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Modified Ball Bond Shear Test for Determination of Adhesion Strength of Cold Spray Splats

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Abstract

A modified ball bond shear test was developed to measure the adhesion of cold sprayed splats to both bulk substrates and cold sprayed coatings. The technique was applied to the deposition of Ti feedstock powder (spherical morphology, 29 μm average dia.) onto a commercially pure bulk Ti plate and onto Ti coatings prepared with the same feedstock powder as the splats. Both an adhesion strength and adhesion energy were measured, with trends in adhesion examined as a function of in-air particle velocities between 580 and 825 m/s. Comparisons between cold spray splat on bulk materials versus coatings were conducted for two deposition conditions (694 and 825 m/s). Generally, the adhesion for splats deposited at the higher deposition condition were insensitive to the substrate material, while splats deposited at the lower deposition condition adhered better to the bulk material than to the coatings.

1. Introduction

Cold spray is an emerging coating technology [1] that makes use of a converging/diverging nozzle and a high pressure, heated gas source (usually nitrogen) to create a high velocity gas flow. Metallic particles, usually in the size range of 10 – 100 microns, are injected into this gas flow and propelled to supersonic velocities. Coating growth occurs at relatively low temperatures compared to other spray technologies. When processing conditions are optimized, the technique can produce near fully dense coatings. Despite the promise this technique has shown, questions remain about the quality of the material deposited in terms of adhesion strength of coating to substrate, and cohesion strength between cold sprayed particles that make up the coating.

Typically, the adhesion or bond strength of cold spray coatings is determined using ASTM C-633-99 [2]. Other methods are similar to the standard [3] and involve the deposition of coatings onto the ends of polished cylindrical grips. One face of a cylinder is coated and then glued to a counterblock with an epoxy resin. A tensile test is conducted and, if failure takes place at or near the coating/substrate interface, an adhesion strength is calculated from the peak force and the coated area. One drawback of this technique is that substantial quantity of material is needed to coat the cylinders. Another pitfall is that if the coating/substrate adhesion strength is high, the failure often takes place in the epoxy or at the epoxy/coating interface. In this case, the test only provides a lower limit for the adhesion strength. Determination of cohesion strength, or bonding strength between sprayed particles, is even more problematic to determine. Sometimes, a qualitative approach is taken, particle/particle interfaces are examined by light optical microscopy or scanning electron microscopy [4]. One may also use the depth obtained in a scratch tests as an indirect measure of cohesion [5]. However, when quantitative data is sought,

coatings must be deposited onto specialized test geometries [6] that require careful machining and a substantial quantity of sprayed material.

This study is on an alternative test method for determination of the adhesion strength of cold sprayed material to substrates and previously sprayed coatings. The second of these might be reflective of cohesion strength. The test method is based on the ball bond shear test commonly used to test the adhesion or shear strength of solder balls in microelectronics packaging [7-8]. In place of the solder ball, our modified test is conducted on single splats, with a stylus applying a tangential force along the substrate and shearing the splat. For these set of initial tests, commercially pure Ti splats were studied, with trends examined between the measured adhesion strength and experimental variables, including deposition velocity, substrate material and substrate properties.

2. Experimental Procedure

Cold sprayed specimens, consisting of single splats, were deposited by using a Kinetic 4000 (CGT GmbH, Germany) cold spray gun. The traverse speed of the cold spray gun over the substrate was 1 m/s and the feed rate of the powder to the gun was 2 – 5 g/min. The deposition was carried out with a nitrogen gas preheated to temperatures ranging from 300°C to 800°C and gas pressures that was varied from 2 to 4 MPa. Under these conditions, deposition velocities ranging from 580 m/s to 825 m/s were achieved. The particle velocity was measured in a free jet with a DPV2000 (Tecnar Automation, St. Bruno, QC, Canada).

The first set of samples consisted of 0-46µm feedstock Ti powder deposited on milled, commercially pure (CP) Ti plates with 3 mm thickness (McMaster-Carr, Aurora, OH, USA). The CP-Ti powder was produced by plasma atomization (Raymore Inc., Boisbriand, QC, Canada) and was previously characterized and found to have an average particle diameter for 29 µm [9].

The second set of samples consisted of cold spray splats deposited onto previously prepared cold sprayed CP-Ti coatings of varied coating thicknesses and varied deposition velocities on a CP-Ti plate. The coatings were deposited with a powder feeding rate of 20 g/min and a gun traverse speed of 0.33 m/s. The coating thickness was varied by changing the number of gun passes from 10 to 30 passes. The coatings were milled to a flat surface finish prior to deposition of splats. Table I lists all the samples and their respective deposition conditions.

The substrate with cold spray deposited splats of coatings were cut and mounted into a cold mount epoxy. The sample cross-section was then polished up to 0.05 μm colloidal silica. To measure the splat flattening ratio, the mounted cold spray splats were examined with light optical microscopy (LOM) with measurements made with image analysis software (Clemex Vision Professional 5.0, Clemex Technology Inc., Longueuil, QC, Canada). Splat flattening ratio, FR, was measured by taking a ratio of splat width to splat height. Porosity of cold spray coatings was also measured by LOM at a magnification of 200x. An average was obtained from three or more micrographs from random locations in the coating. High resolution imaging was performed with a variable pressure scanning electron microscope (Hitachi S-3000N VP-SEM, Japan). High resolution images of the cold spray deposited splats were taken in plan view and in cross-section.

The root-mean-square (RMS) surface roughness of the cold spray coatings was determined from 50 x 50 micron scans with an atomic force microscope (Veeco Dimension 3100, Santa Barbara, CA, USA) operating in contact mode. The hardness of the Ti coatings and the bulk Ti plate was measured with instrumented indentation (Hysitron Ubi3, Minneapolis, MN, USA). An indentation of 1 mN load and 200 $\mu\text{N/s}$ indentation load was carried out on the coating surface that was polished to 0.05 μm colloidal silica. A matrix of 49 indents was evenly

spaced in the coating region that was 300 μm x 300 μm in size. The indent spacing of 43 μm was maintained.

Shear testing was performed using a Micro-Combi Scratch Tester (CSM Instruments, Inc., Needham, MA, USA) with a semicircular-tipped stylus (see Fig. 1). Prior to testing, the size of the each splat was measured by a light optical microscope attached to the instrument. The average diameter was taken from an optical image and the height was found by measuring the difference in focal height between the substrate and the top of the splat. These measurements were also used to calculate a flattening ratio of the splats tested.

During shear testing, the applied normal force was held constant via a force-feedback loop at 30 mN for most splats and 300 mN for splats deposited at high deposition velocities. The substrate moved at a speed of 150 $\mu\text{m}/\text{min}$ with respect to the stylus. During each shear test the stylus tangential force, the stylus height as modified by the force-feedback loop, and acoustic emissions were recorded. When the stylus encountered the splat edge during testing, tangential force gradually increased to a peak and then dropped sharply at splat failure (see Fig. 1). Between four and ten splats were shear tested per sample.

The data obtained from shear testing of the splats was analyzed in two ways. First, an adhesion strength was calculated as the peak tangential force, $F_{T\text{ Peak}}$, from which the baseline tangential force, $F_{T\text{ Baseline}}$, had been subtracted, divided by projected area of the splat.

$$\text{Adhesion Strength (MPa)} = \frac{F_{T\text{ Peak}} (\text{mN}) - F_{T\text{ Baseline}} (\text{mN})}{\text{Projected Splat Area } (\mu\text{m}^2)} * 1000 \quad \text{Eq. 1}$$

For the second method, the energy of de-adhesion was calculated for each shear test by integrating the area under the tangential force curve from the point of splat-stylus contact to the point of failure. This was normalized to the projected area of the splat. Prior to integration, the baseline friction force data was subtracted from the peak.

$$\text{Adhesion Energy (kJ m}^{-2}\text{)} = \frac{\int_{\text{Contact}}^{\text{Failure}} [F_T(x) - F_{T \text{ Baseline}}] dx \text{ (mN } \mu\text{m)}}{\text{Projected Splat Area (}\mu\text{m}^2\text{)}} \quad \text{Eq. 2}$$

3. Results and Discussion

Splat Morphology

In Figure 2, SEM images of cold spray splats deposited at increasing deposition velocity condition are presented. The splats deposited at low deposition velocity demonstrate a low degree of splat deformation. Splats retain their original spherical morphology. As the deposition velocity increases, the splats become more deformed and pancake-shaped.

Splats deposited at varied deposition velocity conditions demonstrated material jetting at the splat impact site. This phenomenon is generally believed to be the formation of an adiabatic shear instability and the primary bonding mechanism for cold sprayed particles [10-13]. However, at lower deposition velocities, the jetting region is barely visible (c.f. Fig. 2(a) and 2(d)). With increasing deposition velocity, the jetting region becomes more evident in both plan view images (see Fig. 2(b) and 2(c)) and cross-section (see Fig. 2(e) and 2(f)).

The extent of deformation during splat impact and bonding was explored through measurements of the flattening ratio. Results from the two techniques, from images of splat cross-sections and from plan view observation with the optics on the scratch tester, were found to be statistically very similar to one another. The two datasets were combined and averaged. A total of 8-25 splats were measured for each deposition condition. The trend of flattening ratio with deposition velocity is plotted in Figure 3. Consistent with the micrographs presented in Figure 2, the flattening ratio was found to increase with increasing deposition velocity. While the flattening ratio remains somewhat constant at a value of about 2.2 for deposition velocities below 650 m/s, above this speed, the ratio increases up to a value of around 3.5 for splats deposited at 825 m/s.

Shear Testing – Cold Spray Ti on Bulk Ti

Measurements of adhesion strength and adhesion energy for Ti splats on Ti plates are presented in Figures 4(a) and 4(b). Both methods of analysis reveal similar trends of increasing adhesion with increasing deposition velocity. This result is in good agreement with the observations of splat morphology. At lower deposition velocities, limited jetting occurs and the splats appear to be bonded by mostly conformal adhesion, which is a relatively weak bonding mechanism. As deposition velocity increases, the jetting region becomes more pronounced. This phenomenon is tied to the formation of an adiabatic shear instability that provides a temperature rise at the bonding interface [10-13]. Thus, the degree of metallurgical bonding would also increase with increasing deposition velocity.

Two other interesting trends are found in Figure 4. First, splats deposited at 500, 750 and 800°C pre-heat temperature always had a higher average adhesion compared to those deposited at 300°C. This trend is observed even for the case where splats deposited at 300°C and 4 MPa had a higher deposition velocity than splats deposited at 500°C and 2 MPa. If differences in gas pre-heat temperature result in particles arriving to the substrate at different temperatures as well as different velocities, those particles at higher temperatures will be more easily deformed and should be more strongly bonded. To see if this temperature effect is significant, more work is necessary at these overlapping conditions of gas pre-heat temperature and pressure that lead to similar velocities.

The second trend in Figure 4 is that the analysis for adhesion energy versus that of adhesion strength gives a slightly different trend for the highest deposition velocities. For the adhesion strength measurement, data at 770 and 825 m/s show very similar adhesion, whereby

analyzing the energy, the trend has splats at 825 m/s with higher adhesion. This may be some indication of the failure mode of the splat. By including the width of the peak in the analysis, some accounting of the failure mode of the splat is made (i.e. brittle versus ductile failure). So, the trend in the adhesion energy plot may be a better indication of the extent of metallurgical bonding that occurs.

Shear Testing – Cold Spray Ti on Cold Spray Ti Coating

The bonding of Ti splats to a coating of cold sprayed material was also investigated. As shown in Table I, two deposition conditions were chosen both for coatings and splats: 500°C, 3 MPa (694 m/s) and 800°C, 4 MPa (825 m/s). Figure 5 compares the results for adhesion strength for the lower deposition condition and higher deposition condition on the various substrates. For the lower deposition condition, the adhesion of these splats to cold sprayed material was lower compared to the values on the Ti plate. However, at the higher deposition condition, the adhesion strength to the cold spray coatings was always as strong as or stronger than the adhesion to the Ti plate.

The main difference between the cold spray coatings and the Ti plate was the level of porosity and the mechanical properties. Table II lists the properties for these materials. The Ti plate has very minimal porosity and a hardness of around 3.2 GPa. The cold spray coatings have higher porosity, but also a higher hardness as compared to the Ti plate. The surface roughness of the coatings is comparable to the Ti plate. Upon impact, cold sprayed particles deform and develop a jetting region. These processes depend on both the mechanical properties of the particle and the substrate, with softer substrates allowing for an overall increase in deformation and jetting [14]. The results for the lower deposition condition have stronger adhesion to the softer substrate compared to the harder cold sprayed coatings. In contrast, the higher deposition

condition appears to be relatively insensitive to the mechanical properties of the substrate. Of course, the range of properties realized here is small. Deposition on softer or harder materials than those studied here may result in a variation in adhesion for the higher deposition conditions as well.

Comparison to Literature

Only a few studies of cold sprayed Ti report a bond strength. Marrocco, et. al. [15] measured a bond strength of around 20 MPa for Ti coatings produced from non-spherical Ti powders. Their coatings showed porosity in the range of 17-24% [15], much higher than was realized for coatings produced with similar conditions to this study [10]. Wang, et. al. [16] studied cold sprayed Ti coatings produced from an angular powder. The best bond strength was around 20 MPa for a coating with 1.6% porosity. Higher porosity levels resulted in lower bond strengths.

Higher bond strengths are found for cold sprayed coatings produced with spherical powder [17]. Bae, et al. measured a bond strength of around 48 MPa for Ti sprayed at 650 m/s onto a CP-Ti substrate. When they sprayed with He at velocities of 950 m/s, their bond strength exceeded the strength of their epoxy (greater than 85 MPa).

At the lowest deposition conditions, our results are similar in magnitude to those found in the literature for bond strength measured by traditional means on cold sprayed Ti. When the deposition conditions increase, velocity and temperature, the adhesion strength rises above 100 MPa and eventually reaches roughly 240 MPa. These are strengths that are practically unmeasurable with any traditional bond strength test. An accepted value for the ultimate shear strength of CP-Ti is 380 MPa [18]. If bonding between splats is completely metallurgical bonding, this value may be considered an upper limit for our measurements. As deposition

velocity increases, our measurements of adhesion strength approach this value, reaching approximately 63% of the bulk shear strength. Thus, considering the bulk properties of Ti and the trend of an increase in metallurgical bonding for the highest deposition conditions, our technique appears to provide a reasonable measurement of the adhesion strength.

4. Conclusions

A modified ball bond shear test was developed for determining the adhesion strength of CP-Ti splats deposited by cold spray to various substrates. Results were presented for CP-Ti splats on a CP-Ti plate and on coating previously cold sprayed onto Ti plates. For deposition onto CP-Ti plate, the adhesion strength and adhesion energy rose as a function of deposition velocity. The highest adhesion strengths were observed for velocities above 770 m/s and were on the order of 240 MPa. Splats were also deposited onto previously cold sprayed material. When the splats were deposited at high velocity (825 m/s), the adhesion strength was independent of the material being deposited onto. When the splats were deposited at lower velocity (694 m/s), the adhesion strength was lower when deposited onto cold spray material as compared to the results on CP-Ti plate.

5. Acknowledgements

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Table I – A list of the cold spray specimens produced for this study.

Splats (Temperature, Pressure)	Velocity (m/s)	Substrates, Thickness in mm
Ti 300°C, 2 MPa	580	CP-Ti plate, 3
Ti 300°C, 3 MPa	625	
Ti 300°C, 4 MPa	642	
Ti 500°C, 2 MPa	636	
Ti 500°C, 3 MPa	694	CP-Ti plate, 3 Ti 500°C, 3MPa, 10 pass (0.8) Ti 500°C, 3MPa, 30 pass (2.7) Ti 800°C, 4MPa, 10 pass (0.7) Ti 800°C, 4MPa, 30 pass (2.5) } Onto CP-Ti plate, 3
Ti 500°C, 4 MPa	724	CP-Ti plate, 3
Ti 750°C, 3 MPa	770	CP-Ti plate, 3
Ti 800°C, 4 MPa	825	CP-Ti plate, 3 Ti 500°C, 3MPa, 10 pass (0.8) Ti 500°C, 3MPa, 30 pass (2.7) Ti 800°C, 4MPa, 10 pass (0.7) Ti 800°C, 4MPa, 30 pass (2.5) } Onto CP-Ti plate, 3

Table II – Properties of the substrate and cold spray coatings

Sample	Roughness RMS (μm)	Hardness (GPa)	Porosity (%)
Ti Plate	0.42	3.3 ± 0.5	< 1
Ti 500°C, 3 MPa, 10 pass	0.46	3.7 ± 0.2	1.6
Ti 500°C, 3 MPa, 30 pass	0.29	4.3 ± 0.3	1.2
Ti 800°C, 4 MPa, 10 pass	0.26	3.9 ± 0.5	1.0
Ti 800°C, 4 MPa, 30 pass	0.25	4.0 ± 0.3	1.7

Figure Captions

Figure 1: A schematic of the semicircular stylus tip (left). A photomicrograph of a splat shown to scale with schematic stylus tip (middle) and the testing direction indicated. A peak in tangential force during a successful shear test (right).

Figure 2 - SEM images of top view a)-c) and cross-section d)-f) of cold spray splats deposited on CP-Ti plate at a) and d) 300°C 4 MPa (642m/s), b) and e) 500°C, 4 MPa (724m/s) and c) and f) 800°C, 4 MPa (825/s) gas preheat temperature, gas pressure and particle deposition velocity. An increasing degree of splat deformation and jetting is observed as the deposition velocity increases.

Figure 3: Average flattening ratio of Ti splats on Ti substrate with a standard deviation error bar. Square data points represent splats deposited at 300°, triangles at 500°, and circles at 750° and 800°.

Figure 4 – The shear test results for titanium cold sprayed on titanium plate. Square data points represent splats deposited at 300°, triangles at 500°, and circles at 750° and 800°.

Figure 5 - Shear test results from cold spray splats on cold sprayed titanium coatings compared to cold spray on solid Ti plate.

Figure1

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Figure2

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Figure3

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