NISTIR 4958

Quality Control Tests for Adhesion of Paint on the Panels of Tactical Rigid Wall Shelters, Phase II

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March 1993
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U.S. Department of Commerce
Ronald H. Brown, *Secretary*

National Institute of Standards and Technology
Raymond Kammer, *Acting Director*

Prepared for:
U.S. Army Natick Research, Development and Engineering Center
Tactical Shelters Branch
Natick, MA 01760

ABSTRACT

At the request of the U.S. Army Natick Research, Development and Engineering Center, a practical method was developed for measuring the adhesion of paints applied to shelters. As recommended in the Phase I report, a pull-off test based on the use of a commercially available pneumatic testing device was chosen. The procedure includes ways of controlling the substrate stiffness, a parameter that affects the test results, and a process for obtaining acceptable levels of adhesion for a particular substrate. The estimated precision (standard deviation) of the method is 9 percent. In a pilot study to determine the extent to which small differences in surface preparation would affect differences in pull-off test results, it was found that, at least for aluminum, the procedure was insensitive to small differences in surface preparation.

Key Words: adhesion; adhesion tests; aluminum; bond strength; building technology, coatings; paint; relocatable structures; tactical rigid wall shelters; test method

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1. INTRODUCTION

1.1 Background

The U.S. Army uses a range of tactical rigid-wall shelters in situations requiring highly mobile work, living, or storage facilities. The exterior and interior aluminum-skin surfaces are painted during the manufacturing process. Service paint failures associated with poor adhesion have been identified. Although a tape adhesion test [1] has been used for quality control, it has been deemed undesirable. It is not a quantitative test and the results depend upon the adhesion of the tape to the painted surface, a factor that is difficult to control [2]. Thus, at the request of the U.S. Army Natick Research, Development and Engineering Center, the National Institute of Standards and Technology (NIST) conducted a study to develop an improved procedure for assessing the adhesion of paint on tactical shelters.

The study was conducted in two phases. In Phase I, the essential and desirable attributes of adhesion tests were identified and the state of technology of tests that measure bonding properties of paints were assessed in a report titled "Quality Assurance Tests for Adhesion of Paint on Tactical Rigid Wall Shelters" [3]. The essential attributes identified were ability to determine adequacy of adhesion, reproducibility, low cost, ease of use, operator-independence, and safety. Desirable attributes identified were ability to provide quantitative results non-destructively, applicability to both flat and curved surfaces, and capability of providing information on failure mode (i.e., adhesive vs cohesive). The recommendations of the Phase I study included using a pull-off test for assessing bond strength, developing a draft standard procedure for its use and investigating the feasibility of using a non-destructive evaluation procedure for assuring bond strength.

The objectives of Phase II were to follow up on the recommendations of Phase I by developing a detailed procedure for using a pull-off test for assessing the bond strengths of paints on shelters and preparing a draft standard test method, as well as discussing the feasibility of using a non-destructive evaluation procedure. The approach to developing the pull-off test was to examine existing data on commercially available portable adhesion-strength test equipment, examine factors or conditions that might affect the test results, and develop data regarding expected pull-off strengths for paints used on shelters. This report describes the results of these studies and provides a draft standard for assessing the adhesion of paints on shelters. For the non-destructive evaluation procedure, the literature was reviewed to provide information for a recommendation in this area.

1.2 Characteristics of Mechanical Adhesion Tests

As implied in the Phase I report [3] and discussed in detail by Mittal [4] and others [5, 6, 7], mechanical adhesion-test procedures (e.g., pull-off, peel and lap shear tests) provide measures of the force or work of detachment or separation of a paint from a substrate; they are a measure of "practical adhesion" but not of interfacial adhesion (the sum of all intermolecular or interatomic interactions between a paint and a substrate), per se. This is because separation takes place at the weakest part of the system, not necessarily at the paint/metal interface and because mechanical adhesion measurement procedures depend upon the parameters of the test procedure, including rate of applied force, stiffness of the substrate, temperature, and paint type and thickness.

As for any test procedure, it is important to understand the sensitivity of the procedure to changes in the parameters. This is especially important when tests are being carried out in an environment, such as a factory, in which it is difficult or not feasible to keep the parameters constant. Some data are available on the effects of temperature, application rate of the applied force, and substrate stiffness on mechanical-test results. As for the temperature effect, data reported for several adhesives show that the strength of single-lap joints loaded in tension did not vary by more than 10 percent for temperatures from about 15 to 30°C [8]. With respect to the rate of application of the of applied force, changes in the rate (over a range covering more than two orders of magnitude) had essentially no effect on the pull-off results for a poly(vinyl chloride)-coated steel substrate, as shown in Table 1 [9]. The measurements were made using a laboratory tensile testing machine and the substrate was stiffened with a pull-off fixture adhered to the backside of the test panel, as described in ISO 4640 [10]. Together, these data indicate that pull-off strength results should be only minimally affected by the small variations ($< 10°C$) in ambient temperature and rate of application of the applied force in the range expected to occur in a factory environment. Thus, no further investigation of the effects of small variations in either of these two parameters on test results was done in this study.

Changes in the configuration of pull studs and substrate thickness, however, have been shown to have large effects on pull-off strength results [11, 12]. This is illustrated in Figure 1 where the pull-off strength results for an epoxy applied to sheet steel of varying thicknesses are reproduced from reference 11. Sickfield [12] has explained this relationship by noting that the bending moments in the substrate (commonly referred to as stiffness) at the periphery of the pull-stud decrease as the substrate thickness increases. Hence, it is essential to understand the effect of the stiffness or rigidity of the substrate (resistance to bending) when conducting in-situ testing of shelters, since the stiffness of the substrate at a given point of test will depend upon factors such as the type of construction of the panel, the location of the test area, and the thickness and alloy type of the aluminum skin. Stiffness effects were investigated by varying aluminum substrate thickness and configuration as described in Section 3.1.

2. EXPERIMENTAL

2.1 Design

Experiments were conducted to address the following issues: effect of substrate stiffness on the results, effect of honeycomb core on substrate stiffness, sensitivity of pull-off strength results to slight variations in surface preparation, and typical pull-off strengths of paints applied to shelter skins.

Table 1. Relationship Between Rate of Application of Load and Pull-off Strength of a Paint on Steel [9]

Rate of Application of Load (Cross-Head Speed), mm/min	Pull-off Strength, MPa
1	12
10	15
200	15

2.3 Equipment

Pneumatic testing devices were selected for this study after examining the precision data for portable pull-off testing devices for paints on steel obtained in an ASTM round-robin (see Table 2) [13]. As shown in the table, the estimates of coefficient of variation for results obtained using a pneumatic device are lower than that for the other devices, either mechanical or hydraulic, when tests are conducted as described in ASTM D 4541. The PATTI[1], a commercially available pneumatic adhesion tester, and an experimental pneumatic device [14] were used in this study. Both met the requirements of the test method for pneumatic devices. The experimental device, developed by NIST, was used when the pull-off strength of the samples was expected to exceed the capacity of this laboratory's PATTI. (The capacity of either of these devices can be increased by increasing the ratio of the working area of the piston (an air-filled chamber) to the area of the pull-stud.)

The effective areas of the two pistons were 2,600 mm^2 (4 in^2) and 3,600 mm^2 (5.6 in^2) for the PATTI and the experimental device, respectively. The area of the pull-stud used with either of the pistons was 130 mm^2 (0.2 in^2). With a maximum air supply pressure of 0.7 MPa (100 psi), the corresponding maximum stresses that can be applied to a paint by a pull-stud are about 14 MPa (2000 psi) or 20 MPa (2900 psi), respectively for the two pistons. When either test device was used, a two-part epoxy adhesive was used to adhere the pull-stud to the surface.

[1] Certain manufacturers' names of commercial equipment are identified in this report to adequately describe the experimental procedure. Such an identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the equipment, instruments, or materials identified are necessarily the best available for the purpose.

Table 2. Precision of Pull-Off Strength Measurements Using Portable Devices[1]

Instrument	Coefficient of Variation, %	Degrees of Freedom
Intralaboratory		
Pneumatic Tester	8.5	48
Mechanical Tester 1 Hydraulic Tester Mechanical Tester 2	12.2	129
Total		177
Interlaboratory		
Pneumatic Tester	8.7	20
Mechanical Tester 1 Hydraulic Tester Mechanical Tester 2	20.6	58
Total		78

[1]From ASTM D 4541 draft research report [13].

2.4 Experimental Procedures

2.4.1 Substrate Stiffness

To investigate the effects of the thickness of the aluminum skin of a shelter panel and the panel construction, i.e., foam core or honeycomb, two sets of experiments were conducted. In one, three different thicknesses (1.6, 3.2, and 9.5 mm) of sheet aluminum (used to represent a skin) and two unpainted aluminum-skinned foam-core panels, having aluminum skin thicknesses of either 1.2 or 2.8 mm (0.5 or 0.11 in), were used. The thicknesses of the aluminum skins were measured using a micrometer, capable of being read to the nearest 2.5 μm (10^{-4} in). The aluminum surfaces were prepared for painting by degreasing with acetone and hand-sanding with 180 grit abrasive paper. An alkyd paint, TT-P-102, was applied using a drawdown blade to achieve a nominal 25 μm (10^{-3} in) dry film thickness. The paint was allowed to dry for 4 $\pm$ 0.5 hours at room temperature and to cure for 44 $\pm$ 1 hours at 50°C in an air-fed oven. Four pull-studs were attached to each thickness of aluminum substrate using an epoxy adhesive. Pull-off tests were conducted as described in ASTM D 4541.

In the second set of experiments, the potential effect of the configuration (size and shape) of the cells of the honeycomb core on the local stiffness of an aluminum skin of a shelter panel was investigated. As illustrated in figure 2, a pull-off stud could be positioned in different locations relative to the cell walls of the honeycomb. It was considered that the stud position relative to the honeycomb could affect the local stiffness of a skin and consequently the pull-off results, depending upon the size of the cells of the honeycomb. To investigate this potential effect, six pull-studs were attached to each of eight factory-coated honeycomb panels. The largest openings in the honeycomb were about 6 mm (0.25 in). Since the configuration of the honeycomb could not be determined from the coated surface, the pull-studs were positioned randomly across the panels. A significant effect of the honeycomb configuration on the pull-off results would result in an increased coefficient of variation of the replicate measurements as compared with that of replicate measurements on a substrate of uniform stiffness. The coefficient of variation of results obtained from the pull-off strength testing of aluminum skins in which the stiffness could assumed to be constant (i.e., skins stiffened by gluing to plywood) was used in the comparison.

2.4.2 Surface Preparation Study

Studies show that adhesion failures of painted surfaces in service are often related to surface preparation [15]. If improper surface preparation were found to significantly affect the initial (e.g., within a few days following application) pull-off strength of a paint, the pull-off test could supplement quality control procedures for surface preparation. Therefore, the sensitivity of the method to variations in surface preparation of the aluminum prior to painting was investigated. The surfaces of three sets of specimens were prepared in different ways and painted. Pull-off tests were then conducted. The substrates were made of 2024-T4 aluminum; their dimensions were 100 mm x 100 mm x 6 mm (4 in x 4 in x 0.25 in). In all three cases, the substrates were first degreased by repeated rinsing with methylene chloride. No further cleaning was done on the first set. The second set was treated with a sulfuric acid solution as described in ASTM E 864 [16] following the degreasing. The third set was cleaned in the same manner as the second set but was then contaminated with a thin layer of castor oil. The contamination was achieved by applying an oil/ethanol solution (about 0.1 percent by mass) to the surface of the etched aluminum using a drawdown blade having a 125 μm (5×10^{-3} in) clearance. The theoretical oil thickness on the surface was about 100 nm (4×10^{-6} in). Visually, the surface appeared oily. The cleaned and treated substrates were kept in a desiccator prior to painting. A two-part polyamide epoxy paint was applied to each substrate using a drawdown applicator having a 125 μm (5×10^{-3} in) clearance. The resulting dry film paint thickness was 75 μm (3×10^{-3} in). The paint was allowed to cure for six days at ambient laboratory conditions prior to adhering the pull-studs using an epoxy adhesive cured at 75°C for 30 minutes. The pull-off strength of each specimen was measured according to D 4541.

3. RESULTS AND DISCUSSION

3.1 Effect of Stiffness

The results of measuring the pull-off strength of painted aluminum substrates of varying thicknesses are shown in Figure 3 and Table 3. Pull-off strengths ranged from about 1 MPa (145 psi) to 15 MPa (2200 psi). All the failures were cohesive in the paint film and they were closer to the aluminum surface for thinner substrates as compared to thicker substrates. The specimens having substrate (i.e., skin) thicknesses of 1.2 mm (0.04 in) and 2.8 mm (0.1 in) were the skins of a foam-core shelter panel. As is evident in Figure 3, the foam core does not appear to substantially stiffen the aluminum substrate as the pull-off strengths of paints applied to the skins of these foam-core panels follow the same trend as those applied to aluminum substrates.

The results of the measurements made on specimens of shelter panels containing honeycomb are shown in the Table 3 along with results from the painted aluminum skins that were glued to plywood to increase their stiffness [3]. The pull-off strengths of the interior and exterior sides of the honeycomb panel were different, with the green exterior paint having a pull-off strength of 6.1 MPa (900 psi) and the interior paint a pull-off strength of 9.1 MPa (1340 psi). The coefficients of variation ranged from 5 to 11 percent for the honeycomb panels and from 2 to 16 percent for aluminum substrates glued to plywood.

The Bartlett test of homogeneity of variance, as described in [17], was used to determine whether there was a significant difference between the variances (squares of standard deviations) for measurements taken on aluminum-skin honeycomb panels and on aluminum skins adhered to plywood. For a 5 percent level of significance, the hypothesis that all the results were the same could not be rejected. Thus, the position of the pull-stud with respect to the honeycomb configuration did not significantly affect the experimental pull-off strength results, as determined using this measurement procedure. That is, with this particular honeycomb and aluminum-skin configuration in which the cell and stud size were about the same, no evidence of local stiffening of the aluminum skin by the honeycomb was detected. Using both data sets, the pooled estimate of the coefficient of variation of the test procedure is 9 percent which is the same as determined in the ASTM D 4541 round-robin for a pneumatic device [13].

Table 3. Pull-off Strengths of Paint on a Variety of Aluminum Substrates and Configurations

Sample Set	Number of Specimens	Mean Stress at Failure, MPa	Coefficient of Variation, %
Paint on 1.2 mm thick aluminum, foam core	4	1.5	2.8
Paint on 1.6 mm thick aluminum	4	2.1	5.2
Paint on 2.8 mm thick aluminum, foam core	4	3.6	4.9
Paint on 3.2 mm thick aluminum	4	5.7	7.4
Paint on 9.5 mm thick aluminum[2]	4	15.0	10.3
Honeycomb panel - Green Paint, Side 1	12	5.7	6.9
Honeycomb panel - Green Paint, Side 3	6	6.3	11.2
Honeycomb panel - Green Paint, Side 5	6	6.3	11.2
Honeycomb panel - Green Paint, Side 7	6	6.2	10.0
Honeycomb panel - White Paint, Side 2	6	9.4	9.1
Honeycomb panel - White Paint, Side 4	6	9.5	5.0
Honeycomb panel - White Paint, Side 6	6	8.4	6.4
Honeycomb panel - White Paint, Side 8	6	9.0	9.2
Skin glued to plywood, Interior paint[1]	5	7.3	15.6
Skin glued to plywood, Interior paint[1]	3	8.9	11.6
Skin glued to plywood, Interior paint[1]	4	10.7	16.1
Skin glued to plywood, Exterior paint[1]	5	6.5	5.4
Skin glued to plywood, Exterior paint[1]	5	7.1	9.1
Skin glued to plywood, Exterior paint[1]	5	6.9	1.7

[1]Data from reference 2, NISTIR 90-0610

[2]Value exceeded range of pneumatic device; pull-off strength determined using laboratory tensile testing machine.

3.2 Surface Preparation

The results of the surface preparation study are shown in Table 4. The mean pull-off strengths for the three sets of specimens showed little variation, ranging from 19.3 to 19.9 MPa (2800 to 2900 psi). All failures were cohesive within the paint film. However, there were differences in the appearance of the failed surface. For the panels in which the aluminum had been acid etched and not contaminated, the surface of the aluminum was hidden by the paint. For most of the specimens of the other two sets, the aluminum substrate was visible over part of the test area through a very thin layer of paint indicating failure closer to the aluminum substrate. This may indicate that the oil contamination caused a weakening of the so-called paint boundary layer (paint just next to the substrate) decreasing its cohesive strength.

Table 4. Pull-off Strengths of a Paint on Aluminum Prepared Using Different Procedures

Method of Surface Preparation	Mean Stress at Failures, MPa	Coefficient of Variation, %
Degrease Only	19.6	3.2
Degrease and acid etch	19.9	0.6
Degrease, acid etch and oil contaminate	19.3	2.0

The data were analyzed using the Duncan Multiple Range Test [17] to determine if there were significant differences among the means. Using only the data from these tests to calculate the estimated variance of the test procedure, the differences between the results obtained for surfaces which were only degreased and those which were contaminated with oil are different (5 percent level) from those that were degreased and acid etched. The results of the degreased only and the oil-contaminated surfaces were not different at a 5 percent level of significance. However, the differences in the pull-off strengths are small, and would not have been significant if the estimate of the variance of the test procedure had been calculated by pooling the data shown in Table 3. (The difference in the estimates of variance is probably due to the types of specimen and the period of time over which the measurements were made. For the surface preparation study, all of the pull-off strengths were obtained in one day on similar specimens; the other data were obtained from several types of specimens and taken over a period of several months.)

These small differences in pull-off strengths for specimens properly and improperly prepared indicate that this test would be unacceptable as a quality control tool to detect small differences in surface preparation. From other studies, it appears that the sensitivity of a mechanical test to differences in surface preparation can be increased in two ways. First, the type of test may affect the failure mode and a peel test may be more sensitive to different types of surface preparation, or surface contamination. Rossiter [18] found that, for seams of single-ply roof membranes, a T-peel test was much more sensitive to various types of contamination than a lap-shear test. Additionally, the sensitivity of the test could perhaps be improved by subjecting specimens to immersion in water prior to testing. Kinloch [10] has reported that the initial lap-joint strengths of epoxy/aluminum alloy joints were the same for three chemical treatments (45 MPa), but ranged from about 30 to 40 MPa (4400 to 5900 psi) after 1000 h of immersion in water at 50°C.

3.3 Acceptable Level of Pull-Off Strengths

A value for an acceptable level of pull-off strength is needed for a pull-off test to be part of an overall quality control procedure. However, based upon the results presented above, an acceptable level will depend on, as a minimum, the skin stiffness at the location of the test. As discussed, in this study pull-off strengths for the same paint system varied from 2 Mpa to 16 MPa (2350 psi), depending upon the substrate thickness (or effective stiffness). This does not mean that a test for pull-off strength could not be used in quality control. However, it does mean that the parameters of the test must be kept constant when making comparisons of pull-off strength, and acceptable values may need to be determined for each case.

Control of stiffness of a painted shelter substrate (panel) for pull-off test purposes could be accomplished in at least two ways. First, the test could be conducted in the same specific location for a given shelter type, e.g., midway between two window openings or over a structural beam adjacent to a doorway. Second, a test specimen (i.e., a piece of sheet aluminum like that used in making shelter wall or roof panels) could be carried along in the manufacturing process receiving the same pretreatments and paint as a shelter panel and tested in a prescribed way. The test requirements would include a procedures for stiffening the sheet, if appropriate. In either case, acceptable values of pull-off strength and failure type would have to be determined based upon properly performing paints.

Other parameters (not investigated in the present study) that should be considered in selecting an acceptable pull-off strength include paint type and thickness. Although, in this study, large differences in cohesive strengths of paints were not observed, variations have been reported. For example, Walker [19] reported variations of pull-off strength from 20 MPa (2900 psi) for an alkyd paint to 32 MPa (4600 psi) for an epoxy. All failures were cohesive within the paint. The paints were applied to an aluminum substrate which was effectively stiffened prior to the test. As to paint thickness, Sickfield [12] reported that for reactive systems, the change in pull-off strengths was less than about 10 percent for paint thicknesses from 100 to 300 μm. Thus, if possible, acceptable pull-off strength values should be determined for each paint system.

3.4 Feasibility of Non-Destructive Testing Procedures

The literature was reviewed to investigate the possibility of using non-destructive evaluation procedures for assessing bond strength of paints on shelter panels. It contains many references to non-destructive procedures to detect delaminations in composite materials [20,21,22], but is contradictory regarding whether existing methods can assess bond strength (e.g., will not work [23], and can work [24]). A recent paper highlights one of the difficulties; that is characterizing the various mechanical properties and morphological conditions that may affect the output of the method [25]. This information is needed in order to prepare samples having varying bond strengths while keeping other parameters that affect test response, but are unrelated to bond strength, constant. Other difficulties are preparing specimens with varying bond strength and characterizing the bond strength, per se. As discussed above, mechanical adhesion methods depend upon material and procedure parameters, in addition to bond strength. However, as evidenced by the number of papers published and conferences held on this subject, many researchers are working in the area and advances are being made. Procedures may soon be available for quality control use for assessing bond strength of paint on aluminum.

4. RECOMMENDATIONS

Based on this study, the following recommendations are made:

- Adopt a pull-off test method as a quality control test for paint adhesion for shelters, realizing that a test carried out shortly after cure of the paint system will not provide a sensitive measure of the quality of surface preparation. Develop the specific test procedures and program needed to implement the test. These tests could be conducted on either a specific area of the shelter or on a separate panel carried along through the manufacturing process. In either case, an acceptable value of pull-off strength should be established for a particular paint system. The acceptable value must be determined using the same testing parameters that would be used in the quality control procedure and be obtained from a similar paint system that is performing satisfactory. A suggested method for carrying out pull-off strength tests is presented in the appendix.

- Validate the draft ASTM procedure for use of a pneumatic pull-off strength (adhesion) test for assuring quality of paint/aluminum adhesion.

- Based on the preliminary results that the three different surface preparations had only a small affect on the pull-off strength of the system, carry out further laboratory studies to investigate the feasibility of including a short aging test in the quality control procedure to improve the sensitivity of the test to small differences in surface preparation or oil contamination. For example, based on the literature, immersion in water for a short time may improve the sensitivity of a pull-off strength test to differences in surface preparation of the metal. Additionally, investigate the sensitivity of a peel test to differences in surface preparation. This recommendation is analogous to that found for the roofing industry.

- Continue to review the literature on the use of non-destructive evaluation procedures for assessing bond strength and initiate a laboratory study when there is additional evidence that a technique may be practical for quality control.

5. ACKNOWLEDGMENTS

This study was supported by the U.S. Army Natick Research, Development and Research Center. The authors extend their thanks to David Mikelson for his assistance in providing research support, guidance and specimens. The authors also thank Geoffrey Frohnsdorff and Robert Mathey for informative discussions and review of the document.

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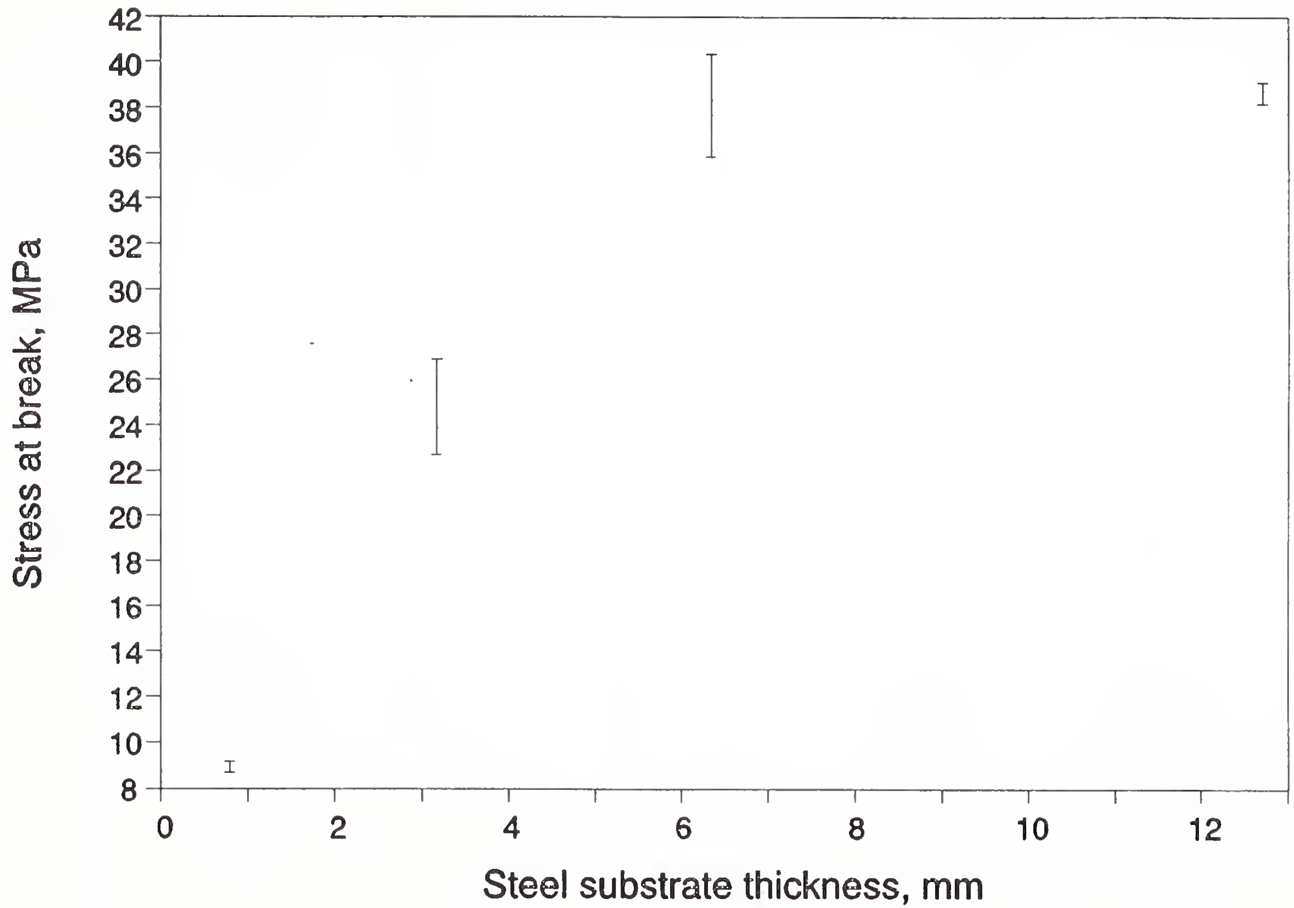

Figure 1. Results of pull-off measurements of an epoxy adhesive applied to sheet steel of varying thicknesses. The limits are $\pm\ 2\sigma$ for each data set.

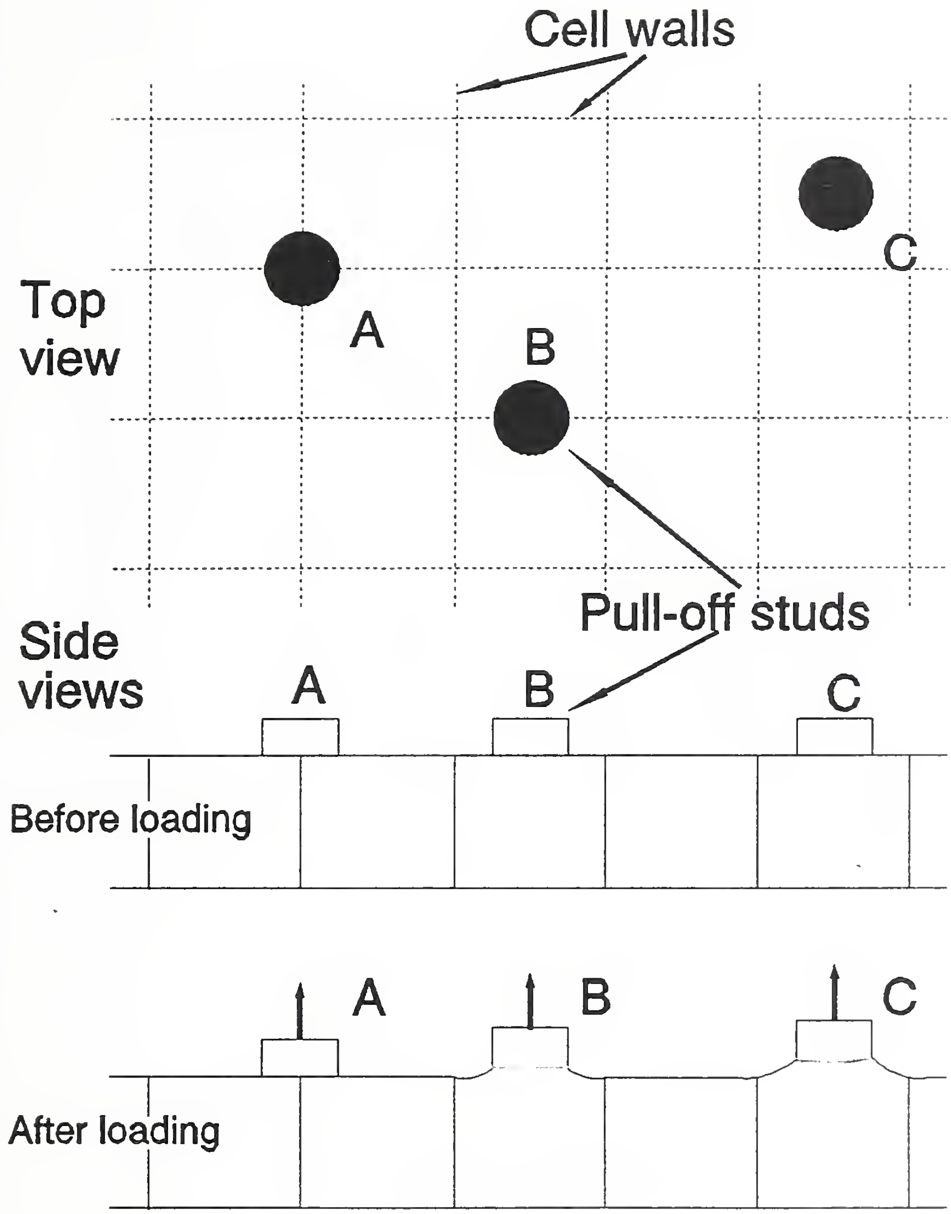

Figure 2. Schematic illustration of possible effect of a honeycomb core on substrate stiffness. Because of the effects of the cells walls, effective stiffness of the substrate may be greater at A than at B, and greater at B than at C.

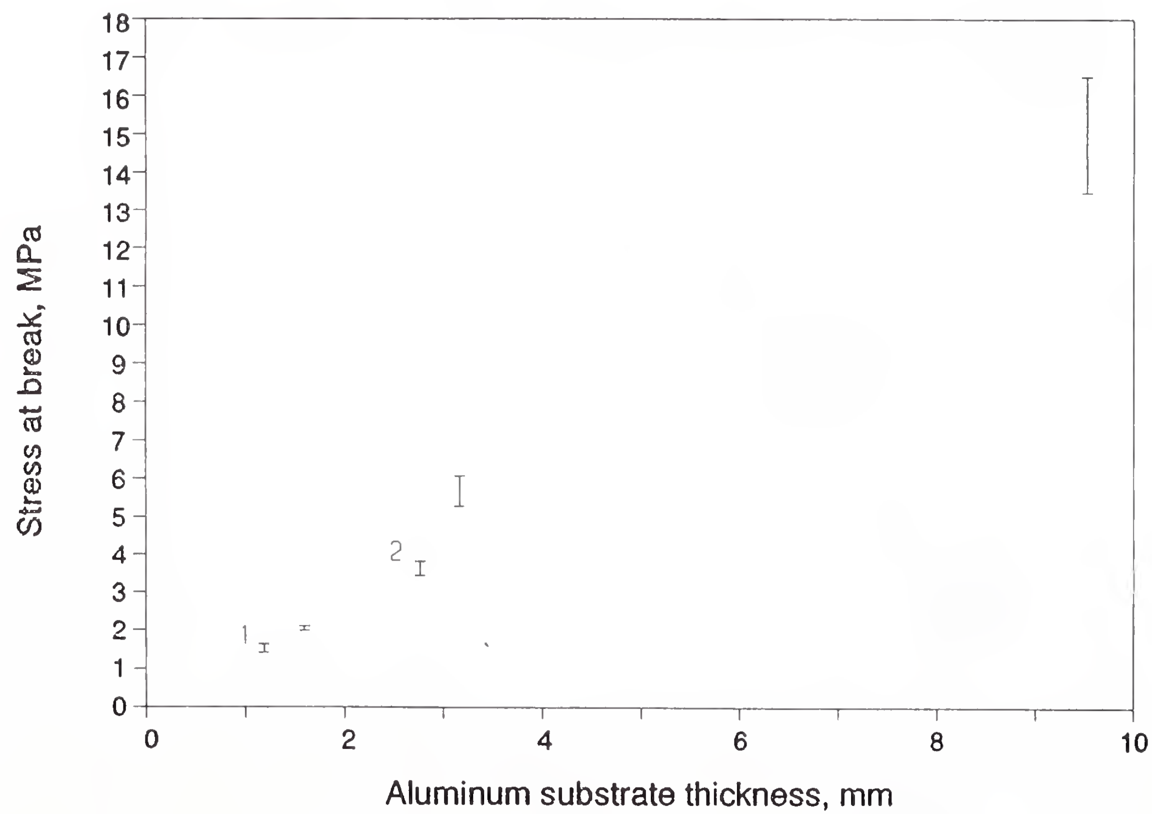

Figure 3. Results of pull-off measurements of an alkyd paint on aluminum as a function of the aluminum sheet thickness. The data set marked "1" is for the pull-off of paint applied to the aluminum skin of a 38 mm (1.5 in) thick foam-core panel; data set marked "2" is for the pull-off of paint applied to the aluminum skin of a 56 mm (2.2 in) thick foam-core panel. The limits are $\pm 2\sigma$ for each set of data.

APPENDIX A Proposed Standard Test Method for Assuring Coating/Substrate Bond-Strength Quality on Shelters

Proposed Standard Test Method for Assuring Coating/Substrate Bond-Strength Quality on Shelters

1. Scope

1.1 This test method covers a procedure and apparatus for evaluating the pull-off strength of coatings on wall or roof panels of tactical shelters. Pull-off strength, commonly referred to as adhesion, is determined by measuring the greatest perpendicular force, in tension, that a surface area can bear before a plug of material is detached or debonded. Adhesion measured in this way is a practical measure of adhesion [1] and depends upon several factors in addition to the coating/substrate-bond strength. These factors include the cohesive strengths of the coating and substrate; the type of pull-off tester; and the substrate and pull-stud stiffnesses (resistance to bending) [2].

1.2 Since the stiffness of the substrate of a wall or roof panel of a tactical shelter may vary from location to location depending upon the type and size of the panels and their construction (e.g., honeycomb, foam and beam), this method provides two ways to control this factor and reduce its unwanted effect on test results: 1) perform tests at the same relative location on each shelter panel, and 2) perform tests on separate test panels, prepared during shelter manufacture to represent the coated shelter surfaces typical of production lots. In the second case, the test panel are prepared from additional pieces of the sheet aluminum used in the shelter construction which are conditioned, pretreated and coated under the same conditions using the same procedures and materials as the shelter panels.

1.3 To establish acceptance criteria, performance levels for pull-off strengths of coating systems/substrate types are predetermined for each system. This is necessary since the acceptable level is likely to be different for different shelter- panel/coating systems, because the test results depend upon the stiffness of the aluminum substrate and the coating system.

2. Referenced Documents

2.1 ASTM Standards

D 2651	Practice for Preparation of Metal Surfaces for Adhesive Bonding[1]
D 3933	Practice for the Preparation of Aluminum Surfaces for Structural Adhesives Bonding[1]
D 4541	Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers[2]

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[1] Annual Book of ASTM Standards, Vol. 15.06, American Society for Testing and Materials, Philadelphia, PA 19103-1187, 1992.

[2] Annual Book of ASTM Standards, Vol. 6.01, American Society for Testing and Materials, Philadelphia, PA 19103-1187, 1992.

E 691 Practice for Conducting a Interlaboratory Test Program to Determine the Precision of Test Methods[3]

3. Summary of Test Method

3.1 This test method uses a self-aligning, pneumatic tester as described in ASTM D 4541. The equipment and supplies needed consist of: 1) an air pressure control module, 2) a pneumatically actuated piston with an air chamber, 3) connecting hoses, 4) pull-studs and cut-off rings or other device to remove excess adhesive, 5) epoxy adhesive, and 6) a source of compressed air which can be regulated to produce a constant pressure up to 0.7 MPa (100 psi). Loads are limited by the strength of the epoxy adhesive/pull-stud/coating interfaces and the maximum load that can be delivered to the pull-stud by the piston. Assuring quality of coating/aluminum bonds is carried out by one of two procedures using pneumatic adhesion testing equipment. In one, the pull-off strength of a coating is determined at specific locations of the shelter panels while, in the other, a test panel is prepared along with the shelter panels and goes through all the steps of cleaning, treating and coating. A piece of 13 mm (0.5 in) thick plywood is glued to the backside of the test panel to increase substrate stiffness. Reference values of adhesion for use in acceptance criteria are determined by conducting pull-off tests on coated surfaces known to be satisfactory. For purposes of quality control, the pull-off strength is compared with the reference values.

4. Significance and Use

4.1 The pull-off strength of a coating is an important performance property that has been used in specifications and quality control procedures. Many methods for its determination have high coefficients of variation. This method provides procedures to obtain a quantitative determination of pull-off strength and, by minimizing the effect of substrate stiffness on test results, make the method suitable for measuring pull-off strengths of coatings on shelters or on specimens representative of shelter walls or roofs. This methods may not be sensitive to small variations in surface preparation.

5. Apparatus A schematic of the apparatus is shown in Figure 1 and described in detail in ASTM D 4541. It is briefly described below.

5.1 *Adhesion Tester*, a commercially available self-aligning pneumatic tester meeting the precision performance obtained for a pneumatic tester reported in ASTM D 4541. A PATTI Jr. has been found to be appropriate for conducting these tests[4].

5.2 *Pull-Studs*, having a flat surface on one end that can be adhered to the coating and a means of attaching to the tester on the other end (e.g., threads). Aluminum pull-studs

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[3] Annual Book of ASTM Standards, Vol. 14.02, American Society for Testing and Materials, Philadelphia, PA 19103-1187, 1992.

[4] Available from SEMicro Corp., 15817 Crabbs Branch Way, Rockville, MD 20855.

with the flat surface having a diameter on the order or 12 - 18 mm (0.5 - 0.7 in) have been found to be suitable for this purpose.

5.3 *Cut-off rings or Other Tool for Removing Excess Adhesive,* plastic rings having the same inner diameter as the outer diameter of the pull-stud or other tool (e.g., spatula, wooden stick) to displace the excess adhesive away from the stud, providing a reproducible pull-off test area.

5.4 *Adhesive,* for securing the pull-stud to the coating[5,6].

5.5 *Sandpaper and solvent,* for preparing the coating to be tested and the pull-stud prior to test. Sandpaper having a grit no coarser than 180 grit and ethanol have been successfully used.

6. Preparation of the Test Surface

6.1 *Test Surfaces - Two Types* Pull-off tests of either type shall be carried out on the complete coating system, i.e., primer, intermediate coat, if any, and topcoat. They shall be carried out on each coating system used in the shelter or as agreed upon between shelter manufacturer and user. The type of test, coating systems to be tested, and procedures shall be described in the quality control documents.

6.1.1 Shelter Wall or Roof Panels No special specimen preparation is needed. Tests will be conducted on a shelter wall or roof panel after it has been coated. Locations for conducting the tests must be specified in the quality-control documents. The locations should be such that at least five pull-off tests can be conducted where substrate stiffness is expected to be the same. For quality control procedures, a minimum of three pull-off tests will be conducted in each location. (Five tests are used in determining acceptance pull-off values.)

6.1.2 Test Panels Two test panels shall be made from an additional sheet of the material used for the skin in the fabrication of the shelter panels. Their dimensions shall be not less than 300 mm (12 in) by 300 mm (12 in). They shall be treated and conditioned in the same manner as the shelter panels. While the skins are being bonded to their cores, the two test panels sheets shall be bonded to the smooth side of interior grade A-D plywood, having a thickness of at least 13 mm (1/2 in) to provide added stiffness. When the shelter panels are coated, the corresponding test panels are to be coated in the same manner as the shelter panels. A minimum of three pull-off tests shall be conducted on each panel. Separate test panels shall be prepared for each coating system used on panels in the shelter to represent these surfaces.

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[5]Structural Adhesive Table Kit, Scotch Weld Adhesive 1838B/A, and Hysol Epoxy Patch Kit 907 have been found satisfactory for this purpose.

[6]Versiloc 201 and 204 with accelerator have been found satisfactory for this purpose.

6.3 *Coating Cure* The coatings shall be cured for the time and under the conditions recommended by the coating manufacturer, unless otherwise agreed upon by shelter manufacturer and user. The cure time and conditions shall be established for each type of coating system and specified in the quality-control documents.

7. Test Procedure

7.1 *Preparing Test Surfaces for Pull-Off Tests.* Mark the perimeters of the pull-off circular target areas of either the shelter or test-panel with a pencil or pen. [Note: A target area can be no closer to an edge of a surface or another test area than the radius of the piston being used.] In the target area, rub the coating surface lightly with 180, or finer, grit sandpaper. Then, lightly wipe the target area with clean cheesecloth dampened with ethanol. [Note: a slight abrasion of the coating surface in the target area improves the bond strength of the adhesive-coating bond. The ethanol removes surface contamination.] The target shall not be touched by bare skin after cleaning. [Note: Oil from the skin may contaminate the coating surface and reduce the bond to the pull-stud.]

7.2 *Adhering the Pull-Studs to the Coating.* Attach pull-studs to each target area following the steps described below.

1) Ensure that the pull-stud has been abrasively blasted and is free of spent adhesive or coating material. Rinse the pull-stud surface with acetone or other appropriate organic solvent to remove any oily contamination. [Note: ASTM D 2651 and D 3933 are typical of well-proven methods for cleaning metal surfaces for adhesive bonding.]

2) Mix the epoxy thoroughly on a clean, non-adsorbing, inert surface, using the proportions of materials and mixing procedure recommended by the adhesive manufacturer. [Note: Departures from the recommended proportions and mixing procedures reduce strength of the bond.]

3) Using a spatula, apply a thin layer of epoxy to the whole of the target area. Also apply a thin layer of epoxy to the face of the pull-stud and work it in to the roughened surface.

4) Allow the epoxy to glaze over (about 10 seconds) and then press the face of the pull-stud straight down on the target area. [Note: Do not allow the pull-stud to skid, as voids or other defects may be introduced in the epoxy-glue layer. These defects may lead to premature failure in the glue layer.] Maintain pressure on the stud for about 1 minute to assure obtaining a reproducible thin adhesive joint.

5) Remove excess adhesive around the pull-stud in one of the two following ways: 1) while holding the pull-stud in place on the target area, place a cut-off ring around the pull-stud (knife edge down) and press it firmly onto the test surface or 2) use a spatula or other tool to remove the excess adhesive from around the stud. [Note: Failure to keep the test area constant leads to less precise results. If a tool is used to remove excess adhesive, ensure that

the stud is not moved less defects are introduced that may degrade adhesive bond quality. Rings will release more easily when coated beforehand with soap or vacuum.]

6) After curing the adhesive for a minimum of 24 hours at 23 ± 5°C or other conditions as agreed upon by shelter manufacturer and user, remove the cut-off ring, if used, as follows. With the thumb of one hand push down hard on the pull-stud. With the free hand, grasp the cut-off ring with a pair of pliers. Slightly squeeze the cut-off ring and gently rotate to remove. [Note: Cut-off rings must be removed with care so as not to disturb the adhesive joint which could lead to adhesive bond failure.]

[Note: Pull-studs may be reused but the adhesive must be thoroughly removed and the abrasive-blasted surface restored. Hardened epoxy that remains on the surface of spent pull-studs can be removed as follows. Place the pull-studs in a container of boiling water and allow to remain for five minutes. With tongs, remove a pull-stud and insert the thin edge of a small spatula between the epoxy and pull-stud surface and peel the epoxy away. This technique requires that the epoxy be removed quickly once it has been exposed to the air. If the pull-stud loses too much heat or moisture, the epoxy will re-adhere. If this happens, put the pull-stud back in boiling water for a minute and repeat the process. Wet, uncured epoxy may be removed from the mixing spatula and other equipment by wiping the coated surface with cheesecloth saturated with ethanol.]

7.3 *Instruction for Using Tester*

Carry out the pull-off tests in accordance with ASTM D 4541 and the tester-manufacturer's directions. Record the indicated force attained at failure or maximum force applied. Observe and record the locus of failure of each test, that is within a coating layer or at an interface. A convenient scheme for describing type and location of failure is detailed in ASTM D 4541. Briefly, label the substrate A, primer B, midcoat C, etc., the adhesive Y, and the pull-stud Z. A cohesive failure (within a coating layer) is indicated by the layer in which it occurs, such as B, C, etc. Interfacial failures are defined by the letters corresponding to the layers, such as A/B.

If the failure is within the epoxy adhesive (Y) or between the epoxy adhesive and either the pull-stud (Y/Z) or the coating (e.g., Y/D), the measured pull-off strength is lower than it would have been if the failure had occurred in the coating system. However, the result is acceptable if the mean of the test results meets or exceeds the acceptable level. But, if the mean result is too low, then an additional test should be carried out since only a lower bound was determined for pull-off strength in the failed test. [Note: Ensure that the pull-stud is clean, the coating surface is slightly roughened and cleaned of debris and the epoxy adhesive is mixed properly to minimize the likelihood of failure between epoxy adhesive and either the coating or pull-stud. ASTM D 2651 and D 3933 provide additional information for preparing metal for adhesive bonding.]

7.4 *Analysis and Interpretation of Results*

Use the instrument calibration factors to convert the indicated force at failure for each test of a coating system into the actual stress applied to the pull-stud in MPa (psi). Record the value for each test and determine and record the mean of the test results. Outlying observations may occur. Procedures for treating outlier results should be agreed upon between shelter manufacturer and user prior to data analysis. [Note: It is advisable also to calculate the standard deviation of the results to ensure that the test procedure is in control, as described in ASTM E 691.]

8. Determining Acceptable Level of Adhesion

Use the appropriate procedure to determine a minimum acceptable level of pull-off strength, unless otherwise agreed upon by shelter manufacturer and user. Ensure that the coating materials meet their material specifications. Also ensure that cleaning and treatment of panels and coating application have been done properly and coating curing requirements have been met.

8.1 *Testing on Shelter Wall or Roof Panels* Perform at least five pull-off tests in each test location on coatings having acceptable performance in accordance with Section 7. The locations should be chosen where substrate stiffness is expected to be uniform. A minimum acceptable level for quality control purposes is 50 percent of the mean of the five measurements providing that the relative standard deviation (RSD) is no greater than 15 percent. Repeat the procedure if the RSD is greater than 15 percent. [Note: An RSD greater than 15 percent in the second set of measurements may indicate a either non-uniform coating parameters (e.g., material, bond strength) or a non-uniformly stiff substrate in the test area. An alternate test location should be considered.]

8.1 *Testing on Separate Specimens* Increase the number of test specimens to three to improve the estimate of the pull-off strength of the coating system. After adhering the aluminum specimens to plywood, determine the pull-off strengths of the coatings as described in Section 7. A minimum acceptable level for quality control purposes is 50 percent of the mean of the 9 measurements provided that the relative standard deviation of these measurements is no greater than 15 percent. Repeat the procedure if the RSD is greater than 15 percent. [Note: An RSD greater than 15 percent in the second set of measurements may indicate a either non-uniform coating parameters (e.g., material, bond strength) or a poor bond between the aluminum skin and the plywood, resulting in a non-uniformly stiff substrate.]

9. Records

9.1 Record the date, shelter lot number, testers name, description of testing equipment, type of panels used, coating type and batch numbers, and pull-off results of all measurement. Note any departures from the specified procedures or conditions. In addition, follow recording procedures defined in the shelter manufacturer's quality systems documents.

10. Keywords adhesion; adhesion tests; aluminum skins; bond strength; coatings; paint; relocatable structures; sandwich panels; tactical rigid wall shelters

11. References

1. K.L. Mittall, "Adhesion Measurements; Recent Progress, unsolved problems and prospects,: in Adhesion Measurements of Thin Films, Thick Films, and Bulk Coatings, STP No. 640, K.L. Mittall, Ed., American Society for Testing and Materials, Philadelphia Pennsylvania, 1978, pp. 5-17.

2. Round robin data from ASTM D 4541 (available form ASTM, 1916 Race Street, Philadelphia, PA 19103) shows that the mean pull-off values for four different devices varied by about a factor of 6, e.g., from 200 to 1200 psi. In addition the intralaboratory and interlaboratory coefficients of variation varied from about 7 to 15 percent, and 6 to 28 percent respectively. A pneumatic self-aligning testing device had the lowest intralaboratory and interlaboratory coefficients of variation of the types of devices included in the round-robin.

a)

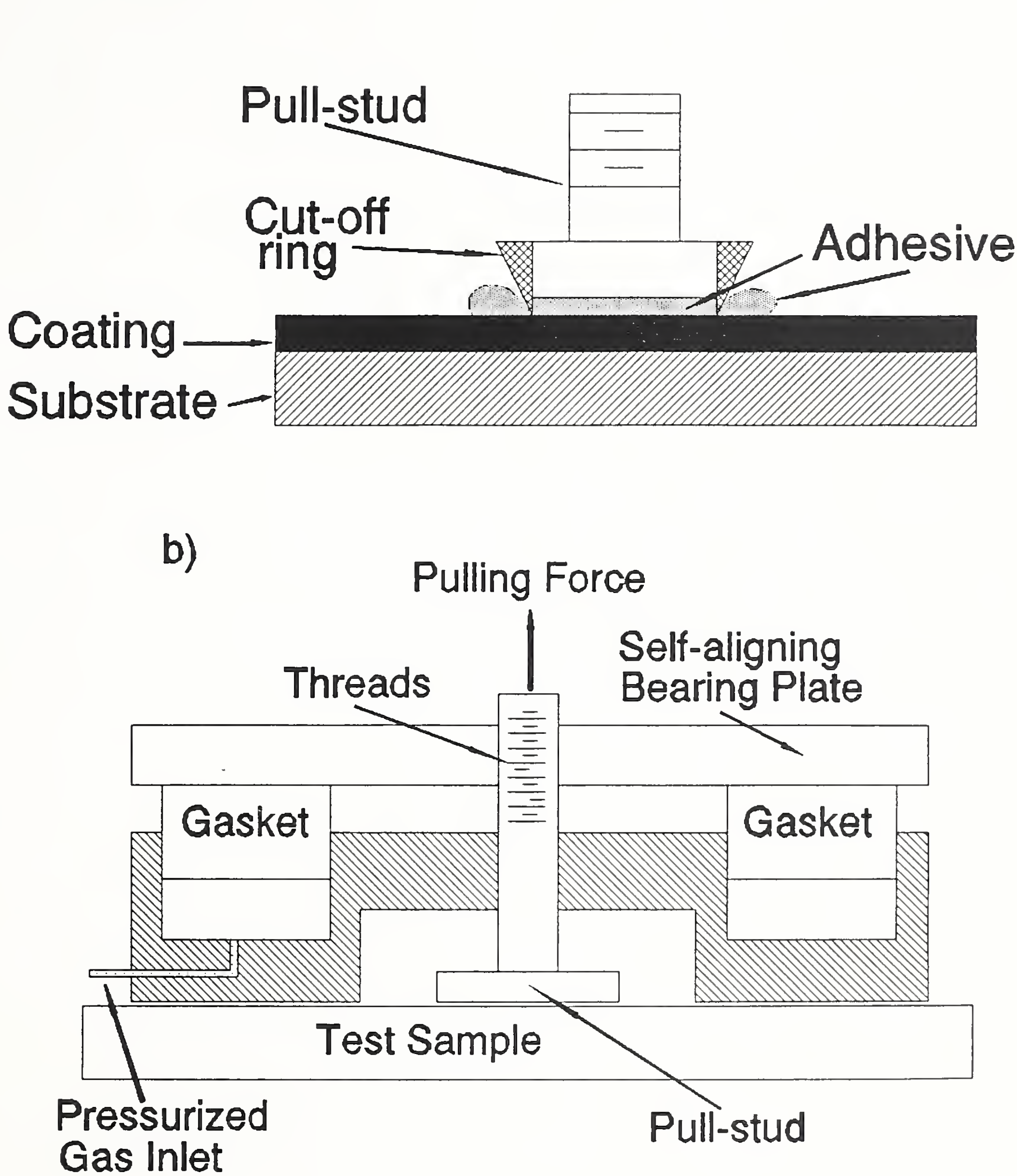

Figure 1. Cross-section schematics of pneumatic adhesion testing device: a) schematic of pull-stud attached to coating and b) schematic of piston attached to pull-stud.