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Interface melding in cold spray titanium particle impact

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ABSTRACT

A focused ion beam (FIB) was used to characterize deformation and bonding behavior of titanium particles, cold sprayed onto a titanium surface. By milling away half of each particle a cross-section was obtained, revealing the particle–substrate interface. It was found that in some sections, voids lined the interface due to mismatch in particle- and substrate-surface topologies. In other places, the interface could not be discerned due to closure of the voids. It is shown that this effect was related to adiabatic shear instability, which caused violent jetting, ejection of molten material from the particle, and thermal softening of the interfacial layers. Deformation characteristics and variation in splat profiles due to microstructural influences, and the effect of particle size are also discussed.

1. Introduction

Cold spray is a process for the building up of coatings by high velocity impact of powder particles [1]. The particles, typically 1–50 μm in size, are accelerated within a supersonic gas stream towards a substrate surface. Unlike in other thermal spray processes, the material is not melted in-flight. Therefore, coatings may be deposited that are free from quenching stresses, oxidation and other thermal defects.

There have been several studies of cold sprayed titanium [2–9]. Titanium and its alloys exhibit high strength-to-weight ratios, oxidation resistance, corrosion resistance and biocompatibility which make them an attractive choice for demanding applications. By cold spray, titanium has a higher critical velocity for deposition than other, more ductile metals [8]. Nevertheless, a deposition efficiency (fraction of sprayed powder which remains in the deposit) of 100% is readily obtainable using relatively inexpensive N_2 as the accelerating gas [7]. Additive manufacture of titanium components by cold spray (spray forming) is also being actively pursued [3,4]. In order to achieve high strengths within cold sprayed Ti parts and coatings it is necessary to understand the mechanism of bonding between cold spray particles.

Bonding of cold spray particles comes about as a result of the plastic deformation that occurs upon impact. Owing to their small size, the deformation period (or contact time) of individual particles is generally less than 100 ns [10]. Deformation occurs at high strain rates (up to ~10^7 s^{-1}) under near-adiabatic conditions. Plastic strain is highly localized in a layer of material at the interface where intense shearing occurs. This interfacial shearing is manifest as thin, outward-moving 'jets'. In finite element models (FEM) of particle impact the velocity needed to induce adiabatic shear instability has been shown to correspond to the experimentally-determined critical velocity for deposition [11,12]. Thus, there appears to be a dependence of particle adhesion on the interfacial jets. Mechanistically, it has been proposed that the jetting action is violent enough to disrupt passive oxide films, allowing intimate contact of the metal surfaces and formation of metallic bonds across the interface [13,14].

FEM also shows that the temperature rise due to adiabatic plastic deformation within the interfacial jets generally approaches or may even reach melting point. The latter is supported by empirical observation of 'splashes', spheroidized ejecta [15,16] and other signs of melting. For instance, in bimetallic systems (e.g. when copper particles are sprayed onto aluminum) intermetallic compounds form at the interface due to melting [17–19].

Away from the interface within the bulk of the particle, strains are considerably lower and there is minimal temperature rise [11]. In other words, melting is an interfacial phenomenon only, and the vast bulk of the cold sprayed material remains well within the solid state.

A further, important outcome of FEM simulations is that when strains and temperatures are mapped over the particle–substrate interface, it is found that they are much greater in the peripheral zone (i.e. within the shear jets) than at the base (so-called south pole [11]) of the particle [11,17]. Thus if bonding is associated with adiabatic shearing, then only a fraction of the contact area may in fact be bonded. This has implications for the adhesion of cold sprayed coatings onto surfaces, as well as for the cohesive strength within the deposits themselves. Assadi et al. measured the bond strength copper coatings and obtained a value 20% of the ultimate tensile strength of copper. This

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figure, they noted, was in good agreement with their modeling of 580 m/s Cu-on-Cu impact, in which only 15–25% of the interface was found to be subject to shear instability [11].

There is however, only a small body of experimental evidence that directly links bonding with the peripheral shear zones. It has been done by examination of rebound sites of metallic particles against rigid surfaces. When aluminum particles strike a ceramic surface [15] or when Al–Si particles impact mild steel [20], a thin, Al-rich layer is left behind due to a brief, adhesive interaction between the two bodies. The layer is generally ring shaped, due to this interaction occurring predominantly at the edges of the particle. In another study, the 'scrubbing' action of Ti particles' jets has been demonstrated by impact onto a gold-plated surface. Abrasive thinning or removal of the 30 nm Au film was found to occur, again in a ring-shaped pattern [21].

Although in the broader literature a number of different cold spray bonding scenarios have been investigated, the one with the broadest practical relevance is that of a particle impacting a surface composed of the same material, as it relates to the process of coating buildup. Therefore, in this study the impact of titanium particles onto a flat, polished titanium surface was investigated. While a cold sprayed surface is far from microscopically flat, the flat surface is nonetheless a simpler geometry which facilitates a more controlled study of particle deformation and bonding. Furthermore it is hoped that the obtained data will serve as a useful empirical base for computer models, which tend to focus on the spherical particle – on – flat surface geometry. Selected particles were milled with a focused ion beam (FIB) to make an exact cross-section. This revealed the total shape of the deformed particle and substrate. It also allowed close examination of the particle–substrate interface to determine the location at which bonding was more favorable.

2. Experimental methods

Gas atomized grade 2 titanium powder (TLS Technik, Bitterfeld, Germany) was chosen as the feedstock (Table 1). The particle size distribution was determined by laser measurement using a Malvern Mastersizer X (Malvern Instruments Ltd., Malvern, Worcestershire, UK). The median particle size (d_{50}) was found to be 21.89 μm, $d_{10} = 12.12$ μm, and $d_{90} = 34.17$ μm. The particle morphologies were approximately spherical (as seen in the SEM shown in Fig. 1) although from measurements of particle dimensions in two orthogonal directions a mean particle aspect ratio of 1.12 was calculated. Small 'satellite particles' attached to larger ones were relatively few.

The substrates were 11 mm-thick disks cut from round, 25.4 mm-diameter grade 2 titanium bar stock. An average grain size of 17.4 ± 0.8 μm was measured by optical image analysis. The flat surface of the disks was polished using progressively finer diamond suspensions with applied force limited to 10 N. The final polishing step was done with 0.25 μm colloidal silica.

Cold spray was performed with a CGT Kinetiks® 4000 system. N_2 carrier gas was accelerated through a CGT 24TC converging–diverging nozzle with circular cross-section. This nozzle had a diverging section length of 129 mm, throat diameter of 2.7 mm and an exit diameter of 6.6 mm. The chosen gas stagnation temperature and pressure were 600 °C and 3.0 MPa, respectively. The cold spray gun was held by an ABB IRB 2600 robot arm.

Titanium splats were deposited on the polished samples using a method that differed from the commonly used 'wipe test' [15,22]. In the wipe test, the nozzle is moved quickly past the sample to spatter particles in a line across the surface. This method suffers the following limitations.

- Due to divergence of the beam it is not possible to distinguish particles that arrived at the surface along a straight trajectory in the center of the beam, from those that were deposited slightly ahead or slightly behind.
- The temperature of the surface at the moment of impact of the particles cannot easily be measured or calculated.

Others [19] have used a mask between the gun and substrate which opens briefly, usually by moving the mask so that a slit comes momentarily in line with the particle jet. The problem of determining the particle position within the diverging beam is also solved by this approach.

In this study, the cold spray jet was moved onto the polished disk (at a nozzle standoff distance of 30 mm) and held there for 40 s to ensure that the substrate surface temperature had reached a steady state condition. The powder feed was then briefly turned on and then off. The nozzle was held in position for a further ~2 s, to allow any residual particles in the feed lines to exit through the nozzle, and then was moved away. A circular spot pattern resulted, with particles more concentrated in the center, and diminishing in frequency with distance, until no particles were found >5.5 mm from the center. This method was used to spray several disk samples. However upon inspection under a microscope, only two were chosen for further study on the basis of their

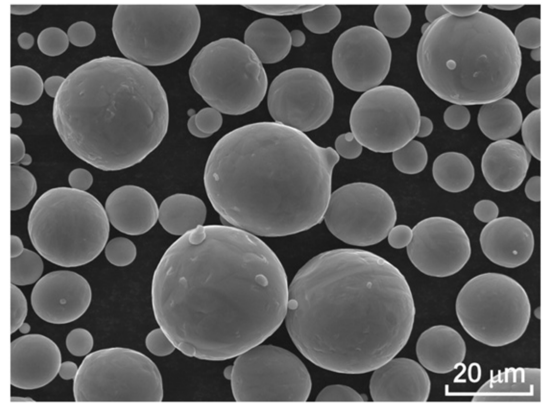

Fig. 1. Scanning electron micrograph of titanium powder feedstock.

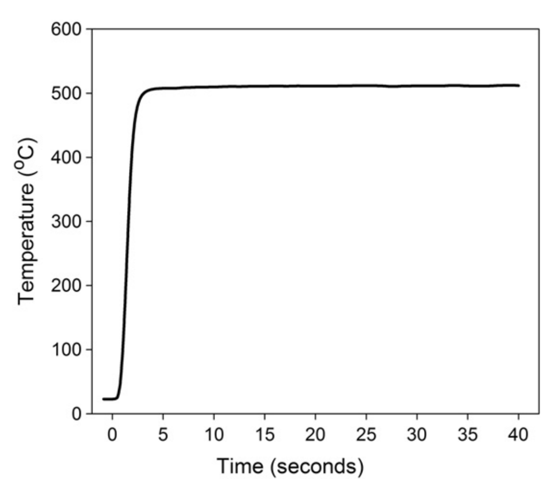

Fig. 2. Substrate surface temperature rise due to cold spray gas jet impingement.

Table 1
Titanium powder composition (from supplier, TLS, Germany).

	C	Fe	O	N	H	Zn	Ti
Element-%	0.004	0.03	0.18	0.008	0.001	<0.002	remainder