should be spherical in geometry. Indenter material should be diamond.

5.3.2 The scratch should be applied at a rate of 0.5 mm to 10 mm per minute.

5.3.3 The loading rate of the normal force should be applied at 5 mN to 200 mN per minute.

5.3.4 The scanning preload should be conducted with an applied force of 0.1 mN to 1 mN.

5.4 The following is an example of one particular application of the test ranges. This example is based on automotive clear coats on a metal substrate.

5.4.1 Indenter size of 2 µm.

5.4.2 Scratch speed of 3 mm per minute.

5.4.3 Loading rate of 40 mN per minute.

5.4.4 Scanning preload of 0.2 mN.

5.4.5 Data acquisition rate of 3 µm between data points.

NOTE 1—To optimize test parameters for a particular coating, experiments may need to be conducted as different combinations of applied load and indenter radius will cause differing damage in coatings. A smaller indenter radius (sharper tip) will tend to cut the coating and apply a higher contact pressure, whereas a larger indenter radius (blunter tip) will tend to tear the coating and apply a lower contact pressure.

6. Test Specimen

6.1 The substrate for the paint coating should be a smooth, plane, rigid surface, such as those specified in Practices D609 and D823.

6.2 The thickness of the coating being tested, determined in accordance with either Test Methods D1005 or D7091, should be uniform within 5 % of coating thickness.

6.3 At least three scratches shall be performed on each test specimen.

6.4 The surface of the specimens shall be free of any dirt and oils. Care should be taken when cleaning samples: solvents should not be used as they may modify the surface properties

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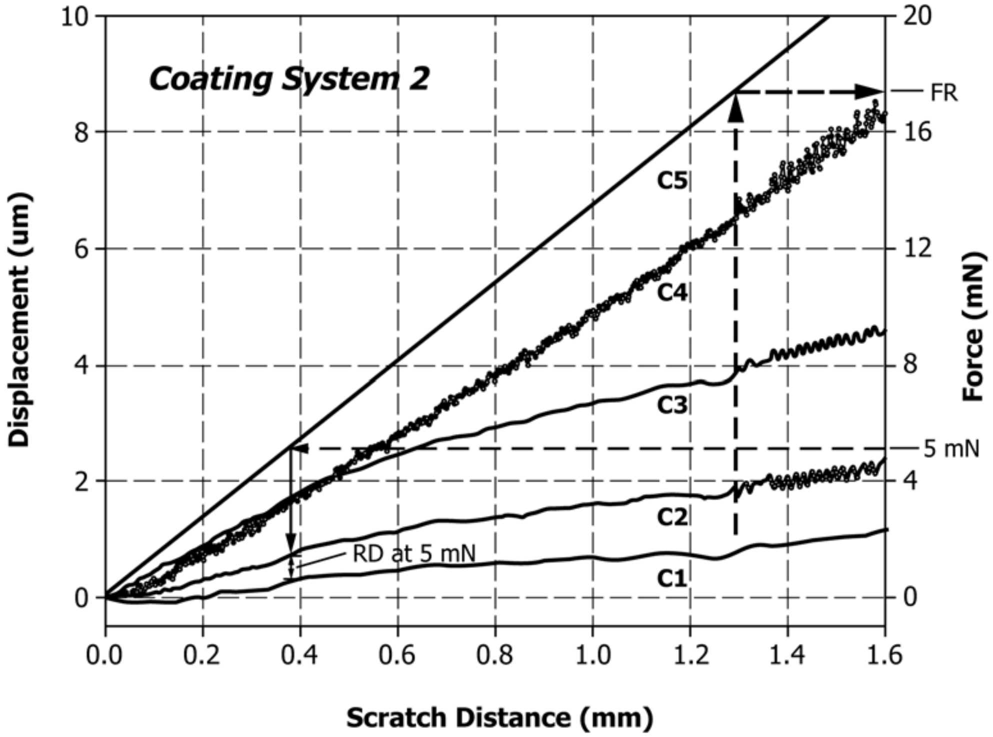

FIG. 1 Example - Typical Data from a Nanoscratch Test (1)

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of polymer-based coatings. For removing dust, it is recommended to blow off particulates with compressed air from a clean source (without oil contamination).

6.5 The specimen size should be sufficient to be adequately secured to the nanoscratch instrument, but not so small as to interfere with the movement of the indenter tip or its supporting cantilever.

NOTE 2—It is recommended that substrates with similar compliances be used when comparing different coatings.

7. Conditioning

7.1 Cure the coated test specimens under conditions agreed upon between the purchaser and seller that reflect the conditions of curing of the paint in actual service.

7.2 Condition and test the test specimens at 23 °C ± 2 °C (73.5 °F ± 3.5 °F) and a relative humidity of 50 % ± 5 % for at least 24 h, unless the purchaser and seller agree on more suitable test characteristics, as specified in the Standard Atmosphere of Specification D3924.

8. Procedure

8.1 Secure the specimen to the moveable stage on the instrument with the surface to be measured located perpendicularly to the indenter tip. Ensure the panel is held rigidly to the stage and cannot be moved by the action of the subsequent scratch test.

8.2 Carefully move this area under the indenter and bring the indenter tip close to the sample surface.

8.3 The complete scratch test consists of three distinct steps. In all three steps, the indenter follows the exact same path across the sample surface.

NOTE 3—A set of sample test parameters can be found in 5.4.

8.3.1 Perform a prescan to measure the topography of the undamaged coating. Apply the lowest load that the instrument can apply but that makes no permanent damage. The prescan, scratch, and postscan must be performed on the same line.

8.3.2 Instruct the instrument to begin making a scratch to produce damage to the coating. Allow the instrument to ramp to the desired normal force at a controlled rate. At the end of the scratch, return the indenter tip to its starting position at the beginning of the scratch.

8.3.3 Without delay perform a postscan, where the indenter tip is scanned along the scratch, measuring the residual topography of the damaged area. This should be done with the lowest load the instrument can apply.

NOTE 4—Prescan and postscan should only be used if the instrument has force feedback control, otherwise significant error may be incurred.

NOTE 5—The prescan and postscan need to be conducted consistently (with the same scanning parameters) before and after the scratch load is applied. This is done to accurately measure recovery aspects since these aspects will vary with time.

8.4 The complete scratch test shall be repeated two more times at different locations so that there are a total of three scratches per test panel.

8.5 Results of typical data from a nanoscratch test are presented in Fig. 1[4]. The graph consists of five curves labeled C_1 through C_5. If required, correct the data by curve fitting so that zero indenter penetration and residual depth (RD) corresponds to zero applied normal force.

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[4] The boldface numbers in parentheses refer to the list of references at the end of this test method.

8.5.1 Curve 1 (C_1) shows the topography of the unscratched surface along the scratch path. It is a measure of the vertical displacement of the indenter tip during a low (~0.2 mN) constant load prescan.

8.5.2 Curve 2 (C_2) shows the topography of the damaged surface along the scratch path immediately after the scratch test was concluded. It is a measure of the vertical displacement of the indenter tip during a low (~0.2 mN) constant load scan through the completed scratch.

8.5.3 Curve 3 (C_3) shows the vertical displacement of the indenter tip during the scratching process.

8.5.4 Curve 4 (C_4) shows the tangential force that arises between the coating and the indenter tip.

8.5.5 Curve 5 (C_5) shows the applied normal force on the coating surface.

9. Calculations

9.1 From analysis of the force/displacement versus scratch distance plot produced (Fig. 1), plots of various properties relating to the mechanical behavior of the coating versus scratch distance can be generated:

9.1.1 Penetration depth (PD) of the indenter under the applied normal force can be calculated by subtracting the surface topography measured during the prescan, curve 1 (C_1), from the displacement measured during the scratch, curve 3 (C_3).

$$PD = C_3 - C_1$$

where PD means penetration depth, and C_3 and C_1 correspond to curves 3 and 1 respectively.

9.1.2 The magnitude of residual depth (RD), otherwise known as permanent plastic deformation, to the coating can be calculated by subtracting the surface topography before the scratch, curve 1 (C_1), from the topography after the scratch, curve 2 (C_2).

$$RD = C_2 - C_1$$

9.1.3 Elastic recovery (ER) of the coating is the difference between the displacement during the scratch, curve 3 (C_3), and the surface topography after the scratch, curve 2 (C_2).

$$ER = C_3 - C_2$$

9.1.4 The ratio of tangential force, curve 4 (C_4), to the normal force, curve 5 (C_5), is a form of the friction coefficient (C_f).

$$C_f = C_4/C_5$$

9.2 Plastic resistance (PR) at a particular normal force can be calculated by dividing the normal force by the magnitude of the permanent damage at that normal force before fracture occurs. Selecting the spot for measurement to be at a higher applied normal force results in values that reflect a more true plastic resistance. This gives a value for plastic resistance that is relatively constant in units of force per unit of damage depth, or mN/µm.

$$PR = F_N/RD$$

where:

PR = plastic resistance,
F_N = the normal force in mN, and
RD = permanent plastic deformation or residual depth in µm.

NOTE 6—Only at very low normal force values does the plastic resistance differ radically. In the case of Fig. 2, the material fractured at 17 mN so plastic resistance should be measured at a lower force (measurement point selected was 5 mN). Plastic resistance values should be evaluated in the area of the curve that is relatively constant and in the area before fracture occurs.

9.3 Fracture resistance (FR) can be determined by locating the point where normal force, tangential force, penetration depth of the indenter and permanent damage begin to fluctuate wildly. This is the point where the first fracture occurs. Any subsequent increase in normal force only leads to increased fracture. This mechanical quantity is known as the *critical load* and has units of mN.

NOTE 7—Onset of the fracture can also be identified with an integrated video microscope (with at least 1000× magnification) or Atomic Force Microscope. In this case, calibrate the position of the microscope with the scratch tip prior to the start of the scratch test.

9.4 Plastic deformation and fracture are the two damage mechanisms that will have an effect on the coating performance. As shown in Fig. 3 a and b, the morphology of these two types of deformation is quite different. This difference in morphology is what has the most profound influence on the appearance of the coating. Plastic deformation is calculated in 9.1.2 and is also known as the magnitude of permanent damage or residual depth (RD).

9.5 Data for several coatings may be compared using a graph of plastic resistance versus fracture resistance, as shown in Fig. 4.

9.6 For some coating types, it may be of interest to calculate the plastic resistance based on the width of the scratch. In this case, the plastic resistance can be defined as the ratio of applied normal load divided by the width of the scratch at that point along the scratch. This methodology is similar to that described in Test Method G171 for the measurement of scratch hardness. It requires a means of directly measuring the scratch width; this can be achieved by microscopy or by stylus profilometry.

10. Report

10.1 Report the following information:

10.1.1 Mean and range of fracture resistance (FR) and plastic resistance (PR) or residual depth (RD) values, and where these values are measured, obtained for each sample.

10.1.2 Type of coating, substrate and coating techniques used.

10.1.3 Time and temperature of sample conditioning.

10.1.4 Indenter size and shape.

10.1.5 Applied load range (minimum and maximum), loading rate and scanning force.

11. Precision and Bias

11.1 *Precision*—Six samples, each of three automotive clearcoats with differing chemistry, named System 1, 2, and 3, were tested at Dupont Marshall Labs, CSM Instruments and FPL for repeatability and reproducibility and discrimination. Statistically, the rule of thumb is at least 30 data points are needed to assess variability. The data set with three

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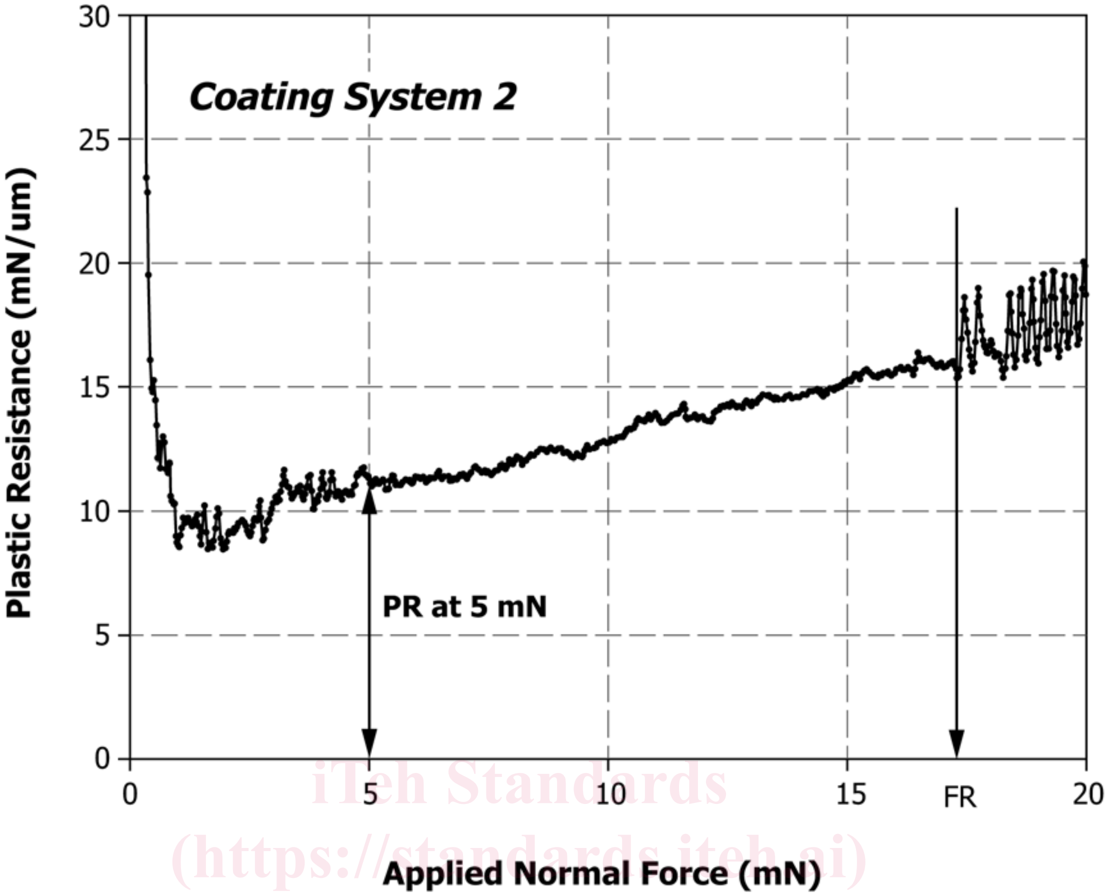

FIG. 2 Example - Variation of Plastic Resistance (PR) with Respect to Applied Normal Force (2)

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laboratories, three different samples (systems) and six measurements each, yields a total of 54 results for analysis and is used to demonstrate the method's repeatability and reproducibility. In all cases, the CSM Instruments Nano-Scratch Tester (NST) was used (**3**). The software MINITAB was utilized to perform the statistical analysis.

TABLE 1 Statistical Results of Fracture Resistance

Materials	Mean	Sr	SR	r	R
System 1	8.8556	0.2685	0.3290	0.7518	0.9213
System 2	18.9111	0.6241	1.3024	1.7476	3.6467
System 3	31.5822	1.0954	1.8195	3.0671	5.0946

TABLE 2 Statistical Results of Residual Deformation

Materials	Mean	Sr	SR	r	R
System 1	0.2087	0.0199	0.0243	0.0557	0.0680
System 2	0.4150	0.0206	0.0296	0.0576	0.0828
System 3	0.1346	0.0276	0.0298	0.0773	0.0834

11.1.1 Fracture Resistance:

11.1.1.1 Table 1 shows the statistical results of fracture resistance. Here Sr is the average value of standard deviation measured by the three individual groups, and SR is the overall standard deviation of all 18 samples from the three groups. Values of r and R are 2.8 times of Sr and SR, respectively. Repeatability is characterized by the values of Sr and r. Reproducibility is characterized by the values of SR and R.

11.1.1.2 Fig. 5 further illustrates the repeatability, reproducibility and discrimination of the test method regarding fracture resistance measurements. A Gage R&R study with ANOVA method is given in Fig. 6.

11.1.2 Residual Deformation:

11.1.2.1 Table 2 shows the statistical results of residual deformation. Fig. 7 further illustrates the repeatability, reproducibility and discrimination of the test method regarding residual deformation measurements. A Gage R&R study with ANOVA method is given in Fig. 8.

11.1.3 Plastic Resistance:

11.1.3.1 Table 3 shows the statistical results of plastic resistance. Fig. 9 further illustrates the repeatability, reproducibility and discrimination of the test method regarding plastic resistance measurements. A Gage R&R study with ANOVA method is given in Fig. 10.

11.1.4 The data in Tables 1-3, indicate that relatively large errors (ratio of deviation to mean) occur during the residual deformation measurement and plastic resistance measurement of coating system 3. This is due to the unusual, rubbery behavior of the material, which results in more than 97 % immediate deformation recovery (in comparison to 70 % to 80 % for most coatings) and makes measurement of residual

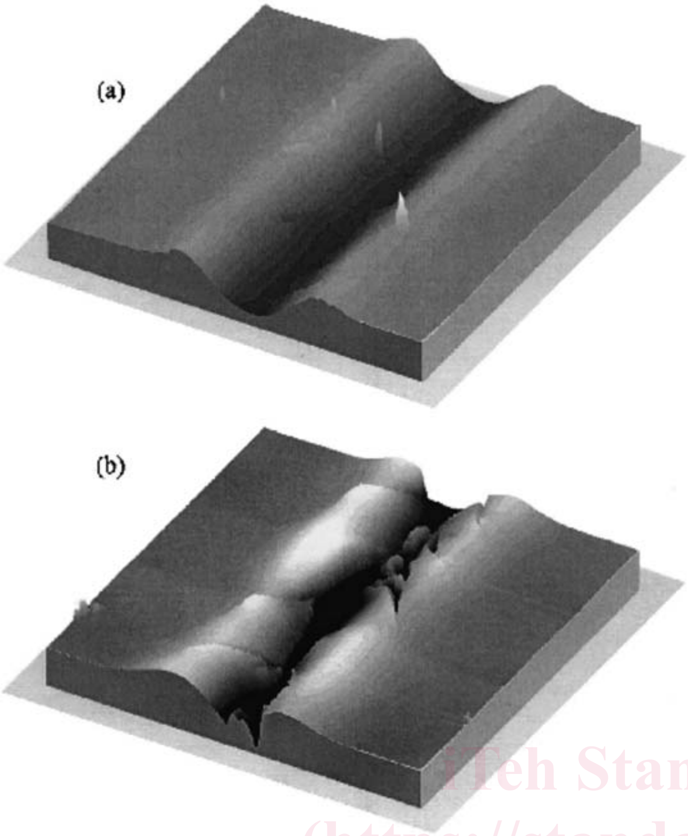

FIG. 3 AFM Images of a Scratch before Fracture (a) and after Fracture (b)

TABLE 3 Statistical Results of Plastic Resistance

Materials	Mean	Sr	SR	r	R
System 1	24.2866	2.3665	3.0237	6.6262	8.4663
System 2	12.1097	0.6227	0.9179	1.7434	2.5702
System 3	38.9381	8.8602	9.4925	24.8084	26.5791

deformation more challenging. The errors are further exaggerated when calculating plastic resistances because a low value of residual deformation in the denominator greatly impacts the plastic resistance calculation.

11.2 *Bias*—This procedure has no bias because the values for plastic deformation and fracture resistance, though resembling fundamental mechanical properties, are defined only in this test and in that respect are highly subject to the variables of the test itself, such as the scratch speed and loading rate.

12. Keywords

12.1 coatings; gloss; mar or organic coating mar resistance; scratch resistance

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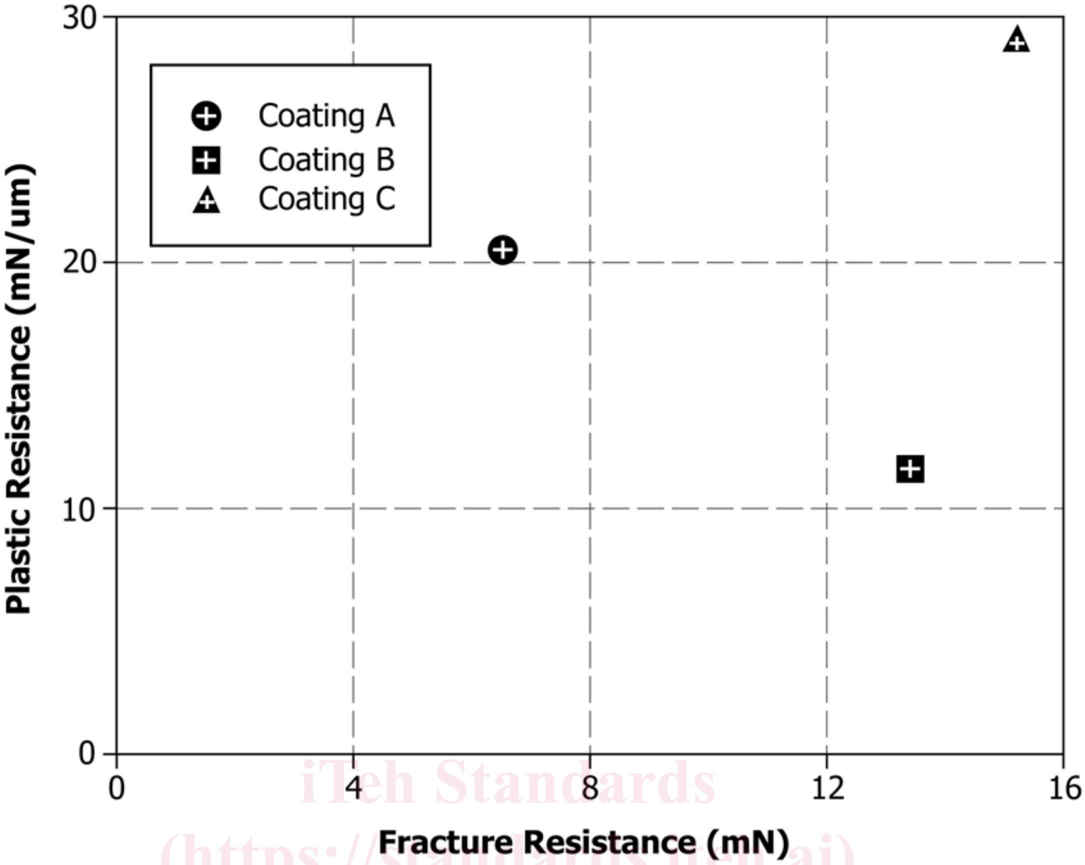

FIG. 4 Example - Mar Resistance of Coatings A, B, and C

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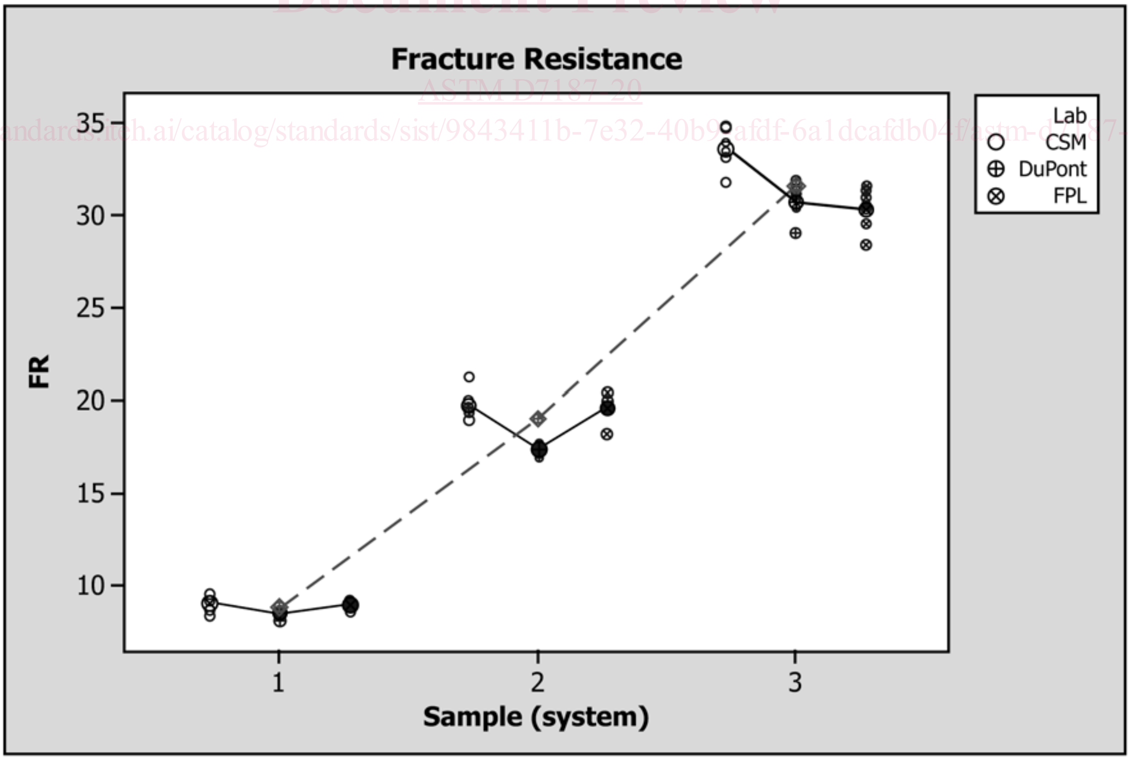

FIG. 5 Repeatability, Reproducibility and Discrimination of Fracture Resistances