TENSILE ADHESION TEST MEASUREMENTS ON PLASMA-SPRAYED COATINGS

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ABSTRACT

The adhesion strength of plasma-sprayed coatings to a substrate is an often-used but poorly-understood term. The methods of determining the adhesion strength are quite simple and have the advantage of providing qualitative information about the mechanical properties of coatings. However quantitative measurements for the adhesion properties of coatings are not routinely carried out.

This paper examines the tensile method for finding the adhesion property of plasma-sprayed coatings. The inadequacies of this method are clarified and a fracture mechanics approach is proposed. In this manner the variation in tensile strength for any set of samples may be ascribed to pre-existing defects within the coating.

KEYWORDS

Plasma-sprayed coating, tensile adhesion strength, mechanical properties, fracture toughness, alumina.

INTRODUCTION

Plasma-sprayed coatings are a sub-group of "thermal spray" coatings. These coatings have utility for corrosion protection, wear resistance or thermal barrier applications; examples of which are well documented in several "Thermal Spray Conference Proceedings" (German Welding Society, 1983; Nederlands Instituut voor Lastechnick, 1980; American Welding Society, 1976). The coating structure has been well characterized (Gerdeman and Hecht, 1972) as a conglomerate of saucer-shaped particles which result from the high-velocity impact of molten and semi-molten particles onto the substrate surface. Composite coatings, which consist of many different components, and coatings made of different layers, such as a ceramic coating of 0.4mm deposited onto a 0.1mm metallic "bond-coat", are more commonly used rather than single component systems. The material properties of plasma-sprayed coatings which are deposited onto substrates can not be

determined from the individual components of the system.

The adhesion of the coating is of prime importance since this limits the useful life of the coating. The term "strength" is used to describe the material property of the coating which correlates to adhesion and may be expressed as a stress or a fracture toughness quantity. However adhesion, in the context of any coating which may be applied to a substrate, is a ill-defined term. For example a coating may adhere very strongly to a substrate by cracking perpendicular to the substrate interface. These stress relieving cracks may reduce macroscopic delamination (i.e., cracking parallel to the substrate surface) and thus minimize catastrophic failure. This example is clearly different from the case of a coating which is defect free but which still adheres well to the substrate. Analysis of adhesion tests often does not consider the highly cracked structure of the coating as playing an important role in the overall properties of the coating.

EXPERIMENTS

Measurement of Tensile Adhesion Strength

There are three main techniques for the adhesion testing of plasma-sprayed coatings; tensile, shear and fracture mechanics tests. A critical analysis of these methods has been published elsewhere (Berndt and McPherson, 1979). Here it is intended to briefly summarize the tensile adhesion test.

The standard tensile adhesion test (ASTM, 1969) simply involves attaching, with epoxy, a support fixture to the coating so that a tensile force may be applied directly onto the coating, Fig. 1. The ultimate stress at failure is reported as the adhesion strength for that particular coating. It should be pointed out that the stress versus strain properties of the coating, epoxy, and substrate composite are not, at present, used to distinguish the mechanical properties of different coatings.

Failure morphologies such as adhesive, cohesive, or substrate-interfacial modes (Fig. 2) are often not considered of practical importance during adhesion testing; although they certainly have implications with respect to

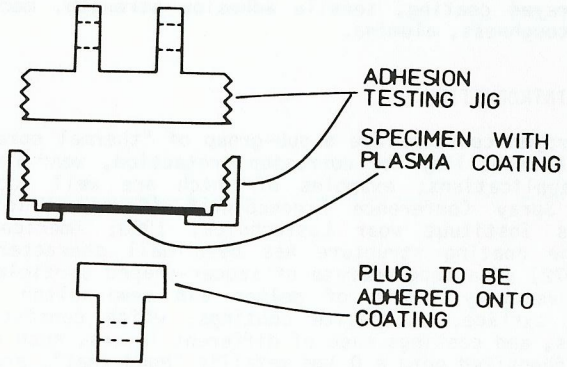

Fig. 1. Concept for tensile adhesion testing of plasma-sprayed coatings.

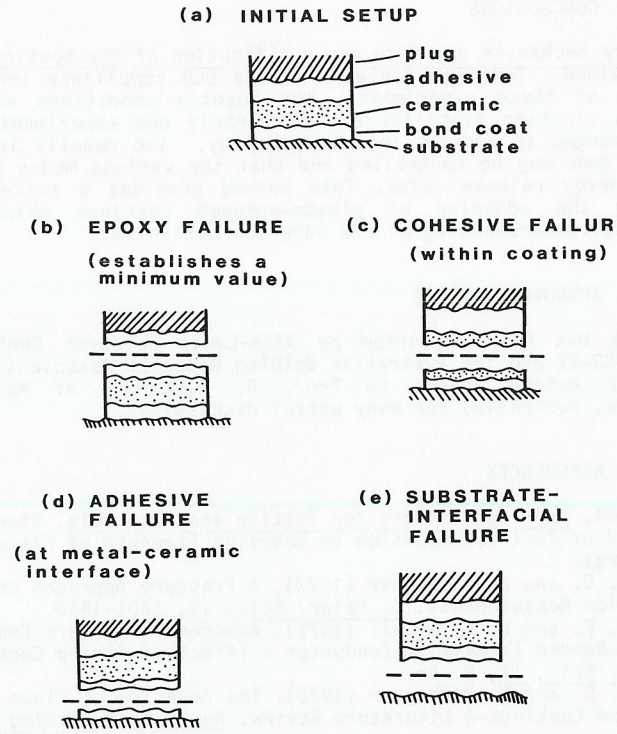

Fig. 2. Modes of failure for plasma-sprayed coatings.

the utility of coatings. For example the major requirement for some applications, such as corrosion protection or wear resistance, is mostly surface protection of the substrate. In these cases partial delamination (i.e., cohesive failure) may be tolerated since a portion of the coating remains attached to the substrate. Even complete coating detachment in localized regions may not be detrimental to the overall performance of the component. On the other hand thermal barrier coatings may be tailored for a specific system use rather than designed to overlay an existing substrate. In these cases even partial coating failure represents total component failure.

This paper concentrates on the fracture mechanics interpretation of coating adhesion. A method of using the tensile adhesion test to estimate fracture toughness properties of coatings is presented.

Literature Data

Much data of tensile adhesion tests has been generated during the routine testing of production coatings. One general observation during the duplicate testing of any coating is that the individual results are usually

distributed over a wide range of strength values. There have been few works which have documented a large number of tests; since only an average value of strength has, in the past, been found necessary to classify any coating system.

Work carried out during a round-robin series of tests by three coating manufacturers (identified as A, B and C) is summarized in Table 1 (Hermanek, 1978). The sample size for each group of coatings from manufacturers A and C is 15; whereas manufacturer B tested 12 bond specimens. This data shows that preparation of the bond-test specimen has a significant effect on the bond strength value and that within each data-group there is considerable variance.

TABLE 1. Tensile Adhesion Strengths of Alumina Coatings(1)

Preparation and Manufacturer(2)		Tensile Adhesion Strength (MPa) Low Value	High Value	Average Value	Standard Deviation	Coefficient of Variance %
1.	A	11.0	35.7	27.7	7.3	26
	B	11.7	42.1	29.3	8.9	31
	C	19.7	47.9	32.1	10.1	31
2.	A	11.2	42.3	25.9	9.2	35
	B	11.0	30.4	22.8	7.3	32
	C	14.4	46.6	24.7	8.2	33
3.	A	7.9	20.3	14.3	3.5	25
	B	6.2	24.8	15.2	6.3	41
	C	24.7	44.4	33.7	6.2	18
4.	A	9.3	22.7	12.9	4.8	37
	B	2.8	23.5	10.2	5.8	57
	C	3.1	9.2	6.1	1.9	30

(1) Original data from Hermanek (1978). Refer to this article for trade names of materials.
(2) Preparations are: 1. epoxy a. 2. epoxy a + sealer. 3. epoxy b. 4. epoxy b + sealer.

Analysis

It is difficult to precisely ascertain the effects of different epoxies and use of sealing agents on the properties of the composite adhesion test specimens. It would be tempting to treat such data as belonging to a normal distribution; or more practically, to conveniently describe the materials properties of coatings in terms of the Weibull distribution (Grissafe, 1965).

If the data is treated as a normal distribution it is easy to calculate the maximum strength of the coatings which is, for the present, arbitrarily taken as the 99th percentile of the normal distribution. These values are presented in Table 2 and show the strength values that, statistically, 1 out

TABLE 2. Fracture Toughness Values Calculated From Tensile Tests

Data Set		Average K_{1C} $MNm^{-3/2}$	Maximum K_{1C} $MNm^{-3/2}$	D/d (1)	Theoretical Maximum Stress MPa	Theoretical Max. K_{1C} $MNm^{-3/2}$	D/d (2)	% Diff. (3)
1.	A	1.56	2.01	0.93	44.7	2.52	0.86	25
	B	1.65	2.37	0.90	50.1	2.82	0.84	19
	C	1.81	2.70	0.89	55.5	3.12	0.84	16
2.	A	1.46	2.38	0.86	47.2	2.66	0.82	12
	B	1.28	1.71	0.92	39.8(4)	2.24	0.84	31
	C	1.39	2.62	0.81	43.9(4)	2.47	0.83	-6
3.	A	0.81	1.14	0.90	22.4	1.26	0.87	10
	B	0.86	1.40	0.86	29.8	1.68	0.80	20
	C	1.90	2.50	0.92	48.2	2.71	0.90	9
4.	A	0.73	1.28	0.83	24.1	1.36	0.82	6
	B	0.57	1.32	0.75	23.7	1.33	0.74	1
	C	0.34	0.52	0.88	10.5	0.59	0.84	14

(1) Calculated from the experimental max. K_{1C} and the average stress value.
(2) Calculated from the 99th percentile of the average stress value (considered as a normal distribution) and the experimental stress value.
(3) Difference between the calculated maximum stress and the experimentally determined high stress; expressed as a percentage of the experimental value.
(4) Note that the calculated maximum stress is less than the experimentally determined stress.

of 100 specimens would exhibit. The main point is that these predicted maximum tensile strengths, based on normal distributions, are not untypical of the maximum tensile strengths of plasma-sprayed alumina coatings. The difference between the calculated and experimental maximum strengths varies from -6 to +31 percent. However these results are not adequate for a thorough statistical analysis. Even if such data may be statistically described there is still no information in regard to the exact nature of the materials properties which give rise to the scatter.

It is reasonable to assume that defects, such as porosity, cracks and lamellar boundaries, may give rise to variation in bond strengths. Thus defects reduce the effective load bearing cross-sectional area and, together with stress concentration, reduce the apparent tensile strength. For the case of defect-free materials K_{1C} can be established from the expression (Pabst and Elssner, 1980)

$$K_{1C} = P \times (-1.27 + 1.72 \times (D/d)) \times D^{-3/2} \quad (1)$$

where K_{1C} = fracture toughness ($N\ m^{-3/2}$)
P = fracture force (N)
D = outside diameter (m) = 2.54×10^{-2}
d = inside diameter of a circumferentially notched specimen (m)

If the samples were not notched then (d/D) = 1 and equation 1 reduces to

$$K_{1C} = 0.353 \times D^{1/2} \times \text{stress} \quad (2)$$

therefore $$K_{1C} = 5.63 \times 10^{-2} \times \text{stress} \quad (1a)$$

The average toughness of alumina coatings may be estimated from equation 1a. Any particular coating may be less strong than another identical coating because macroscopic defects (in general terms) reduce the fracture toughness. Therefore, if the left hand side of equation 1 is set to the maximum fracture toughness value (either the calculated or experimental value) and the load (or stress) on the right hand side varied, then the equivalent defect size may be estimated in terms of a reduction in diameter. When using the calculated values this is equivalent to describing the high strength values (i.e., 1% of the distribution) as either "flawless" or optimallly prepared to minimize defects. The ratio (d/D) can be found (Table 2) and this is a measure of coating integrity.

DISCUSSION

The tensile adhesion test is routinely used in industry and research to measure the "adhesion" quality of plasma-sprayed coatings. A major advantage of this method is that qualitative tests can be easily carried out. However it is worthwhile to examine some of the criticisms against this method. For example the tensile force imposed on the coating may not represent the forces which are observed during the service life of the coating. Thus if service failure results from compressive loading in the plane of the substrate then mechanical property measurements perpendicular to the substrate will not be relevant. Therefore it is important to account for the locus of failure, which relates to the failure mechanism, during the determination of mechanical properties.

A major limitation of the tensile adhesion test has been explaining the wide scatter of results which are observed during tests. The variation in strength may arise from real material changes, such as are observed from different processing procedures (e.g., different substrate surface preparation or plasma-spray deposition parameters) or result from variations associated with the specimen testing methods. It is evident that the small sample size of 12 or 15 specimens does not describe the entire population, however the normal distribution has been used for comparitative purposes. It is assumed that the scatter within any set of data arises from defects within the coating. Fracture mechanics concepts have been used to estimate the proportion of these defects relative to the apparent cross-sectional area.

Caution must be exercised when considering such analyses because the distribution of a small sample is being approximated to normality. It is also assumed that all errors arose from the processing conditions and is not dependent on the testing procedure. However both analyses result in about the same cross-sectional diameter (d) of 18.8-23.6mm. Thus the effective load bearing area of a plasma-sprayed coating is about 55-86% of the apparent area regardless of the specimen preparation technique. Exactly the same calculations may be based on a maximum strength of 99.9% of the assumed normal distribution. Under these conditions the load bearing diameter of the specimen varies from 17.3-22.1mm; which corresponds to about 46-76% of the apparent cross sectional area.

It would be expected that each preparation technique, of presumably identical coatings from each manufacturer, would have the same effective load bearing area and this fact is brought out by the above analysis. It should again be emphasized that not too much physical significance can be placed on the exact (d/D) ratio because of statistical approximations which have already been discussed. The main point is that plasma-sprayed coatings exhibit a low strength of adhesion due to defects. The exact nature of these defects may be ascertained by metallographic examination. The magnitude of (d/D), for a statistically significant sample size, may reflect the homogeneity and integrity of coatings regardless of the test methods used.

CONCLUSIONS

The primary purpose of this work has been to present a limited study on adhesion measurements of plasma-sprayed coatings. The main emphasise has been to critically analyse the experimental scatter for duplicate tests.

A simple method, in application, has been detailed which presents tensile adhesion strength data in a fracture mechanics perspective. An analysis has been performed, on the data available, which suggests an approach to find the overall defect contribution in reducing the apparent strength of coatings. These results need verification with more extensive testing and consideration of other factors such as the locus of failure.

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