JOURNAL OF MATERIALS SCIENCE 29 (1994) 1051–1055

Processing and characterization of glass-filled polyamide composite coatings

Y. BAO, D. T. GAWNE
Department of Materials Technology, Brunel, The University of West London, Uxbridge, UK

Glass–polyamide composite coatings have been successfully plasma sprayed on to steel by simultaneous injection of glass and polyamide particles into the plasma jet. The deposition conditions were selected such that the polyamide melts in the jet in order to provide the matrix, whereas the glass remains solid to avoid overheating the polymer. The deposition efficiency of glass is significantly less than that of polyamide, particularly in the initial stages of spraying, because the solid glass particles deform elastically on impact with the substrate and rebound, whereas the kinetic energy of the molten polyamide particles is absorbed by viscous flow. The presence of the glass filler reduces the wear rate of polyamide by an order of magnitude because of its load-supporting action and reduction of adhesive and abrasive wear. However, there is a minimum in wear rate at a glass filler content of 50 wt%, because higher glass contents adversely affect wear performance by reducing the fracture toughness. The results also show that refining the glass particle size provides a considerable reduction in wear rate of the composite. The first-order theory of adhesive and abrasive wear, in which the wear rate is inversely related to hardness, is shown not to hold for this class of materials.

1. Introduction

Previous work [1] has shown that polyamide 11 coatings can be successfully plasma sprayed providing that the precursor powder particles are completely melted in the plasma while avoiding degradation. Polymers have limited wear resistance and a successful way of overcoming this problem in bulk polymers has been by the use of fillers. In principle, plasma spraying should be well suited to the incorporation of fillers into a polymer matrix, because two or more types of material can be readily injected into the plasma jet. However, little work has been reported on the plasma spraying of polymers and less on polymer composites. The purpose of this work was to investigate the plasma-spray deposition of glass-filled polyamide coatings on steel, particularly the influence of glass content and glass particle size on process efficiency and deposit properties.

2. Experimental procedure

The precursor powder used was polyamide 11 (Rilsan ES grade) supplied by Atochem UK Ltd. The glass filler employed was Spheriglass obtained from Poller-Ballotini Ltd, in three spherical particle sizes: 0–20, 50–100 and 100–200 μm. The polyamide and glass powders were thoroughly mixed in various proportions to give eight precursor powders with glass contents in the range 0–89 wt%. The substrate was a plain carbon steel (080M40 grade) plate of thickness 6 mm, which was degreased, grit blasted with alumina grit (Metcolite C, Metco Ltd.) under a blast pressure of 4 bar and a blast distance of 150 mm to give a surface roughness of 7 μm R_a.

Plasma spraying was undertaken using a Metco plasma spray system with an MBN torch, MCN control unit, 4MP powder feed unit and fluidized hopper. Wear performance was assessed using a reciprocating ball-on-flat machine with a stainless steel ball of diameter 12.7 mm under a load of 40 N.

3. Results and discussion

3.1. Deposition

The glass–polyamide coatings were deposited using the optimal arc power of 20–23 kW and torch traverse speed of 150 mm s^{-1} previously determined for pure polyamide coatings [1]. A plasma gas of argon with 5% hydrogen was used together with a precursor carrier gas of argon. The precursor powder was injected downstream at 45° to the jet longitudinal axis. Fig. 1 gives the cross-section and top surface of a glass-filled polyamide coating, and shows that dense, quality deposits can be plasma sprayed.

Table I presents the compositions of the precursor powders and the corresponding deposits. The glass content of the deposits was determined by ashing the coating in an oven at 600 °C for 2 h and weighing the glass residue. A similar sample of pure glass was subjected to the same oven treatment to account for any mass change in the glass. The data in Table I show that the glass contents of the coatings are substantially lower than that of the precursor powder, particularly for the coarse glass powders, which indicates that significant amounts of glass are lost during deposition.

The above effect may be quantified in terms of relative deposition efficiency, which is the ratio of the filler content in the deposit to that in the precursor

TABLE I Compositions of precursors A–H and coatings

	A	B	C	D	E	F	G	H
Glass particle size (µm)	0	0–20	0–20	0–20	0–20	0–20	50–100	100–200
Precursor glass content (wt%)	0	27	50	60	71	89	70	70
Deposit glass content (wt %)	0	22	36	38	48	66	26	24

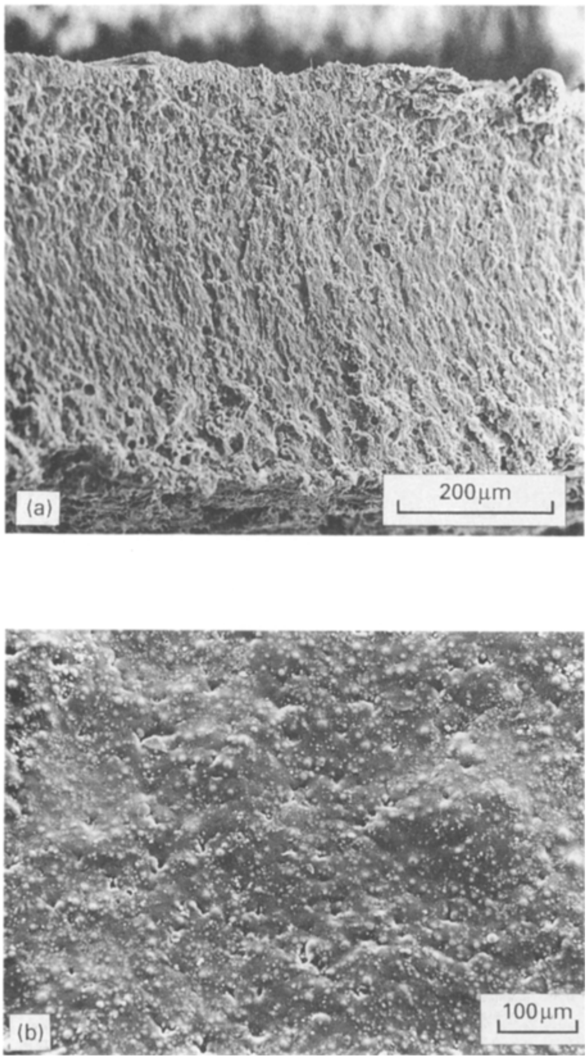

Figure 1 Scanning electron micrographs of glass–polyamide coating: (a) through-thickness section, and (b) top surface.

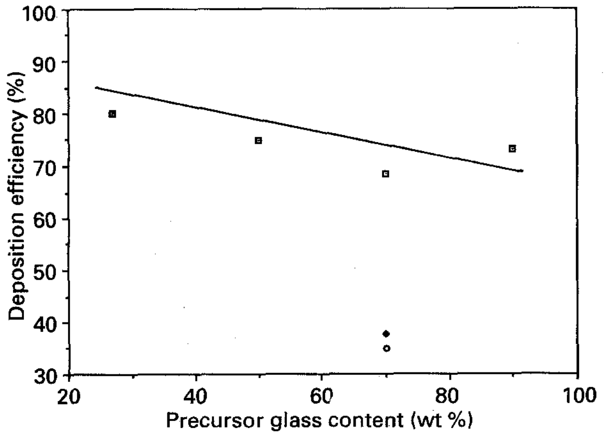

Figure 2 Effect of glass content and glass size on relative deposition efficiency of glass. (□) 0–20 µm, (◆) 50–100 µm, (○) 100–200 µm.

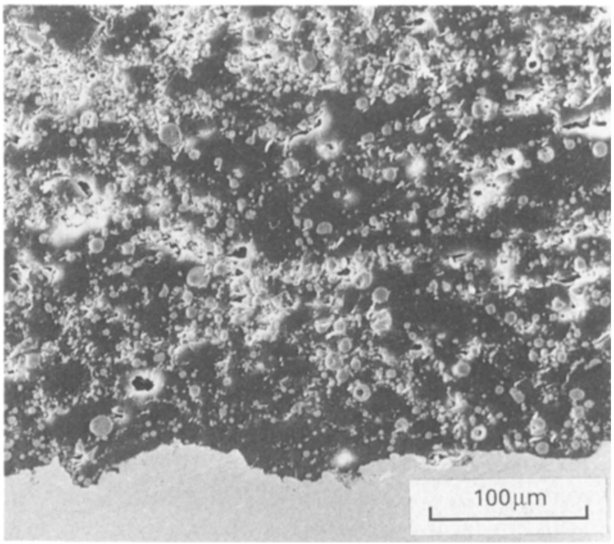

Figure 3 Polished cross-section of glass–polyamide coating with a glass particle size of 0–20 µm; interface left to right, steel substrate at the bottom.

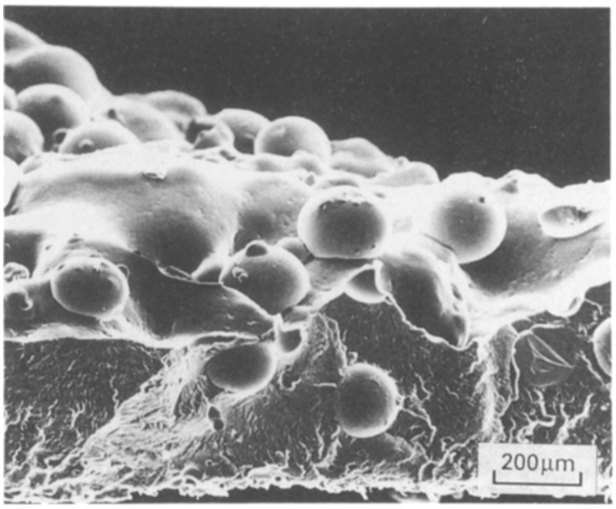

Figure 4 Fracture surface of a cross-section of glass–polyamide coating with glass particle size 100–200 µm.

(the ratio is relative to that of polyamide and is not an absolute value). Fig. 2 shows that the relative deposition efficiency of the small glass particles is approximately twice as high as that of the large particles.

Figs 3 and 4 show cross-sections through the coatings prepared with the 0–20 µm glass particles and the 100–200 µm particles, respectively. The glass particles have remained spherical implying that they did not melt in the plasma, which is to be expected because the operating conditions were designed to melt the polyamide particles rather than the glass. As a result, the glass particles are solid when they impact the substrate. Figs 3 and 4 show that the layer of coating immediately at the substrate interface has a relatively low glass content. This is particularly evident for the coarse glass powder used in the deposit shown in Fig. 4, where the particles are in the outer layers of the deposit.

During the first pass of the plasma torch, the solid glass particles will rebound off the exposed steel surface because much of their kinetic energy is recovered from the reversible elastic deformation of the particles themselves and the steel. On the other hand, the polyamide particles do melt in the plasma jet and their kinetic energy is expended in viscous flow to form splats on impact with the substrate. As the polyamide coating builds up, the glass particles impact on soft polyamide and become embedded in the coating: their kinetic energy is absorbed by plastic deformation of the polyamide at a temperature sufficient to promote particle bonding with the matrix. However, the large particles have correspondingly large kinetic energies and their impact zones can extend deep into the deposit and, in some cases, down to the steel substrate. The kinetic energy of these large particles cannot be completely absorbed by plastic deformation of the polyamide and so elastic deformation of the glass particles, the solidified underlying polyamide layers and possibly the steel takes place, which will result in the rebound of some particles and a loss in deposition efficiency. The effect is responsible for the relative denudation of glass in the polyamide next to the interface and the low deposition efficiency at large glass particle sizes.

3.2. Properties

Fig. 5 shows the wear data for the pure polyamide and glass-filled polyamide deposits. The coatings initially exhibit a high wear rate or running-in before the development of a milder equilibrium wear stage. The running-in stage is due to initial misalignments between mating surfaces and the presence of high spots in the surface profile, which lead to high local pressures and contact temperatures. In the case of the composites, the filler particles are not fully encapsulated by the matrix at the surface and the more loosely bound particles will be rapidly removed, thereby increasing the wear rate.

The data in Fig. 5 are replotted in Fig. 6 to show the influence of glass content on wear rate. The presence of the glass filler reduces the wear rate by almost an order of magnitude. There is a minimum in the wear rate at a glass content of approximately 50 wt %. Fig. 7 shows that the glass filler produces a substantial increase in hardness of the deposits but there is no optimum value as there is for wear resistance. The simple theory of wear [3], in which the wear rate is inversely proportional to hardness, does not therefore apply to this class of materials.

Fig. 8 shows electron micrographs from the wear tracks of pure polyamide, 38 wt % glass–polyamide and 66 wt % glass–polyamide coatings. The pure polyamide (Fig. 8a) shows grooves parallel to the sliding direction, indicative of abrasive wear and evidence of a polyamide transfer film on the steel counterface, suggesting adhesive wear. The debris particles collected during the wear test included flat platelets of similar dimensions to the splats making up the deposit, which suggests that fracture occurs along the splat boundaries. The wear track also gave evidence of failure at splat boundaries, particularly at the edges of the abrasive grooves where the tensile component of the stress system is larger than that in the centre of the groove (Fig. 8a).

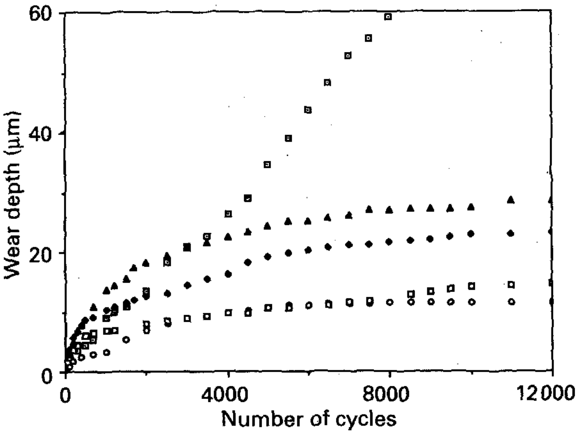

Figure 5 Effect of glass content on the wear of glass–polyamide coating: (⊡) 0 wt %, (△) 22 wt %, (☐) 38 wt %, (○) 48 wt %, (◆) 66 wt %.

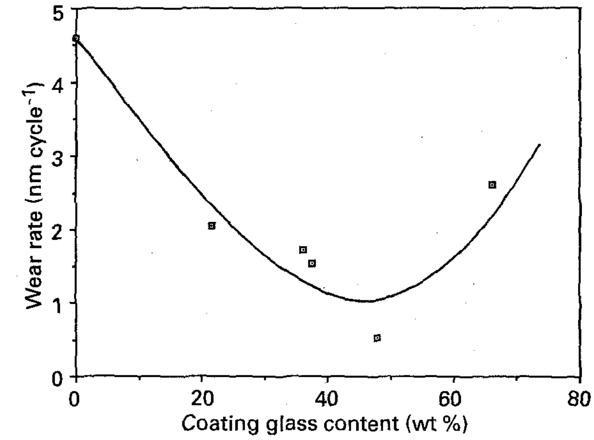

Figure 6 Effect of glass content on equilibrium wear rate of glass–polyamide coatings.

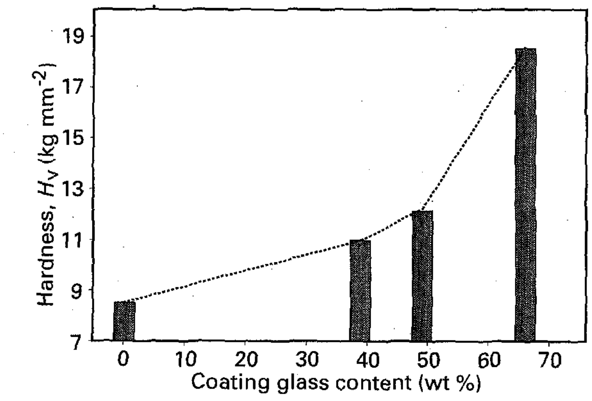

Figure 7 Effect of glass content on the hardness of glass–polyamide coatings.

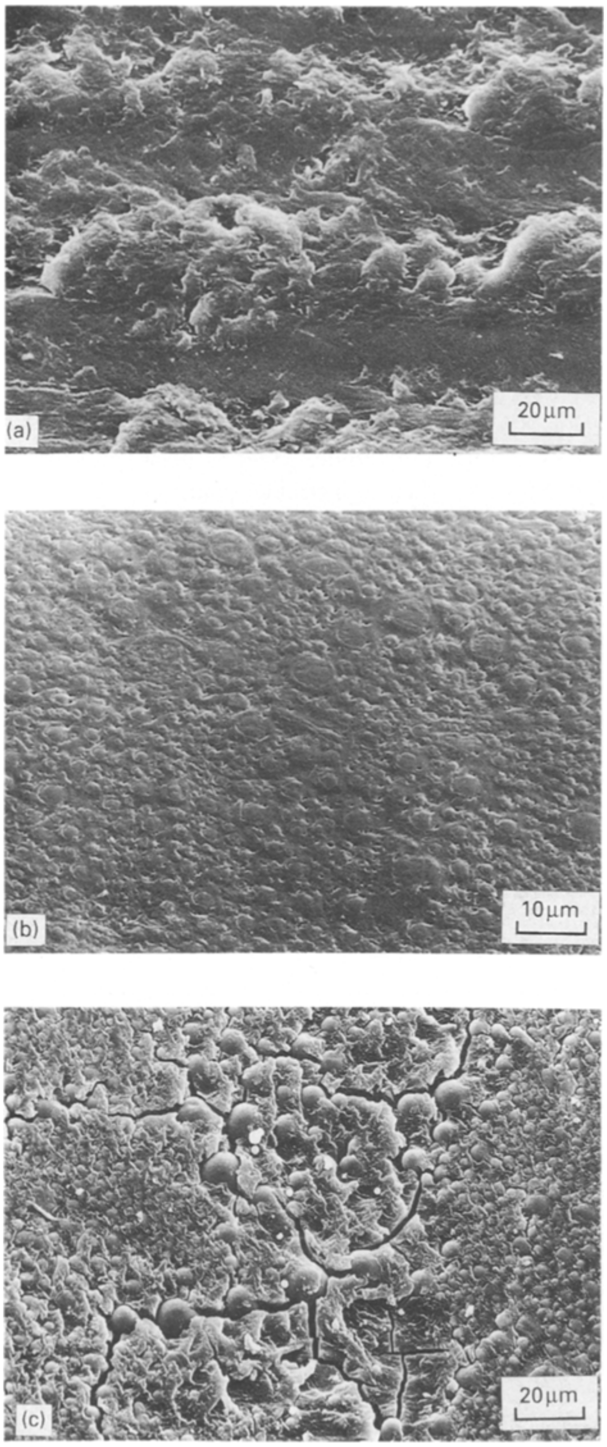

Figure 8 Scanning electron micrographs of wear tracks on glass–polyamide coatings with glass content of (a) 0 wt %, (b) 38 wt % and (c) 66 wt %.

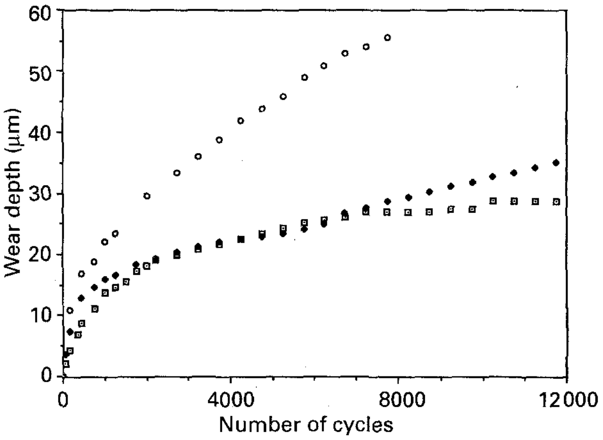

Figure 9 Effect of glass particle size on the wear of glass–polyamide coatings. (□) 1–20 μm, 22% glass; (◆) 50–100 μm, 26% glass; (○) 100–200μm, 24% glass.

The low-wear composite coating, as shown in Fig. 8b, exhibited a smooth, worn surface with little tearing. The glass particles are clearly visible and are worn to a flat profile, which suggests they produce an effective load-supporting action. Lancaster [2] has attributed the improved wear resistance of bulk fibre composites to this mechanism and although the magnitude of the effect is expected to be less with spheroidal particles, it is still expected to be an important factor in the improved wear resistance of the composite coatings.

The high-wear composite coating, as in Fig. 8c, shows more brittle behaviour. Fracture appears to initiate by decohesion at the glass–polyamide interface and propagate along lines of glass particles, particularly in regions of high filler density. The glass particles at the sliding surface are not worn flat, as they are in Fig. 8b, which suggests a less effective load-supporting action in this material as a result of the more rapid removal of material by interparticle fracture. The susceptibility of high particle density regions to this rapid material-removal mechanism implies the importance of achieving a uniform filler dispersion and particle mixing in precursor preparation.

Fig. 9 shows the influence of filler particle size for deposits containing 21–26 wt % glass. The wear rate decreases with particle size with the best wear resistance being shown for the 0–20 μm particle composite even though this possessed the lowest glass content. The most prominent effect is the high wear rate exhibited by the 100–200 μm particle composite. Comparison of Figs 3 and 4 shows that, whereas the fine glass is uniformly dispersed throughout the coating thickness, the coarse glass is concentrated at the outer layers. In particular, many large glass particles are at the surface where they have not been fully encapsulated by the polyamide and are therefore loosely bound. These particles will be easily removed during sliding and the depleted matrix will exhibit a high wear rate.

Figs 6 and 7 indicate that the wear rate is not inversely related to the hardness as expected from simple wear theory [3]. For example, the hardest coating has the highest wear rate rather than the lowest. The simple theory of adhesive and abrasive wear relies on plastic deformation, which is inversely related to hardness and this provides the basis of the wear equation [3]. However, fracture plays a prominent role at high hardnesses, as is demonstrated by comparing Fig. 8c with Fig. 8b. The wear rate is inversely related to fracture toughness [4] which, in turn, tends to be inversely related to hardness. The inverse relationship between wear rate and hardness, therefore, does not hold at high hardnesses.

4. Conclusions

1. Glass–polyamide composite coatings can be successfully plasma sprayed on to steel by control of the

precursor injection, arc power and torch traverse speed.

2. The deposition efficiency of the glass is significantly less than that of the polyamide, particularly for coarse precursor powders owing to particle rebound from the substrate. This effect also leads to a reduction in the filler content of the coating in the layers next to the interface.

3. The presence of the glass filler reduces the wear rate of the polyamide coatings by a factor of 10 owing to its load-supporting action.

4. There is a minimum in wear rate at approximately 50 wt % glass content.

5. Glass contents above 50 wt % increase the hardness of the composite but reduce the wear rate because of a decrease in fracture toughness due to particle decohesion.

6. The wear rate increases with increasing particle size because the large particles are concentrated in the outer layers and are ineffectively bonded to the polyamide.

Acknowledgements

This work has been carried out with the support of the Procurement Executive, Ministry of Defence, the Science and Engineering Research Council, Metco Ltd and ICI plc. The authors thank the above for permission to publish the paper.

References

1. Y. BAO and D. T. GAWNE, *Trans. Inst. Metal Finish.* **69** (1991) 80.
2. J. K. LANCASTER, *J. Phys.* **D1** (1968) 549.
3. J. F. ARCHARD, *J. Appl. Phys.* **24** (1953) 981.
4. A. G. EVANS and D. E. MARSHALL, in "Fundamentals of Friction and Wear of Materials", edited by D. A. Rigney (American Society for Metals, Metals Park, OH, 1981) pp. 439–52.

Received 4 May
and accepted 20 August 1993