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METHODS FOR EVALUATING ADHESIVE SYSTEMS AND ADHESION

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A wide range and variety of test methods are available to determine suitability of various adhesives to adhere dissimilar substrates for long use in the field. Test methods take into account various types of stress such as tension, compression and peel, and environmental conditions which the adhesive bond will encounter in its service life and predict the potential life of the bond. This report will discuss a variety of adhesive test methods to include advantages and disadvantages of each as well as the relation of the results to end performance.

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CONTENTS

FIGURES

INTRODUCTION

New product design presents engineers with the problem of predicting the durability and behavior of an assembly before it is ever marketed, and very likely, before it is even built. To provide a basis for making these predictions, engineers initiate and carry out test programs. The best way to test a new product is to build it as designed and use it as intended for its design lifetime. At best, such a procedure would be impractical and could be economically disastrous; therefore, design engineers must resort to relatively simple and short-time tests. All two frequently the tests and the beautiful plots, charts, and numbers produced become so engrossing that what they measure and how the measurements relate to the product developing is forgotten.

A wide range and variety of common products and structures, particularly military types, could not have achieved their present level of performance without adhesively-bonded substructures. The successful use of adhesives in these structures relies on a balanced interaction between design, testing, and experience.

Since the objective of any test program is to make predictions about the product, it is imperative that what each test measures, and the pitfalls of each test, be understood. Good judgment and common sense in relating the test results to the product's expected performance is necessary. This paper will discuss a variety of adhesive test methods, including advantages and disadvantages of each, as well as explain how the results relate to end item performance.

DISCUSSION

The most common test used in adhesive evaluations is the lap-shear test, ASTM D 1002. Test specimens are relatively inexpensive and easy to fabricate (fig. 1). The only test apparatus required is a tensile testing machine with self-aligning grips capable of maintaining a loading rate of 1,200 to 1,400 psi/min (8.3 to 9.7 MPa/min). Test specimens are bonded according to the manufacturer's instructions, as shown in figure 1, and are then tested to failure. The highest load achieved in units of psi or MPa is recorded and is usually referred to as the lap-shear strength.

A detailed examination of this test method reveals some of the problems inherent in all testing. A common assumption is that in the plane of the bond all loading is in shear. Because the lap-shear construction is asymmetric, application of the tensile load causes a bending moment which tends to deform the specimen. As a result, stresses are concentrated at the edges of the bond. At the ends of the bond, shear stresses have been calculated to be as much as six times the average applied stress. (Peel stresses are also concentrated at the edges of the bond. In addition, these stresses are not constant through the thickness of the adhesive, but tend to be concentrated near the surface of the continuous adherend.) Ultimately, if the concentrated forces exceed the yield strength of the adherend, the assembly can deform in such a way that the force applied by the tensile machine is no longer parallel to the plane of the bond. The bond then fails in a peel or cleavage mode beginning where the load is most concentrated at the edges. A considerable amount of information on lap-shear joint theory and experimental results is available in the literature (ref 1).

The ultimate bond strength of the standard single-lap joint depends not only on the adhesive used but on many other factors as well. Some of these additional factors include adhesive and adherend thickness, adherend surface preparation, modulus of the adherend, and yield strength of the adherend. In addition, slight modifications to the joint configuration can have significant effects on lap joint performance.

The most common method for measuring peel strength is ASTM D 1876, commonly call the T-peel test. A test specimen and the test panel from which it is made are shown in figure 2. Ideally, when the two ends of the "T" are pulled apart in the test, all stress is concentrated in a single line at the end where the bond is being destroyed. The load is applied at a constant speed of about 10 in./min (25.4 cm/min) and the load-versus-distance curve transmitted to a recorder. The average peel resistance can then be calculated from the area under the curve. Stiffness of the adhesive and adherends have significant effects on the results: the stiffer the adherend, the more the load tends to be distributed away from the center line at the leading edge of the bond, causing the apparatus to measure cleavage rather than peel. Stiff adhesives tend to reinforce flexible adherends. As a result, the peel angle does not remain perpendicular to the bond line. Peel strength can change as the peel angle changes.

These difficulties can be avoided by using ASTM D 3167, the floating-roller peel-resistance test. Part of the apparatus for testing, test panel, and test specimen are shown in figure 3. In this test, a flexible adherend is peeled away from a rigid adherend. The flexible adherend is bent around a 1 in. (2.54 cm) roller bearing in such a way that the peel area remains constant. As the flexible adherend is peeled away, the rigid adherend "walks" across the second roller so that the configuration at the leading edge of the bond remains constant. The major pitfall of this test method is if the flexible adherend is to rigid and does not conform well to the firm roller bearing, the peel angle is again uncontrolled. As with the T-peel test, the average peel resistance is calculated by integrating the area under the load versus distance curve.

ASTM D 950 is designed to measure the impact strength of an adhesive bond. This test measures the energy absorbed by a specimen when a 1 in.^{2} (6.45 cm^2) adhesive bond between two blocks of adherend is sheared by a single blow of a test machine hammer, usually a swinging pendulum. A metal-to-metal test specimen is shown in figure 4. Alignment of the sample fixtures is crucial for performing this test.

Misalignment can introduce cleavage forces and asymmetric forces on the bond line, which will influence test results. This alignment difficulty and large data scatter mandate larger sample sets for testing.

A basic shortcoming of these test methods is that they do not represent real life. Adhesive bonds are rarely designed to be used once or to fail the first time they are used, but this is the intent of each of these test methods. They cannot fully indicate the performance of an adhesive. Lap-shear tests will provide a basis for comparing one adhesive to another or will indicate whether an adhesive will bond to an adherend, but the end result is information about lap-shears only. However, the usefulness of the lap shear test to predict adhesive joint performance over a 20 yr period remains unclear. Although we may wish to use tests measuring the durability of bonded assemblies under intermittent or continuous load to better simulate real life conditions, these tests are not nearly as well developed as the simple tests described here. Furthermore, while endurance and fatigue tests provide a better indication of product performance, they are more time consuming and expensive to perform. Typically, the simple shear, peel, and impact tests are used for initial screening. The endurance (durability) and fatigue tests are conducted only on those adhesives and joint designs showing promise.

At least five ASTM standard practices and methods test adhesive bond durability. Atmospheric exposure of adhesive-bonded joints and structures, durability of adhesive joints stressed in peel, and durability of adhesive joints stressed in shear by tension loading are assessed by ASTM D 1828, D 2918, and D 2919, respectively.

In addition, ASTM D 3762 is a test method widely used for measuring adhesive bonded surface durability of aluminum as measured by the wedge test. This method is highly reliable in determining and predicting the environmental durability of the substrate surface preparation.

This method correlates well with service performance in a manner that is more reliable than conventional lap-shear or peel tests. The procedure is used primarily in aluminum-to-aluminum applications, but it may be used for other metals and plastics, provided consideration is given to thickness and rigidity of the adherends. A wedge is forced into the bond-line of a flat-bonded aluminum specimen, creating a tensile stress in the region of the resulting crack tip. The wedge and how it is driven into the specimen is shown in figure 5. The stressed specimen is then exposed to an aqueous environment, usually at an elevated temperature, or to an environment appropriate for the use of the bonded structure. The resulting crack growth is monitored after exposure to the aqueous environment. The effects of variations in adherend surface preparation can easily be seen when the specimens are opened, forcibly if necessary, at the conclusion of the testing. Failure modes (such as adhesive failure, adhesive-to-primer, or primer-to-adherend failure) should be noted at that time.

A variety of test environments may be used with the wedge test. A typical environment is 50°C (122°F) and condensing humidity. When the joint being tested is affected by the chosen environment significant crack growth normally occurs within 1 hr.

ASTM D 3166, fatigue properties of adhesives in shear by tension loading, can also provide reliable data useful for estimating the durability of bonded products. This test method covers the measurement of fatigue strength in shear by tension loading of adhesives on a standard specimen and under specified preparation, loading, and testing conditions. The test machine must be capable of applying sinusoidal cyclic loads to test specimens. The number of cycles to failure of the bond can be used to determine the life of the bonded specimen. A data bank can be assembled which, for example, might reveal that if the failing strength of the bond is 2500 psi (17.2 Mpa), a bonded assembly to withstand 500,000 cycles to 1500 psi (10.3 Mpa), or 10,000,000 cycles to 500 psi (3.4 Mpa) can be expected.

Cleavage tests are closely related to peel tests, discussed previously. ASTM D 1062 tests the cleavage strength of metal-to-metal adhesive bonds. Cleavage strength means the tensile load in lb/in. of width required to cause a 1-in. (25 mm) long separation of a test specimen. The specimens are usually in block form, are machined to specific dimensions, and include holes drilled to accept pins which attach the grips to the specimen. A tensile load is then applied at a rate of 600 to 700 lb/min (270 to 320 k/min). The maximum load in pounds (kilograms) carried by the specimen at failure is recorded, and the cleavage strength is expressed in lb/in. Peel tests are often preferred over cleavage tests because they require less equipment to perform.

ASTM D 3807 covers the strength properties in cleavage peel by tension loading of engineering plastics. Cleavage peel strength is defined as the average load per unit width of bond line required to produce progressive separation of two bonded, semirigid adherends under conditions designated in the procedure. The bonded test panels are cut into 1 in. by 7 in. (25 mm by 177 mm) test specimens. These are then bonded for approximately 3 in. (76 mm) of their length. A tension testing machine is used to apply a load at a constant cross-head speed of 0.5 in./min (12.7 mm/min). The average load in kilonewtons per meter width of specimen required to separate the adherends is determined for the first 2 in. (50.8 mm) of cleavage/peel after the initial peak. This determination is best made from the autographic curve produced by the tension testing machine, using a planimeter.

ASTM D 3433 is a standard practice for determining fracture strength in cleavage of adhesives in bonded joints. The method measures fracture strength of a bonded joint which is influenced by adherend surface preparation, adhesive, adhesive-adherend interactions, and primers. The practice involves cleavage testing of bonded specimens. A tensile force is applied normally to the surface of a crack, causing it to extend.

The load versus load displacement across the bond-line is used to calculate the opening mode fracture toughness and the crack arrest toughness. ASTM D 5041, a newly adapted test method, covers the same objective on adhesively-bonded reinforced flat plastic specimens. This method also calls for a semiquantitative observation of failure modes. Although this is not the widely used wedge test, a wedge is used in the test.

Creep tests of adhesives are covered in ASTM-D 1780 and test methods D 2293, D 2294, and D 4680. ASTM D 1780, the standard practice for metal-to-metal adhesives, covers determining the amount of creep of metal-to-metal adhesive bonds resulting from the combined effects of temperature, tensile shear stress, and time. In this practice, creep is defined as the time-dependent part of the strain that results from exposure to a constant temperature and load. The test specimens are similar to those used in the lap-shear test, ASTM D 1002, except that the specimen length is 5 in. (12.7 cm). Three fine scribe lines are made across the machined vertical edge of the prepared specimen, one across the center of the lap joint and the other two at a distance of 0.030 in. (0.76 mm) from the ends of the lap joint within the lap section. After applying a prescribed load using a direct dead weight or lever procedure, the deformation is measured directly at various time intervals to produce a smooth time-deformation curve. This is accomplished by observing the displacement of the three scribe lines using a calibrated microscope. After a suitable period of time, the load is removed and the specimen is permitted to recover. The adhesive shear stress is reported in MPa or psi and the deformation is given at specific time intervals. The creep rate in in./hr (or mm/hr) is also calculated.

ASTM D 2293 covers creep measurements of adhesives in shear by compressing loading (metal-to-metal); a spring-loaded testing apparatus is used. ASTM D 2294 covers creep measurements in shear by tension loading (metal-to-metal); a different type of spring-loaded apparatus is used in this procedure. ASTM D 4680 is a relatively new (1987) practice for determining creep and time to failure of adhesives in static shear. It uses compression loading (wood-to-wood). The spring-loaded test apparatus is somewhat similar to that used in ASTM D 2293 for metals.

Flexural strength of adhesive-bonded laminated assemblies is determined using ASTM D 1184. This method determines the comparative properties of adhesive assemblies when subjected to flexural stresses with standard shape specimens under specific conditions of pretreatment, temperature, relative humidity, and testing technique. The test specimens are rectangular pieces, 1.5 in. (38 mm) long and 0.75 in. (19.1 mm) wide, machined from laminated panels consisting of eight piles of 0.01 in. (0.3 mm) thick adherend material. Both sides of each ply are coated with an even spread of adhesive and bonded. All specimens are conditioned at 23°C (73.4°F) and 50 relative humidity (18 hr for metal and plastic and 7 days for wood). Testing is carried out under the same conditions. The specimens are tested as simple beams loaded at mid-span.

In ASTM D 3111 on flexibility of hot-melt adhesives by a mandrel-bend test method, properly sized and conditioned strips of adhesive are bent 80 deg over a cylindrical mandrel (rod). The test is repeated with fresh adhesive strips using mandrels decreasing in diameter until the adhesive fails on bending. The flexibility of the adhesive is the smallest-diameter mandrel over which four out of five test specimens do not break. ASTM D 4338 covers flexibility of supported adhesive films by a mandrel-bend test. While the mandrel-test apparatus is identical to that used in ASTM D 3111, in this procedure a steel test substrate coated with a film of adhesive, properly sized and conditioned, is bent 180 deg over a cylindrical mandrel. The test is carried out in the same way as in ASTM D 3111.

A concise compilation of ASTM test methods used in adhesive bonding evaluations can be seen in the Adhesives Technology Handbook (ref 2, now undergoing revision). It should enable the reader to quickly identify relevant ASTM test methods for a number of adhesive testing areas. An ARP*, issued by the Society of Automotive Engineers, is included in that listing.

The 58 test area subjects are listed alphabetically, with some appearing under more than one heading. The complete numerical designation is given; for example, under "Aging" in ASTM D 1183-70 (1987) the "-70" refers to the year of issue or current revision, and (1987) is the year of reapproval with no significant changes in the text. If no data appears in parentheses for a listing then the standard has not been reapproved since the last revision. All ASTM standards were issued by ASTM Committee D-14 on Adhesives and all ASTM D-14 standards are published in the Annual Book of ASTM Standards, Volume 15.06 on Adhesives (ref 3). Copies of individual standards are available as "singles" from ASTM.

The test methods most commonly used to evaluate adhesive bonds are ASTM D 1002 (shear strength), ASTM D 950 (impact strength), and ASTM D 1876 and ASTM D 3167 (peel strength). Although each procedure is useful, it is limited. There is no ASTM method which considers two different forces simultaneously. Each test method will take into effect one particular force and not the combination of the two forces. We shall look at each of these, as well as ASTM D 3166, which is designed to measure fatigue properties of adhesives in shear and to yield information on the endurance of bonded assemblies under static load.

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*ARP's are Aerospace Recommended Practices, published by the Society of Automotive Engineers, Warrendale, PA.

CONCLUSION

The objective of all the tests is to provide a basis for making some predictions about the reliability and durability of adhesively bonded specimens. Tests can be simple, complex, elegant, expensive, inexpensive, fun, or very boring. Most tests do provide some useful information about the adhesive and the adhesively bonded structures if no one takes care to realize what the test means and what the pitfalls may be when interpreting the results. Adhesive manufactures have developed a great deal of information and are delighted to share it with potential users. Often the manufacturers and users can work together to design new tests to measure specific properties of bonded assemblies. This is normally done by members of ASTM Committee D-14 on Adhesives. Care and diligence in setting up test programs and cooperation with adhesive manufacturers can lead to improved products, new and innovative uses of adhesives, higher productivity, lower cost, and more satisfied customers.

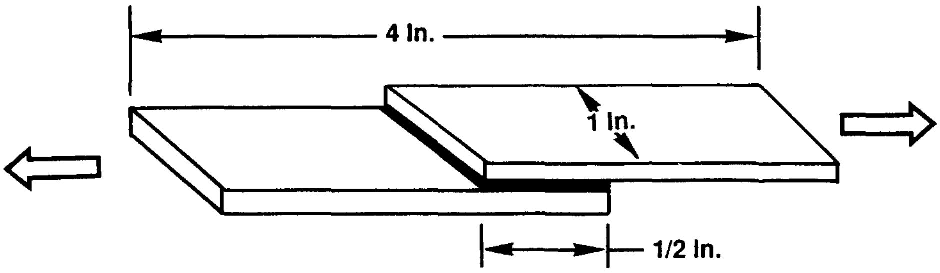
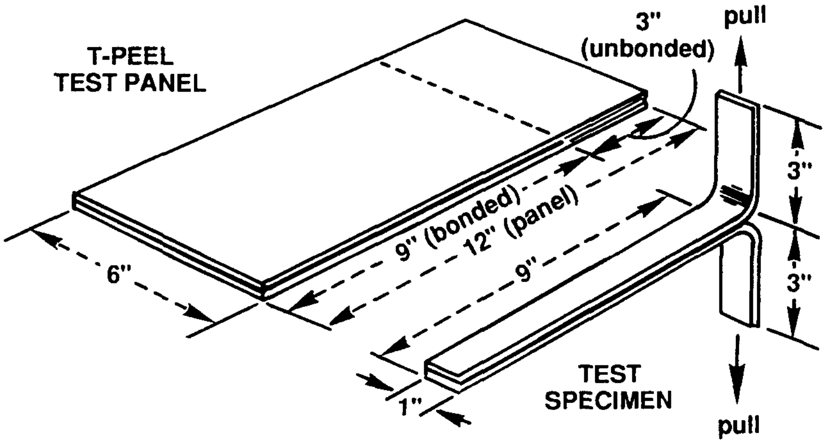
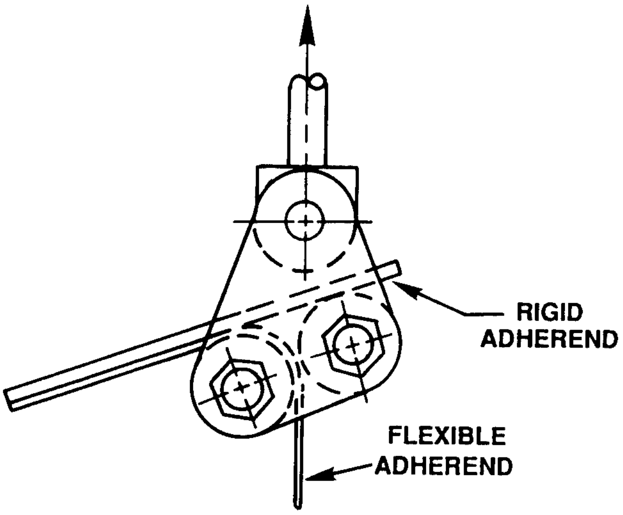


Figure 1
Test specimens for ASTM D 1002 lap-shear test

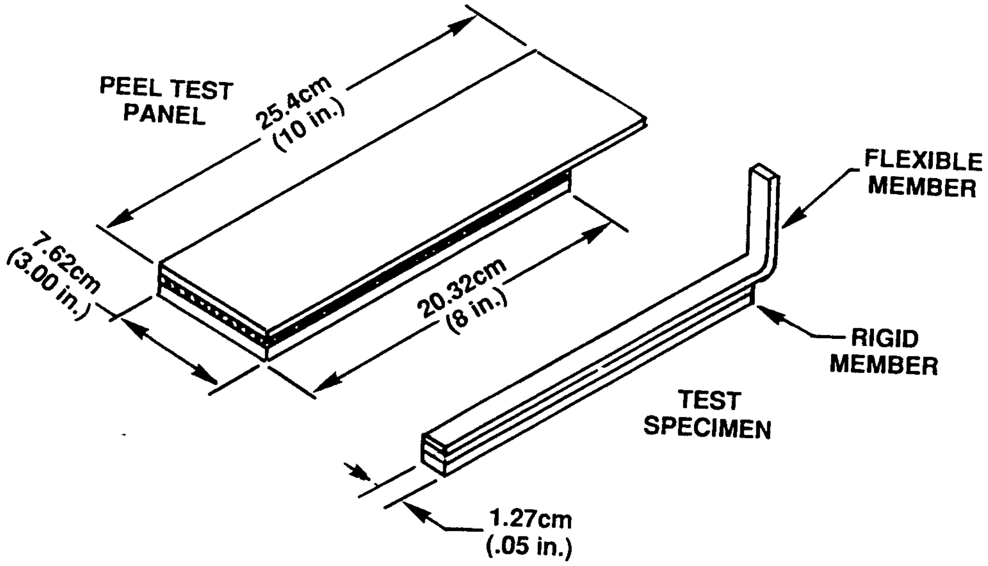


METRIC EQUIVALENTS					
in.	1	3	6	9	12
mm	25	76	152	229	305

Figure 2
Test panel and test specimen for T-peel test

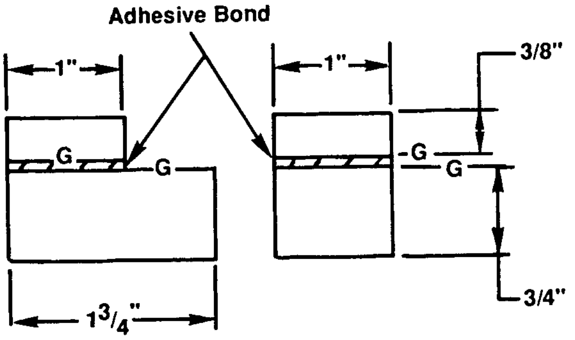


a
Roller drum test fixture



b
Test panel and test specimen

Figure 3
Peel test

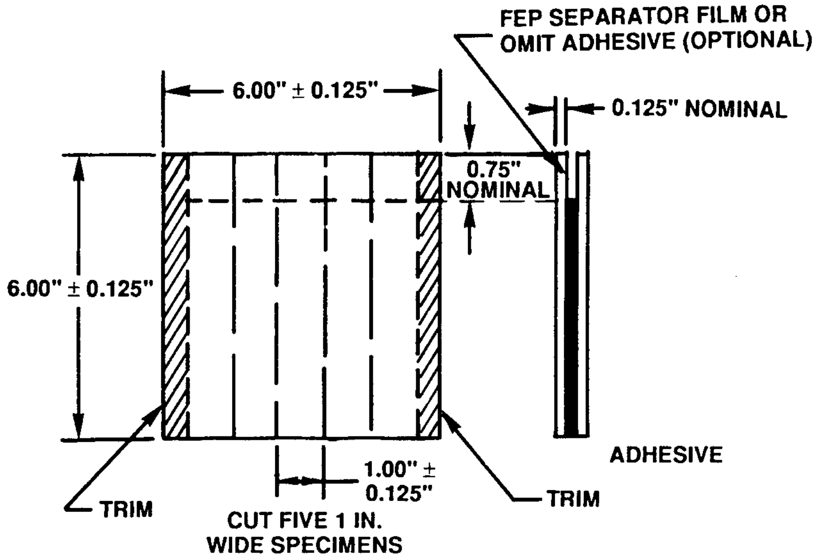


METRIC EQUIVALENTS				
in.	1/4	3/4	1	1 3/4
mm	9.5	19	25.4	44.4

(a) Metal-to-Metal Specimen

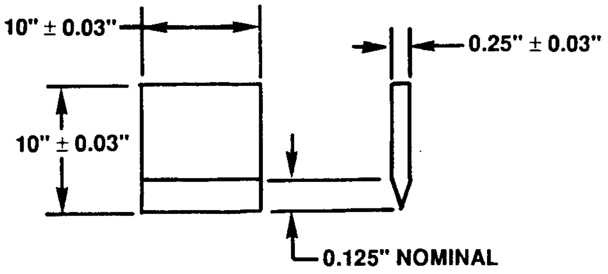
Figure 4
Block shear impact test specimen (metal-to-metal adherends)

WEDGE TEST SPECIMEN ASSEMBLY



WEDGE TEST SPECIMEN CONFIGURATION

ALUMINUM OR STAINLESS STEEL WEDGE



WEDGED CRACK EXTENSION SPECIMEN

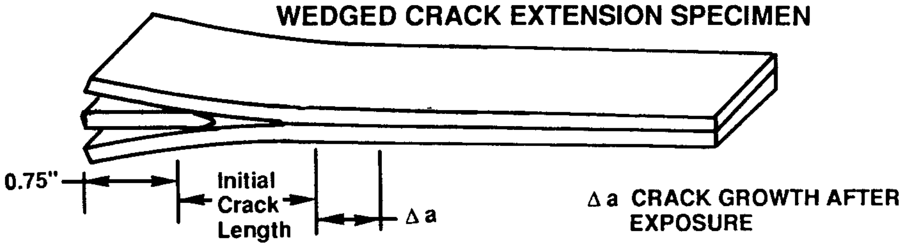


Figure 5
ASTM D 3762 crack extension specimen configuration

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3. American Society for Testing and Materials (ASTM), 1916 Race Street, Philadelphia, PA 19103, Annual Book of Standards, Volume 15.06 Adhesives.

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