A Test for Coating Adhesion on Flat Substrates—A Technical Note

S.H. Leigh and C.C. Berndt

The standard tensile adhesion test (TAT), ASTM C633, has been modified to perform multiple tests on flat and wide substrates. The TAT geometry, which specifies a 1-in. (25.4-mm)-diameter cylindrical substrate, has been used as the pull-off bar. Two renditions of this test were implemented and the Weibull moduli and characteristic stresses for both test methods obtained. The modified TAT, termed as the single-bar (SB) method, yields a higher Weibull modulus and characteristic strength than the other method, which is termed the double-bar (DB) method. It is believed that the different test results between the two methods arise from different stress distributions near the interface of the coating and substrate.

1. Introduction

THE adhesion strength between the coating and the substrate is one of the most important properties from which the lifetime[1-3] and the quality of a specific application can be estimated. Numerous tests can be used to evaluate the adhesion strength of thermal spray coatings.[4-7] The tensile adhesion test (TAT)[8] is relatively simple to perform and is a widely used method in industry and scientific laboratories. However, there are some shortcomings,[3-5,9-11] and many controversies still exist.[5,7,11,12] The TAT is often used as a quality control tool to determine spraying conditions, surface conditions of substrate, grit blasting conditions, coating thickness, etc.

The TAT has been standardized and there are four main standards: ASTM C633-79 (USA),[8] DIN 50 160-A (Germany),[13] AFNOR NF A91-202-79 (France),[14] and JIS H8666-80 (Japan).[15] The test configuration of ASTM C633 is given by Fig. 1. Other standards have the same basic configuration; however, the substrate dimensions vary from 25.4 to 40 mm in diameter.[16,17] This difference in diameter results in a variation of the stress singularity near the edge of the pull-off bar, i.e., the stress distribution in the vicinity of the pull-off bar is not uniform; therefore, the adhesion value and its variation may change between coatings.

Another distinction of the above standards is that the size, shape, and material of the substrate are restricted (e.g., 25.4-mm-diameter cylindrical shape of metal for ASTM C633 compared to 40 mm for the DIN standard). This limits some applications of the TAT to geometries that are determined by standards rather than by the complex geometries of substrates that are currently being spray-coated. As thermal spray technology covers more fields of application, it is expected that there will be more need to utilize a variety of substrates, such as ceramics and materials of complex shape. Some modifications, therefore, need to be made to enable adhesion-type tests on such substrates.

S.H. Leigh and **C.C. Berndt**, Thermal Spray Laboratory, Department of Materials Science and Engineering, State University of New York at Stony Brook, Stony Brook, NY 11794-2275.

In the present work, TATs were performed to evaluate the adhesion strength of thermal spray coatings on flat substrates, i.e., substrates that need not be of cylindrical geometry or metal. This test configuration is compared with another tensile adhesion test method, which is termed as the double-bar (DB) method, and the test results are compared. A Weibull analysis, which is based on the "weak link theory," is used to analyze the TAT data.

2. Modified TAT Methods

The modified configuration of the TAT, designated as the SB method, has been designed for self-alignment and to accommodate flat and wide thermal spray-coated specimens. The basic configuration of the SB method is similar to the Elcometer adhesion test (Elcometer Instruments Limited, Droylsden, England), which has been used to determine the adhesion of paint. Figure 2 represents the SB method, which can accommodate specimens up to 6 × 6 × 6 in. (152 × 152 × 152 mm). The specimen is positioned between two plates that are 7 × 7 in. (178 × 178 mm) wide. The bottom plate has a 1-in. (25.4-mm)-diameter hole at the center, through which the glued pull-off bar passes and provides tensile stress to the specimen. This bottom plate and the universal joints at the top and bottom of the fixture result in a self-aligning TAT system. This geometry also has an advantage in that several tests can be performed on each flat substrate, since the pull-off bar can be repositioned for each TAT.

The DB method is shown in Fig. 3. This method requires no special testing fixture except two pull-off bars and two universal joints, which are attached to both sides of the flat specimen. Figure 4 shows the simple fixture required to align the two pull-off bars during the epoxy curing process. This configuration also allows multiple tests on flat geometries, but at the expense of maintaining alignment of the two glued attachment bars.

3. Materials and Experimental Procedure

Alumina powder (Metco 101) was sprayed on top of the bond coat (NiCrAlY, Amdry 961) onto 6 × 6 × 0.3 in. (152 × 152 × 7 mm) flat substrates. The substrates were grit blasted before spraying using 24-mesh alumina at an air pressure of approximately 60 psi (0.4 MPa).
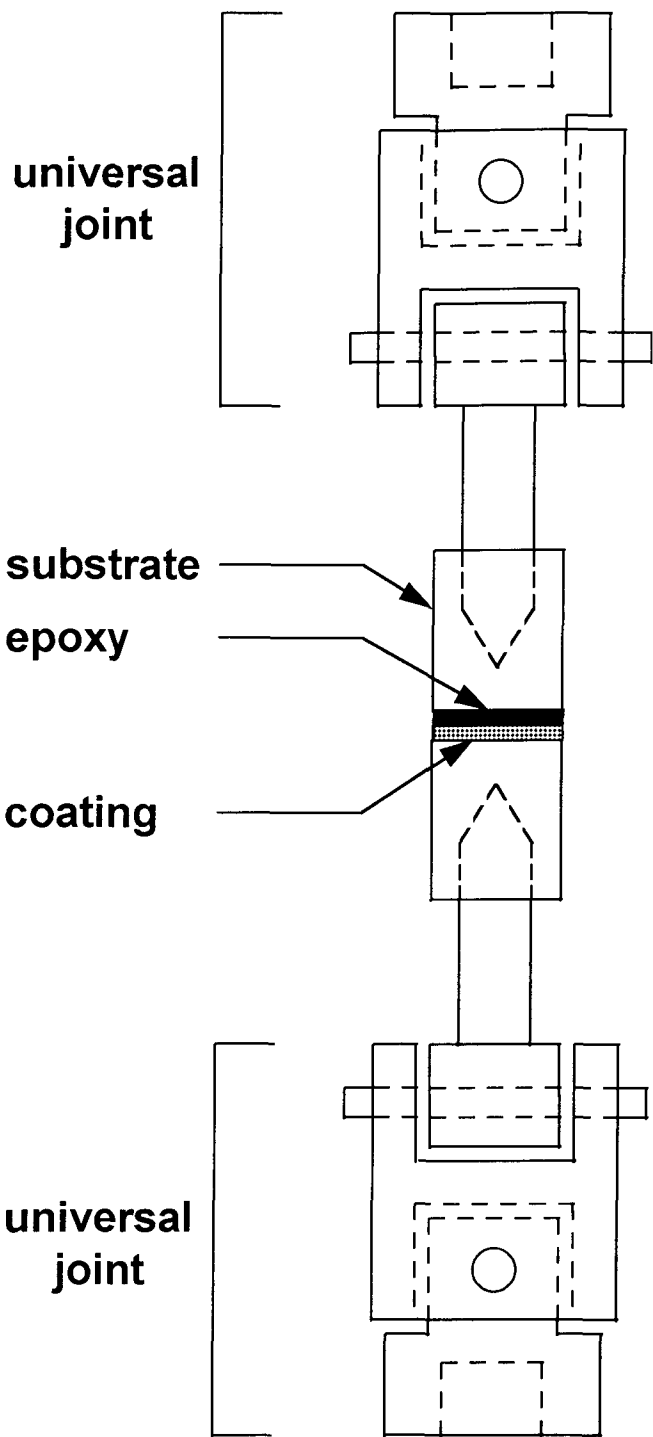
Fig. 1 Configuration of tensile adhesion test specified by ASTM C633.

The plasma spraying process was performed under atmospheric conditions using a Metco 3 MB gun (Perkin Elmer, Metco Division,
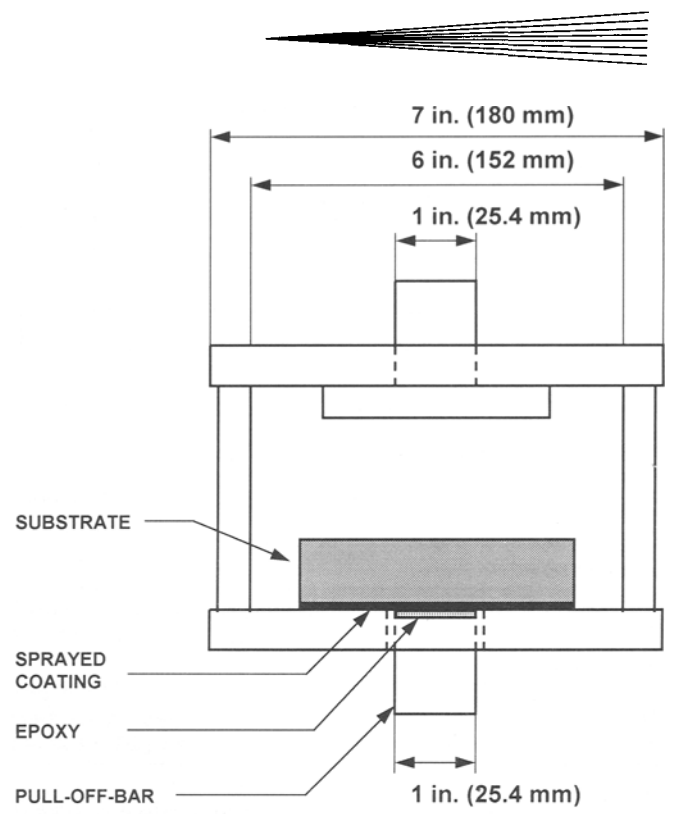

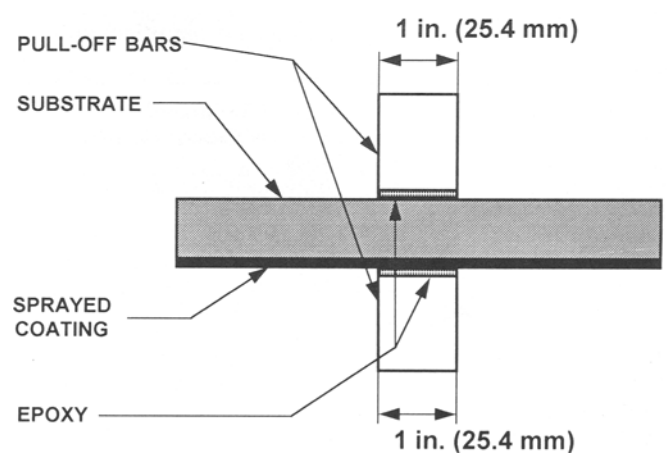**Fig. 2** Schematic of TAT assembly for single-bar (SB) method.

Fig. 3 Schematic of TAT assembly for double-bar (DB) method.

Westbury, NY, USA) (Table 1). The spray condition was not optimized for adhesion strength, since it was not intended that this work focus on the magnitude of adhesion strength but rather on the testing method. The thickness of alumina coating was between 0.006 and 0.008 in. (0.15 and 0.20 mm), and the bond coat was between 0.002 and 0.004 in. (0.05 to 0.10 mm).

Tensile adhesion tests were performed using a hydraulic Instron (Model AW2414-1, Instron Corporation, Canton, Massachusetts). The capacity of the load cell was 200 kN. Calibrations of the load cell were performed automatically by the controller attached to the Instron. The speed of the crosshead was 0.030 in./min (0.013 mm/sec); the test conditions are summarized in Table 2. The fracture stress (average stress) was calculated by dividing the failure load by the testing area [0.7854 in.^{2} (506.7 mm^2)].
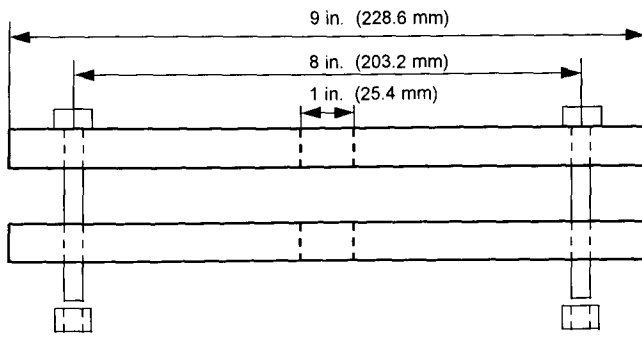
Fig. 4 Fixture for alignment of double-bar (DB) test specimen.

Table 1 Spray conditions for air-plasma spraying of alumina powder

Gun	Metco 3MB
Nozzle	GH
Voltage, volts	60
Current, amperes	500
Argon flow rate, SLPM	85
Hydrogen flow rate, SLPM	15
Powder carrier gas (Ar) flow rate, SLPM	38
Powder feed rate, g/min	50
Spray distance, in. (cm)	3 (8)
Material and average particle size	Al_2O_3 (Metco 101), 53 μm

Table 2 Summary of tensile adhesion test conditions and materials

|	
Test instrument and Model No.	Hydraulic Instron (Instron Corporation,Canton, MA), AW2414-1
Load cell capacity	200 kN
Crosshead speed	0.030 in./min (0.013 mm/sec)
Diameter of pull-off bar	1 in. (25.4 mm)
Length of pull-off bar	1.2 in. (30.5 mm)
Adhesive bonding agent (Epoxy)	FM-1000 film adhesive (American Cyanamid Company)
Adhesive curing temperature	170 °C ± 6 °C (340 °F ± 10 °F)
Adhesive curing time	60 min
Test materials	Alumina coatings on flat substrates [6 × 6 × 0.3 in. (152 × 152 × 7 mm)]
Number of test specimens	i. 20 for single-bar (SB) method

4. Results and Discussion

Figure 5 shows test result histograms of the two TAT methods. The adhesion strengths as measured by the DB method are more widely distributed than those of the SB method, shown in Fig. 6. The arithmetic mean of adhesion strengths measured by the SB method is about 38% higher than the DB method; the SB method yields a higher median and a lower standard deviation.

4.1 Weibull Analysis of TAT Data

The adhesion test result of thermal spray coatings has a large variability, which makes the interpretation of data and interlaboratory comparison difficult. A Weibull analysis is one method of finding a parameter to assess the variability and to index the coating adhesion strength of thermal spray coatings.[18,19]
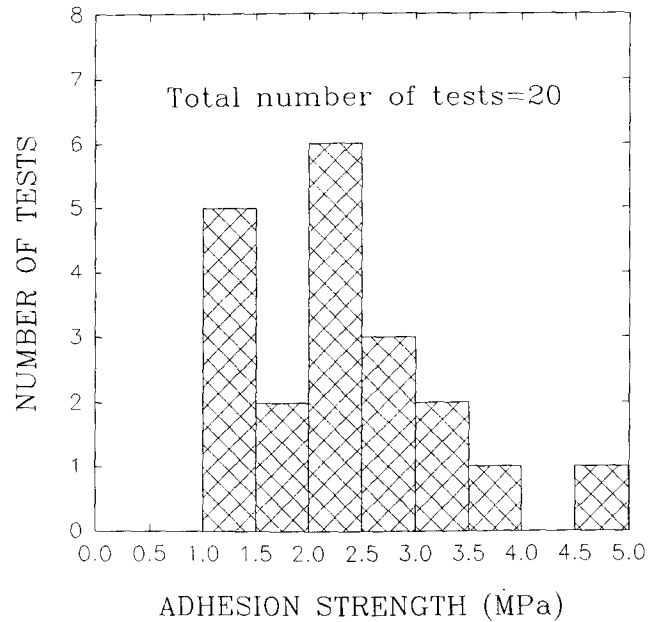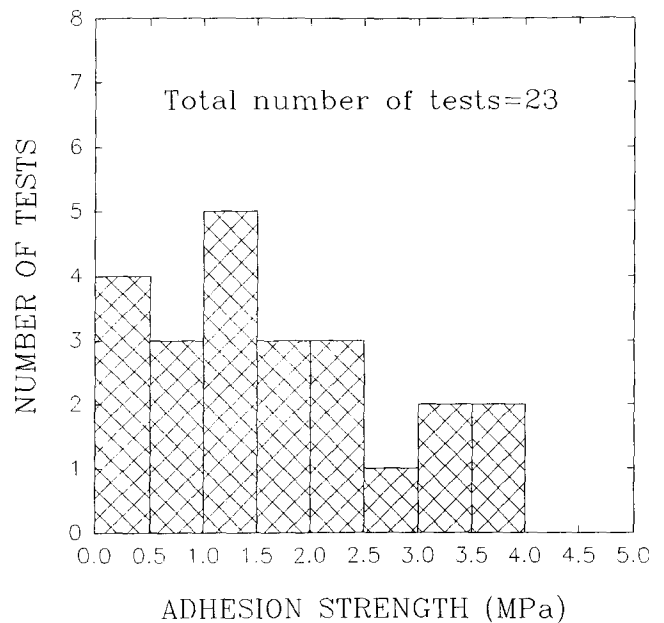
Fig. 5 Adhesion strength vs number of tests for (a) single-bar and (b) double-bar methods.

The Weibull distribution can be expressed in the linear form as:

$$\ln\left[\ln\left(\frac{1}{1-P}\right)\right] = m\ln(\sigma - \sigma_u) - m\ln\sigma_o + \ln V \qquad (1)$$

where P is fracture probability for the stress σ, σ_u is the stress below which fracture is assumed to have zero probability, σ_o is a characteristic stress at which 63.2% of the specimens fail, m is the Weibull modulus, and V is the volume of the specimen.
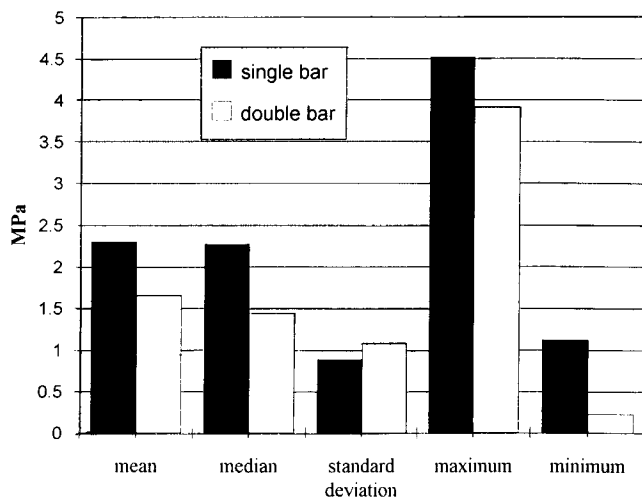
Fig. 6 Summary of tensile adhesion test data.

Fig. 7 Weibull plot for two test methods.

The estimator chosen to determine the value of P for the jth strength[20] was:

$$P_j = \frac{j - 0.5}{n} \qquad (2)$$

where n is the batch size and P_j is the fracture probability (P) for the jth strength.

All of the coating and substrate materials were constant in this study. Therefore, a higher value of m indicates that the test method is a more reliable indicator of failure behavior and the material property of strength.

The Weibull moduli (m) and the characteristic stresses (σ_o) for the two test methods are obtained from the Weibull plot (Fig. 7) and are listed in Table 3. The Weibull modulus is obtained from the slope and the characteristic stress from the intercept on the probability axis of the Weibull plot. The characteristic stress is defined as the stress below which 63.2% of the specimens fail and can be considered a meaningful parameter to index the adhesion strength of coatings. Both the Weibull modulus and the characteristic stress of the SB method are higher than those of the DB test. Berndt[10] calculated the Weibull modulus of plasma-sprayed alumina coatings using the TAT data reported by Hermanek.[21] These values range from 1.4 to 3.8 and can be compared with those obtained in this study, which have quite similar results, i.e., 3.1 and 1.6 for the SB and DB methods, respectively.

4.2 Stress Distribution near Edge of the Pull-off Bar

The TAT is based on the assumption that a uniform stress distribution exists over the entire testing area. The adhesion strength is calculated by dividing failure load by testing area, and the "average stress" at which failure occurs is measured. Thus, stress singularities[22-26] near the pull-off bar that influence the test result are disregarded.

Hopman[27] investigated the stress distribution near the pull-off bar edge of four substrate thicknesses subjected to the SB method using finite element analysis (FEA), as shown in Fig. 8. Although the quantitative stress values and the location of the iso-stress line are most probably different from the present work (since the sample shape and the materials of the test assembly are different),
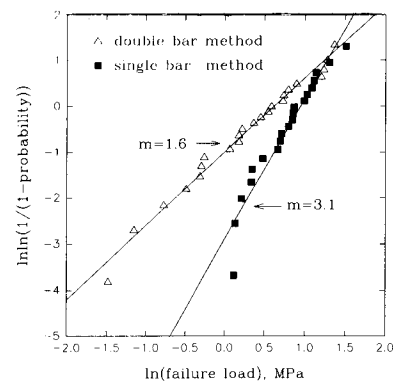
Table 3 Weibull statistics of two adhesion test methods

Test method	Weibull modulus, m	Characteristic strength, σ_o, MPa
Single-bar (SB) method	3.1	2.6
Double-bar (DB) method	1.6	1.9

Hopman's analysis can still be used for a qualitative explanation of the current problem. The applied stress is concentrated near the edge of the pull-off bar, i.e., the singular stress exists in the vicinity of the pull-off bar. As the substrate thickness increases, the stress values in the substrate and the pull-off bar decrease; therefore, the failure stress increases (Table 4). Note in Fig. 8 that a stress field is extended to the bottom side of the substrate. In the case where another bar is attached to this side of the substrate (i.e., the configuration of the DB method), this bar would also produce a stress field that would be expected to influence the overall stress field near the coating/substrate interface. Thus, the DB method yields lower average failure strengths and characteristic strengths than the SB method. Equation 1 is based on the assumption that the tensile stress is uniform over the entire volume of the specimen;[20] therefore, the differences in Weibull moduli probably arise from changes in the stress distribution over the coating/substrate interface.

Figure 8 shows that the stress field at the bottom of the substrate decreases as the thickness of the substrate increases. Thus, it is expected that if the substrate is sufficiently thick [about 1 to 1.5 in. (2.54 to 3.81 mm)], then the test results of both methods would be similar. The tables in Fig. 8 indicate that the maximum stress also decreases as the thickness of the substrate increases; thus, thicker substrates yield higher failure stresses. In addition, longer pull-off bars, which produce more uniform stress distribution near the coating/substrate interface, may reduce the difference between the two test results.[23]

4.3 Comments on the Two Adhesion Test Methods

Two kinds of tests, SB and DB, based on ASTM C633, were performed. Both of the test methods are direct pull-off tests that incorporate various test fixtures. One advantage of these test methods is that coatings on flat substrates can be tested without preparing them in the form of a standardized tensile adhesion test specimen (e.g., 1-in.-diameter bar) and various materials can be used. For example,
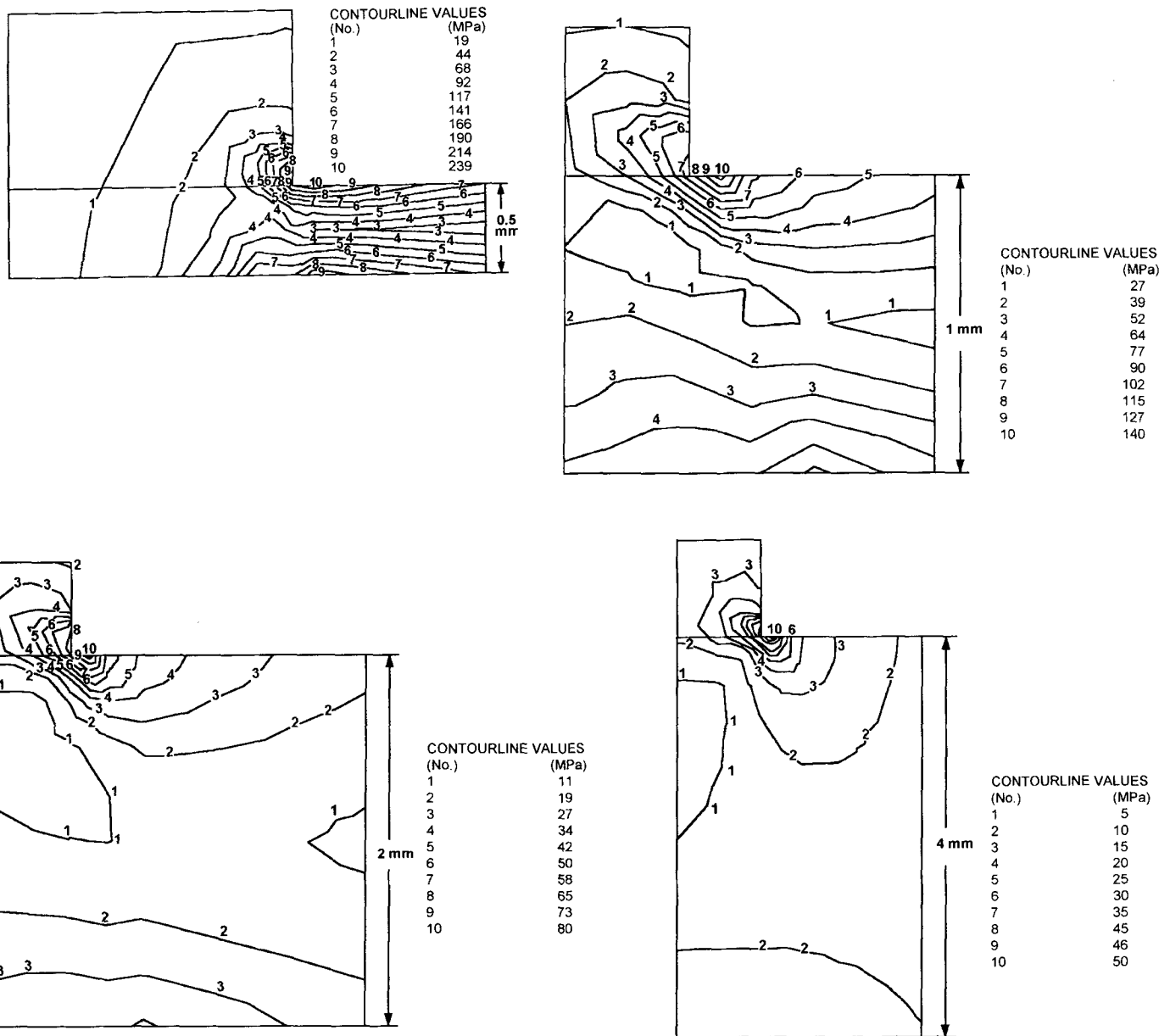
CONTOURLINE VALUES
(No.)   (MPa)
1   19
2   44
3   68
4   92
5   117
6   141
7   166
8   190
9   214
10   239

0.5 mm

CONTOURLINE VALUES
(No.)   (MPa)
1   27
2   39
3   52
4   64
5   77
6   90
7   102
8   115
9   127
10   140

1 mm

CONTOURLINE VALUES
(No.)   (MPa)
1   11
2   19
3   27
4   34
5   42
6   50
7   58
8   65
9   73
10   80

2 mm

CONTOURLINE VALUES
(No.)   (MPa)
1   5
2   10
3   15
4   20
5   25
6   30
7   35
8   45
9   46
10   50

4 mm

Fig. 8 Contour plot of equivalent stresses near the pull-off bar edge for four substrate thicknesses (from Ref 27). All pull-off bar sizes are the same.

ceramics or materials that are difficult to machine can easily be adopted as substrates.

The sample preparation of the SB method is comparatively simple. The DB method has no specific test fixture except the two bars on both sides of the specimen, although a special fixture is required for sample preparation of the DB method to maintain alignment (Fig. 4). In addition, the DB method cannot be used unless both sides of the substrate are flat and reasonably parallel to the coated surface, while the SB method can be used on a specimen that has a flat coated surface.

Every standard adhesion test (ASTM, DIN, AFNOR, JIS, etc.) specifies the specimen size which, in most cases, is less than the size of coatings used in service. The properties of coat-

ings sprayed onto different sized substrates can also be different. For instance, the specimen size may induce different residual stresses that influence the adhesion strength of the coating. Therefore, the larger size of specimens can approach the properties of the product in service. This is one advantage of performing adhesion tests directly on flat and wide substrates.

5. Conclusion

The TAT is relatively simple to perform and is a widely used adhesion test method. Although there are some inherent shortcomings, it can be modified and developed to establish a more

Table 4   Measured stresses and forces at failure of paint coat and calculated maximum stresses (from Ref. 27)

Substrate thickness, mm	Measured		Calculated	
	Mean stress at failure, MPa	Force at failure, N	Maximum stress in substrate, MPa	Maximum stress in dolly, MPa
0.5	2.0	157.0	238	214
1	2.9	216.9	140	102
2	4.2	334.6	80	65
4	7.0	549.8	50	41

suitable testing procedure, since it is still a popular method in industry for quality control and in the laboratory environment for optimization of spray conditions.

A modified TAT, based on ASTM C633 and termed the SB method, was devised to accommodate flat and wide substrates. The test results were compared with another TAT method, termed the DB method. A Weibull analysis was used to analyze and compare the two TAT methods. The Weibull modulus (m) and characteristic stress (σ_o) can be used to characterize the coating integrity and/or coating adhesion. The SB method has a higher characteristic stress and Weibull modulus than the DB method. The difference in the test results seems to be derived from changes in the stress distribution near the interface of the coating and the substrate. Such measurement differences would be reduced by using thicker substrates and/or longer pull-off bars.

Acknowledgments

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