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Aluminum powder of 99.7 wt.% purity and in the nominal particle size range of $-75+15 \,\mu$ m has been sprayed onto a range of substrates by cold gas dynamic spraying (cold spraying) with helium, at room temperature, as the accelerating gas. The substrates examined include metals with a range of hardness, polymers, and ceramics. The substrate surfaces had low roughness ($R_a < 0.1 \mu m$) before deposition of aluminum in an attempt to separate effects of mechanical bonding from other forms of bonding, such as chemical or metallurgical bonding. The cross-sectional area of a single track of aluminum sprayed onto the substrate was taken as a measure of the ease of initiation of deposition, assuming that once a coating had begun to deposit onto a substrate, its growth would occur at a constant rate regardless of substrate type. It has been shown that initiation of deposition depends critically upon substrate type. For metals where initiation was not easy, small aluminum particles were deposited preferentially to large ones (due to their higher impact velocities); these may have acted as an interlayer to promote further building of the coating. A number of phenomena have been observed following spraying onto various substrates, such as substrate melting, substrate and particle deformation, and evidence for the formation of a metal-jet (akin to that seen in explosive welding). Such phenomena have been related to the processes occurring during impact of the particles on the substrate. Generally, initiation of aluminum deposition was poor for nonmetallic materials (where no metallic bonding between the particle and substrate was possible) and for very soft metals (in the case of tin, melting of the substrate was observed). Metallic substrates harder than the aluminum particles generally promoted deposition, although deposition onto aluminum alloy was difficult due to the presence of a tenacious oxide layer. Initiation was seen to be rapid on hard metallic substrates, even when deformation of the substrate was not visible.

Keywords bonding, cold gas dynamic spraying, cold spraying, deformation

1. Introduction

The process of cold gas dynamic spraying (cold spraying) has been developed for over a decade. Investigations have mainly focused on (a) gas dynamics and the design of the convergent/ divergent nozzle (Ref 1-5) and (b) the spraying characteristics of various materials (Ref 5-9). There are relatively few reports addressing the details of interaction between impacting particles and the substrate, that is, the bonding mechanism (Ref 10). A number of researchers have likened the bonding mechanisms in cold spraying with those identified in explosive welding, where bond formation relies on deformation under high pressures (Ref 11). Other workers have examined the possibility of melting of the materials close to the interface during impact and the role of such a phenomenon in bond formation.

A distinctive phenomenon in the process of explosive welding is the generation of a solid-state metal jet. The jet is formed from materials removed from the surfaces under very high static pressure generated from the kinetic energy of the flyer plate. Explosive welding depends upon the formation of clean surfaces by metal jetting and subsequent intimate contact. Using the results of experimental studies on aluminum particles deposited on a rigid substrate by cold spraying, Papyrin et al. (Ref 12) modeled the impact of a plastic particle onto a rigid substrate at velocities that are commonly achieved in the cold spray process. The calculated distribution of the radial component of velocity indicated that metal jetting could take place. However, they also demonstrated that under certain conditions, melting may occur on the surface of the particle in the contact zone but suggested that since this was limited to only a very thin layer, it would not significantly affect the properties of the coating. Similarly, other workers have also argued that both surface melting and the formation of clean metal surfaces by processes akin to explosive welding may contribute to bond formation (Ref 8, 13). However, Dykhuizen et al. (Ref 10) found evidence for the formation of a metallic jet and demonstrated that neither the particle nor the substrate need to be molten to obtain high bond strengths in this process. In recent work, Van Steenkiste et al. (Ref 14) described the deposition of large aluminum particles (> $50 \mu m$) onto a brass

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substrate by cold spraying; they argued that particle melting does not occur, with bonding resulting from severe deformation and subsequent disruption of oxide films on metallic particles allowing nascent metal surfaces to come into contact.

While the possibility and role of particle melting in the cold spray process is seen by most workers to be small, there is some evidence to support its occurrence. Bolesta et al. (Ref 15) deposited aluminum by cold spraying onto a nickel substrate. Using x-ray diffraction, they detected the formation of Ni_3Al and suggested that the interface phase was in the region of 200 to 500 Å in thickness. This indicates that melting may occur as a precursor to the formation of the new phase; such bonding was referred to as a chemisorptional bond.

Alongside the workers who have sought to understand bonding on an atomic level, others have also examined the role of surface topography on the formation of a bond between incoming particles and substrate. Tokarev suggested that particles impacting a substrate in cold spraying first activated the substrate by roughening it; only once this had occurred was a coating able to initiate and grow (Ref 16). Vicek et al. (Ref 9) examined the impact of a range of powder types onto a range of substrate materials in cold spraying. They explained differences in the ability of particles to deposit in terms of the mechanical properties of the particles and substrate and the specific impulse of the impact. They related bonding primarily to the relative ease of deformation of the substrate and particle and concluded that if the particle was significantly more deformable than the substrate, adhesion was not possible.

This article seeks to address the role of substrate material types in the formation of a bond between aluminum particles and the substrate in cold spraying. Bonding is shown to be dependent on the nature of the substrate material and not just on its mechanical properties. Evidence for the formation of a metallic jet is presented along with evidence that melting may occur in specific cases.

2. Experimental Method

2.1 Powder and Substrates

The powder used for cold spraying was a commercially pure (99.7% Al) aluminum powder (Alpoco, Minworth, UK). The particle size was in the range of $-75 + 15 \mu m$; other reports indicate that large aluminum particles have been successfully deposited by cold spraying (Ref 14). The particle morphology was examined by scanning electron microscopy (SEM), and the size distribution was analyzed using a Malvern Mastersizer-S laser particle size analyzer (Malvern Instruments, Malvern, UK). The powder specimen for SEM analysis was prepared by scattering powder onto an adhesive carbon disk so that particles of all sizes could be observed in their original proportion in the powder. The Vickers microhardness of the powder was measured to be 0.315 GPa with a 10 gf load on the indenter.

A wide range of substrate materials were used (Table 1) onto which aluminum tracks were deposited by cold spraying. All substrate materials were used in their as-received condition except the tool steel, which was given various heat treatments to change its hardness. All metallic materials and the alumina were ground and polished, the final polish being conducted with 1 µm diamond paste. The acrylonitrile butadiene styrene (ABS) polymer and glass were used with their as-received surface finish. The hardness of most materials were measured with a Vickers macrohardness tester under a load suitable for the material in question. The hardness of glass was, however, measured with a Vickers microhardness indenter with a 200 gf load. Table 1 shows the range of hardness of the substrates used in this work.

2.2 Rig and Spraying Parameters

Cold spraving was carried out at the University of Nottingham using an in-house constructed cold gas dynamic spraying system. A schematic diagram of the spraying system is shown in Fig. 1. Room-temperature helium was used as the driving gas for particle acceleration and for powder feeding. Helium was selected as it has been shown to allow the development of the very high particle velocities required for cold spraying even at room temperature (Gilmore et al. (Ref 17) demonstrated that particle velocities were ~150 m/s higher when accelerated with room temperature helium than when accelerated with air heated to any temperature up to 500 °C). A De Laval type nozzle was used to accelerate the gas to a supersonic speed. The main driving gas and the carrier gas were supplied from separate sources, with the carrier gas pressure 0.1 MPa above that of the main driving gas to ensure steady feeding of the powder into the main gas stream. The maximum stagnation pressure available for the driving gas was 4 MPa. A Praxair 1264HP (Praxair Surface Technologies, Indianapolis, IN) powder feeder was used, which

Table 1Type and hardness of substrates used for coldspray deposition

Substrate	Specification	Hardness, GPa
Tin	99.5 wt.% Sn	0.08
ABS		0.17
Copper	C105	0.95
Al alloy	6063	0.97
Brass	C260	1.1
Tool steel (normalized)	BS B01	2.18
Carbon steel	AISI 1040	2.33
Glass	Soda-lime	4.45
Tool steel (quenched and tempered)	BS B01	6.22
Tool steel (quenched)	BS B01	8.05
Alumina		10.7



Fig. 1 Schematic diagram of the cold spray system

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can work at a pressure of up to 3.4 MPa. The nozzle was fixed vertically to a frame; the samples moved below the nozzle on a computer-controlled X-Y table that allowed raster scans and tracks of different shapes to be deposited. All coatings were single track and single pass and were deposited with a stagnation pressure of 2.5 MPa at ambient temperature, with a 27 mm/s traverse velocity, a 20 mm standoff distance, and a powder feed rate of 10 g/min.

2.3 Characterization

The cross-sectional area of a single track deposited by cold spraying was estimated by profilometry (Surfcom, Leicester, UK). Five profiles were taken across the track in a direction perpendicular to the traverse direction. The cross-sectional area of the track was taken as an average of the individual cross sections measured.

Substrate surfaces, following deposition of aluminum powder, were examined using SEM with both backscattered electron (BE) imaging and secondary electron (SE) imaging. In cases where deposition was significant, areas toward the edge of the track (where substrate could still be seen) were observed to allow phenomena associated with the initiation of bonding between the particles and the substrate to be explored.

3. Results

Figure 2(a) shows the secondary electron (SE) image of the as-received aluminum powder, while Fig. 2(b) shows the cumulative particle size distribution of the powder. It can be seen that the proportion of particles less than 5.0 µm is very low and that particles generally had a rounded morphology, although some particles were elongated and others had attached satellite particles.

Figure 3 shows the deposit cross section following spraying under standard conditions onto various substrate materials. The hardness of the various substrate materials are shown in Table 1. It can be seen that there is a wide variation in cross section of the deposited tracks, with some tracks exhibiting cross-sectional areas up to around 0.2 mm² (with a corresponding thickness of the order of 100 µm). The most successful deposition was achieved on metallic substrates with hardness greater than 2 GPa. Deposition onto nonmetallic substrates was observed to yield a very thin, interrupted deposit in all cases.

Figures 4 to 8 show SEM images of substrates onto which aluminum particles were deposited. Since the purpose of this investigation was to examine the bonding between the substrate and the deposit (and not bonding within the deposit as it grew), these images were taken from areas at the edges of deposits where the substrate was still clearly visible, but where some interaction with the aluminum particles could be observed. Only micrographs of representative substrates are presented.

Figure 4 shows images of a tin substrate following deposition of aluminum. Here, the substrate was considerably softer than the incoming particles and exhibited significant deformation. Figure 4(a) (SE) shows the cratered surface, with some large crater sizes. The corresponding BE image (Fig. 4b) shows that the large aluminum particles have simply produced craters in the soft tin substrate whereas a relatively high density of very small



Particle size / µm

Fig. 2 (a) Morphology and (b) size distribution of the aluminum feedstock powder



Fig. 3 Cross-sectional area of cold sprayed aluminum track as a function of substrate hardness and type



Fig. 4 SEM images of the surface of a tin substrate following cold spray deposition of aluminum. (a) and (b) are SE and BE images, respectively, of the same area. (c) and (d) are higher magnification SE and BE images respectively of the same area.



Fig. 5 SEM-SE image of the surface of an ABS substrate following cold spray deposition of aluminum

 $(<5 \ \mu m)$ aluminum particles (the dark gray features) have been deposited. This is notable in light of the low proportion of particles in this size range in the powder feedstock (Fig. 2b). Figure

4(c) shows a higher magnification image of one of the large craters surrounded by an array of smaller craters and deposited particles. Again, Fig. 4(d) shows the corresponding BE image. The large crater shows solidification features (the cellular surface structure) that clearly indicate that melting of the tin surface has occurred. Energy dispersive x-ray (EDX) analysis of the solidification features reveals no significant presence of aluminum, presumably due to the low solubility of aluminum in tin. Again, empty impact craters down to about 5 μ m can be seen with all particles that have deposited (the dark features associated with the low atomic number of aluminum) being below this size range.

Figure 5 shows the surface of a polymer, ABS, following cold spraying of aluminum. While a few very small aluminum particles can be seen on the surface, the main feature of the surface is the disruption caused by the impacting particles. The relatively hard aluminum particles have caused deformation and tearing of the surface. Deposition of particles onto the ABS has clearly not been successful.

Figure 6 shows the surface of the aluminum alloy substrate following cold spraying of aluminum. Figures 6(a) and (b) show that there are a large number of very small particles bonded to



(a)





Fig. 6 SEM-SE images at different magnifications of the surface of an aluminum alloy substrate following cold spray deposition of aluminum

the surface along with empty craters in a range of sizes where particles have impacted but not bonded. Some large particles have bonded to the surface; it is not clear whether these have bonded directly to the surface of the substrate or whether these have been deposited on top of small particles that have already bonded at that location. Although the aluminum alloy is significantly harder than the aluminum particles, significant deformation of the substrate can be seen following impact (Fig. 6b). Also, lips of material can be observed around the edges of the largest particle in Fig. 6(b), which may indicate high shear (Ref 10).

Figure 7 shows the surface of the normalized tool steel substrate following cold spraying of aluminum. Figure 7(a) shows a number of large aluminum particles that have bonded to the substrate. The particles have clearly been heavily deformed with lips of material at the edges of the larger particles. What is also noticeable is that the high density of small aluminum particles (as seen on some of the other substrates) is now not observed (Fig. 7b). Figure 7(c) indicates that the deformation of the steel substrate following impact by aluminum is very small. Crosssectional micrographs have confirmed this, with no deformation at all being detectable for the two harder steels. Figure 7(a) also shows the presence of small particles on the surface of the steel between the aluminum particles. These particles have a highly elongated form (Fig. 7c). Figure 7(b) (a BE image corresponding to Fig. 7a) shows that these particles are not aluminum but instead come from the steel substrate. Figure 7(d) shows an EDX dot map for aluminum of the image in Fig. 7(c), which again indicates that these elongated particles are iron-based. All of these particles are highly elongated with a very narrow cross section. Similar particles have not been observed on the softer metallic substrates or on any of the nonmetallic substrates, but are observed for all the steel specimens across the range of hardness.

Figure 8 shows the surface of the glass specimen following cold spraying of aluminum. While some aluminum particles are bonded to the surface, fracture of the glass, typical of erosion damage can also be observed. It is not clear whether the particles that have bonded have done so mechanically due to the increased roughness of the damaged areas on the glass or by another mechanism.

4. Discussion

Vicek et al. (Ref 9) have discussed the bonding of particles in cold spraying primarily on the relative deformability of the particles and the substrate. They indicated that as the substrate deformability decreased, the ease with which particles bond to the surface also decreased. This simple correlation has not been observed in this work. Figure 3 shows the deposit cross-sectional area for different substrate types. Given that the growth rate of the layers is expected to be the same (during growth of a cold sprayed deposit, particles are bonding onto particles that have already been deposited), differences in behavior for the different substrates must be associated with initiation of growth, that is, the ease with which particles can bond with the substrate. It is clear that the ease of coating initiation depends on a number of factors, which are discussed below.

4.1 Interaction with Soft Metals

Of all the metals examined, only tin was softer than the aluminum particles that were being cold sprayed. Very few particles have been deposited onto the tin surface, and those that have are very small ($<5 \mu m$ in diameter; Fig. 4). Figure 2(b) shows that the volume fraction of such particles is very small in the feedstock powder, indicating that they are deposited preferentially, possibly due to their higher velocity in the gas stream. It is clear that the impact of many of the larger aluminum particles on the soft tin has resulted in melting. Hutchings (Ref 18) estimates that more than 80% of the kinetic energy of an impact is released as heat. Whether melting occurs will depend upon the heat capacity of the volume over which the heat is dissipated, the time over which it is dissipated, and the relevant thermal diffusivities and melting points. The low melting point of tin has undoubtedly led to the observation of melting in this case when it has not been observed in other materials. The extremely low mutual solubility of the two metals and the short period during which the melt exists has resulted in no evidence of aluminum in the melted structures.





(a)

(c)



(b)



Fig. 7 SEM images of the surface of a normalized tool steel substrate following cold spray deposition of aluminum. (a) and (b) are SE and BE images, respectively, of the same area. (c) Higher magnification SE image. (d) EDX dotmap for aluminum corresponding to the area shown in (c)

(d)

20 µm

The deposition of aluminum onto the tin substrate was very difficult (Fig. 3). On impact with the particles, the tin melted; the strength of the interface was therefore very low and as such the aluminum particles were able to easily rebound from the surface. Only very small aluminum particles were deposited onto the tin substrate. Whether these formed a real bond or whether they are simply embedded into the surface is at present unclear.

4.2 Interaction with Hard Metals

All of the other metals onto which the aluminum was sprayed can be regarded as hard with respect to the aluminum particles. However, only the hard steel substrates exhibited no deformation following spraying. The degree of deformation in the softer of these metals (e.g., aluminum alloy) was considerable despite being much harder than the impacting particles (e.g., Fig. 6). Again, the preferential deposition of small aluminum particles onto the surface is evident, although some larger particles can be seen bonded to the surface. The bonding of the smaller particles (impacting with a higher velocity) may be preferred as it is these particles that are best able to disrupt the tenacious oxide laver that is known to exist on aluminum surfaces. It is not clear

whether the deposition of small particles is necessary as a bond layer for subsequent deposition of the larger particles, or whether the large particles may bond to the surface of the aluminum alloy substrate directly.

Figure 3 shows that the ease of deposition initiation of the aluminum was significantly lower for the aluminum alloy substrate than for the brass or copper substrates, despite all these substrates having very similar hardnesses (Table 1). The aluminum alloy does have the lowest elastic modulus of the threesubstrate materials, along with the most tenacious oxide layer, and these factors may result in the significant differences observed in ease of initiation.

The interaction of the aluminum particles with the harder steels was different from those of the nonferrous metals in a number of respects. First, initiation of deposition was easiest on these materials. In all the steels, the level of deformation of the substrate was low (indicating that deformation was localized in the aluminum particle); no substrate deformation was discernable on the quenched or quenched and tempered steels. As deposition initiation was less difficult with these substrates, the high density of very small particles bonded to the surface (as seen for the softer metals) was not observed. In contrast, frag-



(a)





Fig. 8 SEM-SE images at different magnifications of the surface of a glass substrate following cold spray deposition of aluminum

ments of an iron-base material have been observed on the surface of the steel. It is most likely that this material is associated with the impact of the aluminum particles onto the substrate; if it were associated with interactions of the aluminum particles with the steel nozzle, then it would be expected that such particles would be found on all substrates. The material fragments are iron base and are formed as part of the metal-jetting phenomenon, akin to that observed in explosive welding. The direct observation of the product of metal jetting indicates how initiation of bonding can be so rapid for the steels when there is so little deformation of the substrate and indicates that this is indeed a metallurgical bond. Indeed, it is the hardness of the steels that localizes deformation in the aluminum particle and promotes the formation of the metal jet.

4.3 Interaction with Nonmetallic Materials

Three nonmetallic substrate materials have been examined in this work as potential substrate materials. Figure 3 shows that the deposit cross-sectional area was low in all cases, across a range of hardness. This indicates that deposit initiation on these substrates is not easy. Figure 5 shows the surface of ABS following cold spraying. Here, due to the low hardness of the polymer, all the damage has occurred in the polymer, and thus the deformation of the aluminum particles necessary for bonding has not taken place. Figure 8 shows the surface of glass following deposition of aluminum. Here, although the hardness of the substrate is in the same range as that of the steels, no fine particle deposition was observed (since metal jetting has not taken place). Also, significant indentation damage of the glass has occurred, leading to bonding of some particles, probably by a mechanical bonding mechanism. Similar behavior was seen with a polished alumina substrate. Thus, although glass and alumina restrict most of the deformation to the aluminum particles, initiation of bonding is difficult. It is proposed that this is due to the inability to form a metallic bond with the substrate in these cases (in contrast to that formed when depositing onto steels).

5. Summary

Aluminum powder has been deposited by cold spraying onto a range of substrate materials. The ease of initiation of coating deposition (associated with bonding between the particles and substrate) has been estimated by examining the thickness of material built up following a single pass of the nozzle.

It has been shown that initiation of deposition onto soft metallic substrates is difficult due to the lack of deformation of the aluminum particles. In fact, deposition onto a tin substrate resulted in melting of the tin. Ease of initiation of aluminum deposition increases as the hardness of metallic substrates increases. Clear evidence of metal jetting (akin to that seen in explosive welding) was observed following deposition onto steel substrates, where the most rapid initiation was observed. Deposition onto aluminum was observed to be difficult, due to the tenacious nature of the oxide film on the substrate, which prevents metal-metal contact. Deposition onto nonmetallic substrates (ranging from polymers to ceramics) was difficult due to the lack of metallic bond that can be formed between the aluminum particles and the substrate in these cases.

It may be concluded that the most successful initiation of bonding of aluminum particles onto substrates of low roughness by cold spraying requires a metallic surface with a hardness higher than that of the particles. In these cases, bonding results from severe deformation under high pressure, akin to that observed in explosive welding. A metal jet is formed, which results in the contact of clean metal surfaces and the formation of a metallurgical bond. Bonding does not require deformation of the substrate, but is hindered by tenacious oxide layers on the substrate, which prevent the contact of nascent metal surfaces. Bonding onto nonmetals is hindered by the impossibility of forming a metallurgical bond.

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