Designation: D4541 – 17

Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers[1]

This standard is issued under the fixed designation D4541; 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 procedure for evaluating the pull-off strength (commonly referred to as adhesion) of a coating system from metal substrates. Pull-off strength of coatings from concrete is described in Test Method D7234. This test offers two test protocols. Protocol 1 (test to fracture) determines the greatest perpendicular force (in tension) that a surface area can bear before a plug of material is detached. Protocol 2 (pass/fail) determines if the coated surface remains intact at a defined load criteria. Fracture will occur along the weakest plane within the system comprised of the test fixture, glue, coating system, and substrate, and will be exposed by the fracture surface. This test method maximizes tensile stress as compared to the shear stress applied by other methods, such as scratch or knife adhesion, and results may not be comparable.

NOTE 1—The procedure in this standard was developed for metal substrates, but may be appropriate for other rigid substrates such as plastic and wood. Factors such as loading rate and flexibility of the substrate must be addressed by the user/specifier.

NOTE 2—The procedure in this standard was developed for use on flat surfaces. Depending on the radius of the surface, the results could have greater variability with lower values and averages.

1.2 Pull-off strength measurements depend upon material, instrumentation and test parameters. Results obtained by each test method may give different results. Results should only be assessed for each test method and not be compared with other instruments. There are five instrument types, identified as Test Methods B-F. It is imperative to identify the test method used when reporting results.

NOTE 3—Method A, which appeared in previous versions of this standard, has been eliminated as its main use is for testing on concrete substrates (see Test Method D7234).

1.3 This test method describes a class of apparatus known as portable pull-off adhesion testers.[2] They are capable of applying a concentric load and counter load to a single surface so that coatings can be tested even though only one side is accessible. Measurements are limited by the strength of adhesive bonds between the loading fixture and the specimen surface or the cohesive strengths of the glue, coating layers, and substrate.

1.4 This test can be destructive and spot repairs may be necessary.

1.5 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

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:*[3]

D2651 Guide for Preparation of Metal Surfaces for Adhesive Bonding

D3933 Guide for Preparation of Aluminum Surfaces for Structural Adhesives Bonding (Phosphoric Acid Anodizing)

D7234 Test Method for Pull-Off Adhesion Strength of Coatings on Concrete Using Portable Pull-Off Adhesion Testers

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

[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 D01.46 on Industrial Protective Coatings.

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[2] The term adhesion tester may be somewhat of a misnomer, but its adoption by two manufacturers and at least two patents indicates continued usage.

[3] 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.

*A Summary of Changes section appears at the end of this standard

3. Summary of Test Method

3.1 The general pull-off test is performed by securing a loading fixture (dolly, stud) normal (perpendicular) to the surface of the coating with a glue. After the glue is cured, a testing apparatus is attached to the loading fixture and aligned to apply tension normal to the test surface. The force applied to the loading fixture is then gradually and uniformly increased and monitored until either the loading fixture is detached, or a specified load value is reached and the test terminated. The two common uses of this test are test to fracture (Protocol 1), and pass/fail testing (Protocol 2). Test to fracture is used to determine a maximum load that can be achieved when a plug of material is detached with the selected testing parameters. Pass/fail is used to verify the results of a testing procedure can meet a minimum load criterion. When the loading fixture is detached, the exposed surface represents the plane of limiting strength within the system. The nature of the plane of fracture is qualified in accordance with the percent of adhesive and cohesive failures, and the actual interfaces and layers involved. The reported load is computed based on the maximum indicated load, the instrument calibration data, and the original surface area stressed. Results obtained using different test devices will vary because the results depend on instrumentation parameters. Variations in results are also expected when tests are performed under different test procedures or environmental conditions (see 4.2).

4. Significance and Use

4.1 The pull-off strength of a coating is a performance property that may be referenced in specifications. This test method serves as a means for uniformly preparing and testing coated surfaces, and evaluating and reporting the results. This test method is applicable to any portable apparatus meeting the requirements for determining the pull-off strength of a coating in this standard (see Annexes).

4.2 Variations in results with the same coating are likely when any parameter of the test is changed. This includes change in glue, load fixture size, substrate coating cure time, pull rate, environmental conditions, if the coating is scored, or using a different device. Therefore, when a series of results will be compared with one another or used for statistical analysis, the type of apparatus, substrate, test procedures, glue type, and if scoring is used should be the same for the pulls considered.[4] It is recommended that these parameters and the environmental conditions allowed during the test be mutually agreed upon between the interested parties.

4.3 The purchaser or specifier shall designate a specific test method procedure; B, C, D, E, or F and test Protocol; 1, or 2, when calling out this standard. In cases where either the Protocol or a pass/fail criterion is not designated, Protocol 1 shall be used.

[4] Reference to potential variability of the adhesion test has been made in various publications, including the assessment of variability completed for the test method found in ASTM Research Report RR:D01-1147.

5. Apparatus

5.1 *Adhesion Tester,* commercially available, or comparable apparatus to the specific examples listed in Annex A1 – Annex A5.

5.1.1 *Loading Fixtures,* having a flat surface on one end that can be adhered to the coating and a means of attachment to the tester on the other end. Optimal size of the loading fixture is determined by the adhesion tester capabilities. The fixture and tester combination should be chosen so that the expected maximum pull load the coating will be subjected to during the test is within the range of the tester.

5.1.2 *Detaching Assembly* (adhesion tester), having a central grip for engaging the fixture.

5.1.3 *Base,* on the detaching assembly, or an annular bearing ring if needed for uniformly pressing against the coating surface around the fixture either directly, or by way of an intermediate bearing ring. A means of aligning the base is needed so that the resultant force is normal to the surface.

5.1.4 Means of moving the grip away from the base to allow the loading of the fixture in as smooth and uniform a manner as possible and so that a torsion free, co-axial (opposing pull of the grip and push of the base along the same axis) force results between them.

5.1.5 *Timer,* or means of limiting the loading rate. A timer is the minimum equipment when used by the operator along with the force indicator in 5.1.6.

5.1.6 *Force Indicator and Calibration Information,* for determining the actual force delivered to the loading fixture.

5.2 *Solvent,* or other means for cleaning the loading fixture surface. Finger prints, moisture, oxides, and dust tend to be the primary contaminants.

5.3 *Sandpaper,* or other means, used to roughen the surfaces for glue application and adherence to the coating. When using sandpaper it is recommended to use 100 grit or finer.

5.4 *Glue*—the material used for securing the loading fixture to the coating. Two component epoxies and cyanoacrylates are two commonly used glues. Select a glue that does not affect the coating properties, flow through the coating or attack the coating.

5.5 *Clamps,* magnetic, mechanical, tape or similar, if needed for holding the fixture in place while the glue cures.

5.6 *Cotton Swabs,* or other means for removing excess glue and defining the adhered area. Any method for removing excess glue that damages the surface, such as scoring (see 6.7), must generally be avoided since induced surface flaws may cause premature failure of the coating.

5.7 *Scoring Tool,* circular hole cutter, or similar tool to score through to the substrate around the loading fixture.

6. Test Preparation

6.1 The method for selecting the coating sites to be prepared for testing depends upon the objectives of the test and agreements between the contracting parties. There are, however, a few physical restrictions imposed by the general method and apparatus. The following requirements apply to all sites:

6.1.1 The selected test area must be large enough to accommodate the specified number of replicate tests. The surface may have any orientation with reference to gravitational pull. Each loading fixture must be separated by at least the distance needed to accommodate the detaching apparatus. For Protocol 1 or to statistically characterize a test area, three or more replications are required.

6.1.2 The selected test areas must also have enough perpendicular and radial clearance to accommodate the apparatus, be flat enough to permit alignment, and be rigid enough to support the counter force. It should be noted that measurements close to an edge may not be representative of the coating as a whole.

6.2 Since the rigidity of the substrate affects results of the test and is not a controllable test variable in field measurements, some knowledge of the substrate thickness and composition should be reported for subsequent analysis or laboratory comparisons. For example, steel substrate of less than 3.2 mm (⅛ in.) thickness usually reduces test results compared to 6.4 mm (¼-in.) thick steel substrates.

6.3 Subject to the requirements of 6.1, select representative test areas and clean the surfaces in a manner that will not affect integrity of the coating or leave a residue. To reduce the risk of glue fracture affecting the test, the surface of the coating can be lightly abraded to promote adhesion of the glue to the surface. If the surface is abraded, care must be taken to prevent significant loss of coating thickness. Clean the area to remove particulates after abrading. Use of a solvent may be necessary to remove all contaminants. If a solvent is required, select one that does not compromise the integrity of the coating.

6.4 Clean the loading fixture surface as indicated by the apparatus manufacturer. Failures at the fixture-glue interface can often be avoided by treating the fixture surfaces in accordance with an appropriate ASTM standard practice for preparing metal surfaces for glue bonding.

NOTE 4—Guides D2651 and D3933 are typical of well-proven methods for improving adhesive bond strengths to metal surfaces.

6.5 Prepare the glue in accordance with the glue manufacturer's recommendations. Apply the glue to the fixture or the surface to be tested, or both, using a method and thickness recommended by the glue manufacturer. Be certain to apply the glue across the entire fixture surface. Position the fixture on the surface to be tested. Carefully remove any excess glue from around the fixture. (**Warning—**Movement, especially twisting, can cause tiny bubbles to coalesce into large holidays that constitute stress discontinuities during testing which may lead to glue fracture.)

6.6 Based on the glue manufacturer's recommendations and the anticipated environmental conditions, allow enough time for the glue to cure. During the glue set and early cure stage, a constant contact pressure should be maintained on the fixture. Magnetic or mechanical clamping systems work well, but systems relying on tack, such as masking tape, should be used with care to ensure that they do not relax with time and allow air to intrude between the fixture and the test area.

6.7 When scoring around the test surface is agreed upon between the purchaser and seller, extreme care is required to prevent micro-cracking in the coating or glue, since such cracks may cause reduced values. Scored samples constitute a different test procedure, and should be clearly reported with the results. Scoring may be required for thick-film coatings, reinforced coatings and elastomeric coatings. Scoring, if performed, can be completed before or after the load fixture is glued to the coating. When performed, scoring shall be done in a manner that ensures the cut is made normal to the coating surface, in a manner that does not twist or torque the test area or impart the loading fixture, and minimizes heat generation, edge damage, or microcracks to the coating or glue and the substrate. For thick coatings it is recommended to cool the coating and substrate during the cutting process with water lubrication.

NOTE 5—A template made from wood with a hole of the same size as the scoring tool drilled through it and secured to the surface may be an effective method to limit sideways movement of the scoring tool.

NOTE 6—-Scoring requirements will vary depending on coating system, chemistry, and thickness. A direct comparison of the unscored result to a scored result is one method to determine if scoring should be performed. Other methods for making this determination may be employed with agreement between the purchaser and seller. Scoring should not be considered for coatings less than 20 mils.

6.8 Note the approximate temperature, relative humidity, and other pertinent environmental conditions during the time of test.

7. Test Procedure

7.1 Test Methods:

7.1.1 *Test Method A (discontinued).*

7.1.2 *Test Method B — Fixed Alignment Adhesion Tester Type II:*

7.1.2.1 Operate the instrument in accordance with Annex A1.

7.1.3 *Test Method C — Self-Alignment Adhesion Tester Type III:*

7.1.3.1 Operate the instrument in accordance with Annex A2.

7.1.4 *Test Method D — Self-Alignment Adhesion Tester Type IV:*

7.1.4.1 Operate the instrument in accordance with Annex A3.

7.1.5 *Test Method E — Self-Alignment Adhesion Tester Type V:*

7.1.5.1 Operate the instrument in accordance with Annex A4.

7.1.6 *Test Method F — Self-Alignment Adhesion Tester Type VI:*

7.1.6.1 Operate the instrument in accordance with Annex A5.

7.2 Select an adhesion-tester with a detaching assembly and loading fixture size that has a force calibration spanning the range of expected values. Mid-range measurements are recommended, but read the manufacturer's operating instructions before proceeding. The adhesion tester shall be calibrated at the lesser of the manufacturer's recommended frequency or every three years.