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ANALYSIS OF THE SHEAR-BOND STRENGTH OF ALUMINA COATINGS[*]

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Alumina coatings were plasma-sprayed onto AISI 304 stainless steel, and the resultant coating-substrate bond strengths were measured by a shear test. The effects of alumina powder particle size and purity, and substrate surface roughness on bond strength were determined. The bond strength data were analyzed using Weibull statistical methods.

The bond strengths on polished surfaces were zero. Increased substrate surface roughness resulted in increased bond strengths. Increased alumina particle size uniformity and higher values of median particle size contributed to increased bond strengths and to more reproducible data, as indicated by higher Weibull slopes. Under fixed conditions of substrate roughness and alumina powder characteristics, the measured values of bond strength were found to decrease as the size of the coated substrate test area was made larger. This decrease was approximated by curves based on probability theory calculations.

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[*]The material presented herein was included in the thesis submitted by the first author to Case Institute of Technology in May 1964 in partial fulfillment of the requirements for the degree Master of Science.

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I. Introduction

The stress at which the bond between a ceramic oxide coating and a metallic substrate fails and the factors which affect this failure stress have important significance in the design of coated aerospace hardware. Previous attempts to determine the bond shear strengths of such coatings have involved techniques (ref. 1 and 2) which have not been entirely satisfactory, because of nonuniformity of loading. Therefore, a testing technique was developed (ref. 3) to improve loading uniformity. This technique was used in the present study to investigate the bond strength of plasma-sprayed alumina coatings on stainless steel. The effects of spray-powder particle-size distributions, powder purity, and substrate surface roughness on the measured bond strengths were determined. The resultant bond-strength data were analyzed by Weibull statistical methods. The influence of the size of the coated substrate test area on the measured values of bond strength was also investigated.

II. Materials

Four commercial alpha alumina spray powders, designated A, B, C, and D, were employed in this investigation. The chemical analysis, particle size range, and median particle size for each powder are presented in Table I. Figure 1 represents the measured particle size distribution for each powder and shows that powders B and C contain more particles below 10 microns than do A and D. Powder C also contains some very large particles. Powders A and D, on the other hand, have lower percentages of particles outside the 10- to 35-micron range. The substrate material, in all cases, was AISI 304 stainless steel.

III. Apparatus and Procedure

The bond shear test apparatus, shown schematically in figure 2, consists of a 1/4-inch stainless-steel-substrate plate to which is bolted a pair of steel rails. The rails act as guides for a movable slider (also 1/4-inch stainless steel). All mating clearances between the slider and the rails are approximately 0.002 inch.

In this test, the large hole in the slider exposes a fixed area of the plate beneath it, which serves as the test area. The entire portion of the substrate plate between the guide rails is polished to a measured root-mean-square surface roughness of less than 3 microinches. Then the test areas are, in most cases, sand- or grit-blasted (with the slider acting as a mask) to measured root-mean-square roughnesses of approximately 115, 225, and 280 microinches.

Following the roughening operation, all parts are cleaned with acetone and reassembled. An alumina coating, approximately 0.030 inch thick, is plasma-sprayed through the hole in the slider onto the substrate at a torch-to-substrate distance of 6 inches. During spraying, the assembly is wrapped in brass shim stock so that only the area to be tested is exposed to the plasma spray. The opposite face of the substrate is, in all cases, air-cooled during spraying (see ref. 4 for torch conditions).

The coated assembly is allowed to cool to room temperature, the shim stock is removed, and the assembly is vacuumed to remove stray alumina particles. The assembly is then mounted horizontally on a test stand, a cable and weight holder are attached to the slider, and the coating-substrate interface is deadweight-loaded in 1-pound increments until shear failure occurs.

The loading uniformity of this test can be inferred because after failure, the coating remains intact as a disk in the slider hole; that is, failure is restricted to the interface region. Further verification of uniformity is provided by photoelastic stress studies of a similar disk shear test (developed to evaluate structural adhesives) presented in reference 5.

In order to evaluate the effects of surface roughness and spray-powder characteristics on the bond shear strength, at least 10 trials per spray-powder - roughness combination were made on 5/8-inch-diameter test areas. The strengths were calculated on the basis of projected geometrical area, $\pi\left(\frac{5}{16}\right)^2$. The resultant data were analyzed by Weibull statistical means. Test area effects (different slider hole sizes) were calculated on the basis of probability theory, and single-bond-strength measurements were made to assess experimentally the correlation between measured and theoretical values.

IV. Results and Discussion

Few observable differences existed among the microstructures of coatings of all four powders. All coatings showed good penetration into the depressions of the roughened substrate, but none showed any evidence of coating-substrate interaction. After testing, examination of the coating at the original substrate interface showed some torn areas. Also, evidence of some metallic inclusions, which might be attributed to sheared off substrate peaks, was observed. Examination of the substrate showed coating material randomly retained at the bottom of surface depressions; there was approximately 10 percent for coatings of powders A, B, and C, and approximately 30 percent for coatings of powder D.

Upon X-ray diffraction examination, the coating material at the original interface with the substrate was found to be 95 to 100 percent gamma alumina for powders A, B, and D, while 50 percent gamma and 50 percent alpha alumina were present for coatings of powder C. The latter, having some larger particles, would be expected to cool more slowly upon impact with the substrate, and thus would be less likely to transform to the gamma phase.

The bond-shear-test data, determined for coatings of the four spray powders on 5/8-inch-diameter test areas of varying roughnesses, were examined by Weibull analysis (refs. 6 to 8). The approach employed plotting the median rank failure probability against the bond strength on Weibull coordinate paper (ref. 4). The data from this analysis are summarized in table II. The median bond strengths (i.e., the strength below which 50 statistical percent of the distribution failed) are also presented as are the slopes of the Weibull plots m. The value of m is an index of material homogeneity and failure reproducibility; higher values of m indicate more predictable failure behavior.

The effect of surface roughness on the median bond strengths of the various coatings is presented in figure 3. This figure shows that on the polished surfaces the bond strength is zero. As surface roughness increases, the bond strengths increase but the effect becomes less pronounced at higher levels. Both factors indicate that the bond is predominantly mechanical. Figure 3 further shows that different starting spray powders produce coatings having different median bond strengths. For example, at 280 microinches, the median bond strengths rank D, A, C, and B. In light of the particle size distributions for these powders (fig. 1) it appears that uniformly sized powders with low percentages of particles above 35 microns or below 10 microns produce coatings with higher bond strengths under the deposition conditions employed.

From tables I and II, no apparent correlation can be made between powder chemistry or total powder impurity content and median bond strength.

The degree of interfacial failure varied (as previously stated) with coatings of different powders. Coatings produced from powders A, B, and C failed more at the coating-substrate interface than did those of powder D. The relatively large amount of coating material retained at the sheared interface indicates that the bond strengths of the latter coating were close to the shear strength of the as-deposited coating material.

Figure 4 shows the experimental bond strengths of coatings produced from powder B, which were deposited on various sizes of test areas preroughened to 115 and 280 microinches. Figure 4 also includes curves representing the calculated relationship between test area and bond strength, based on probability theory. The curves were plotted using points obtained by a technique similar to that presented in reference 9, and the actual employment of that technique to this problem is fully described in reference 4.

The curves in figure 4 show that the calculated mean bond strengths are high for small test areas but decrease with increasing test area. The measured values of bond strength, representing only one experimental determination, approximate the calculated curves. This influence of test area gives insight into the wide differences in bond-strength levels found in the literature (e.g., ref. 10) and is similar to the size effect found for other ceramic materials (ref. 8):

V. Concluding Remarks

1. A high purity spray powder having the narrowest particle size range (3.4 to 37.9 μ), a moderately large median particle size (15.9 μ), and a uniform size distribution produced coatings having the highest median bond strengths on all intentionally roughened surfaces. Coatings of this material failed less at the interface than did those of the other materials, which indicates that coating shear strength as well as bond strength is an important factor for future tests.

2. The powders with higher percentages of particles between 10 and 35 microns (low percentages of coarser and/or finer particles) and higher median particle sizes produced coatings with higher bond strengths, which indicates that particle size uniformity contributed to better bonding in the range investigated. Furthermore, the Weibull slopes for coatings of these materials were higher, which indicates more reproducible data.

3. The bond strengths of all coatings deposited on polished substrates were zero, and increasing surface roughness resulted in increased bond strengths, which indicates a predominantly mechanical bond.

4. The measured bond strengths decreased with increasing test area, and this decrease was approximated by curves based on probability theory calculations. This indicates that the test area selected for future coating studies must be carefully considered, and strength values must be appraised

in light of the test area employed.

5. Within the purity range represented by the alumina powders used, powder purity appears to have no effect on bond strength.

TABLE I. - CHARACTERISTICS OF AS-RECEIVED ALUMINA SPRAY POWDERS

Powder	Supplier's Chemical Analysis		Size range, μ	Median particle size, μ
	Concentration, weight percent	Compound		
A	97.5 2.5	Aluminum oxide Titanium oxide	3.9 to 46.0	18.8
B	98.0 2.0	Aluminum oxide Silicon, ferrous and sodium oxides	3.7 to 38.9	8.6
C	99.49 .05 .1 .1 .35	Aluminum oxide Silicon oxide Ferrous oxide Titanium oxide Sodium oxide	7.3 to 82.0	11.4
D	99.49 .05 .1 .1 .35	Aluminum oxide Silicon oxide Ferrous oxide Titanium oxide Sodium oxide	3.4 to 37.9	15.9

TABLE II. - BOND STRENGTH DATA AND ANALYSIS SUMMARY FOR COATINGS OF ALUMINA POWDERS SPRAYED ONTO STAINLESS STEEL SUBSTRATES OF VARYING ROUGHNESS

Powder	Root-mean-square surface roughness, μin.	Median bond strength, lb/sq in.	Weibull slope, m
A	280	545	10
B	<3	~0	
	115	270	[a]4.0
	225	390	5.4
	280	440	[a]3.8
C	280	510	5.8
D	<3	~0	
	115	380	11
	225	540	7.4
	280	600	7.7

[a]Least mean square.

REFERENCES

1. Ingham, H. S.; and Shepard, A. P.: Metallizing Handbook. Vol. 1, Seventh Ed., Metallizing Engineering Co., Ch. A, 1959.

2. Moore, D. G.: Basic Studies of Particle-Impact Processes for Applying Ceramic and Cermet Coatings. Nat. Bur. of Standards Rept. 6356, NBS, Jan. 1959 to Mar. 1959.

3. Grisaffe, S. J.: Quantitative Analysis of the Bond Strengths of Plasma Sprayed Coatings. M. S. Thesis, Case Inst. Tech., 1965.

4. Grisaffe, S. J.: Analysis of THE Shear Bond Strength of ~~Plasma Sprayed~~ Alumina Coatings ~~on Stainless Steel~~. Proposed NASA Technical Note.

5. Twiss, S. B.: Structural Adhesives - Their Promising Future in Materials Joining. Research/Development, Vol. 15, no. 8, Aug. 1964, pp. 26-30.

6. Barnett, R. L.: Review of Structural Design Techniques for Brittle Components under Static Loads. ARF Rept. 8259, May 1963.

7. Salmassy, O. K. et al.: Behavior of Brittle State Materials. WADC TR-53-50, Part II, June 1955.

8. Weil, N. A.: Studies of the Brittle Behavior of Ceramic Materials. ASD TR-61-628, Apr. 1962.

9. Zaretsky, E. V., et al.: The Effect of Contact Angle on Rolling-Contact Fatigue and Bearing Load Capacity. ASLE Trans., Vol. 5, pp. 210-219, May 1962.

10. Shell, J. S.; and Neilsen, J. P.: Study of the Bond Between Gold Alloys and Porcelain. Jour. Dental Res., Vol. 41, No. 6, 1962, pp. 1424-1437.

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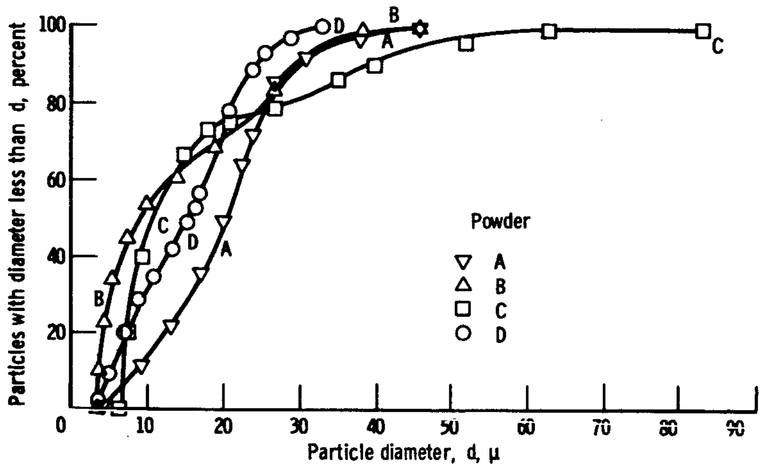

Figure 1. - Particle-size distribution of 500 particles of four alumina spray powders.

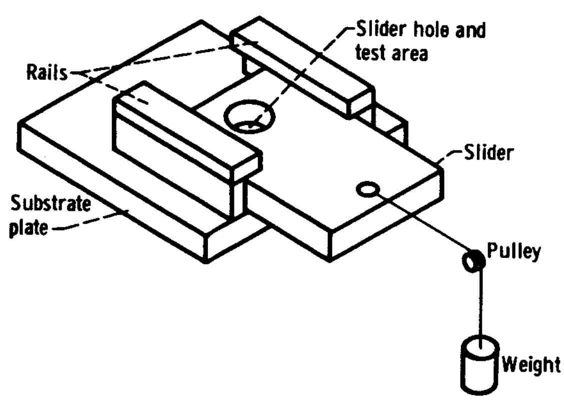

Figure 2. - Apparatus for bond shear-strength test.