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Microporosity and delamination mechanisms in thermally sprayed borosilicate glass coatings

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ABSTRACT

Conventional enamelling requires both the feedstock powder and the substrate component to be heated one or more times in a furnace at 800–900 °C. The process can degrade the substrate and limit the size of the component to the furnace dimensions, which are serious restrictions on the technology. This study concerns the use of combustion-flame spraying as an alternative technique for enamelling. In this process, the heat source (the flame) is separated from the substrate, which enables much lower substrate temperatures and avoids thermal damage. It also removes the need for furnace treatment and opens up the possibility of on-site enamelling and repair. However, experimental trials showed that thick flame-spray coatings delaminated during cooling and had high microporosities due to quenching stresses at the glass-steel interface and inadequate splat flow of small feedstock particles. The research shows that these adverse mechanisms could be overcome by pre-heating the substrate surface to the dilatometric softening temperature and removing fines from the feedstock powder. The control of these two parameters was found to double the adhesion strength, provide coatings of very similar hardness and fracture toughness to conventional enamel as well as deposit coatings of over 1 mm in thickness for heavy-duty corrosion protection. Thermal spraying is well established for ceramic and metal coatings but the fundamentally different structure of glasses requires a different approach. An advantage of combustion flame spraying shown up by this research is that the high energy of the flame accelerates the particles to a high velocity and the resulting impact forces promote the flow of the glass. As a result, lower substrate temperatures may be used with reduced risk of degradation or higher viscosity glasses may be deposited with enhanced properties. The influence of the type of thermal-spray technique on coating quality is also discussed.

1. Introduction

Thick glass-based coatings (1–2 mm) are used for the heavy-duty corrosion and chemical protection of steel components, such as tanks, pipes and mixers in processing plant for the manufacture of chemical and pharmaceutical products. Processing of these coatings follows conventional enamelling procedure by applying glass particles on the metal component and then heating in a high-temperature furnace to fuse the glass particles into a dense and well-bonded coating [1–3]. In practice, the glass and metal substrate are heated together to the same temperature in the furnace, usually 800–900 °C. Degradation and distortion of the substrate at this temperature needs to be avoided, which limits the choice of material, while the component to be coated clearly needs to fit in the furnace, which limits its size. These limitations restrict the application of glass coatings.

This study investigates the use of thermal spraying as a possible means of overcoming these difficulties. Thermal spraying refers to a generic family of processes consisting of injecting powdered materials into a hot, high-velocity gas jet in which they are heated, accelerated and projected onto a substrate to form a coating. The advantage of thermal spraying is that the gas jet acts as the heat source and is separated from the substrate. This enables the glass particles to be fused in the jet, while the metal substrate is kept at a relatively low temperature. It is a one-stage process, no furnace is required and therefore, in principle, there is no size limit on the substrate [4–8]. In addition, removing the need for a furnace provides the possibility of on-site enamelling and repair.

The work described in this paper is aimed at understanding the underlying mechanisms and producing crack-free, adherent borosilicate glass coatings with a thickness of 1000–1200 μm on an alloy steel by combustion-flame spraying. This thickness is required for the corrosion and chemical protection of steel articles in demanding applications. In practice, the requirement of such a high thickness can cause difficulties in manufacture due to cracking and delamination.

Thermal spraying is well-established for the deposition of ceramic and metal coatings [9]. However, these crystalline materials have fundamentally different structures from glasses and this paper shows that a different approach is needed for the deposition of glass coatings. The results obtained on splat flow behaviour, microporosity and the development of mechanical properties are of scientific as well as technological significance.

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Table 1
Thermal properties of Glasses P and G.

Glass	Softening temperature, T_{soft}/°C	Glass transition temperature, Tg/°C	Thermal expansion coefficient (20-400 °C), α/K^{-1}
Glass P1	524	480	8.9×10^{-6}
Glass G	540	516	9.7×10^{-6}
Steel			13.6×10^{-6}

2. Experimental details

Two borosilicate glasses (G and P1) were used in this investigation. They were conventional borosilicate glasses used for enamelling steel as a means of conferring chemical resistance. Glass G was a groundcoat or primer, which was applied directly on the steel substrate. This glass acted as a bond coat between the steel substrate and the topcoat with an intermediate expansion coefficient. Glass P1 was a topcoat or covercoat, which was applied to Glass G for promoting chemical resistance. The powders and coatings were amorphous. The powders were provided by Pfaudler Werke GmbH. Table 1 gives the thermal properties of the glasses used as determined by dilatometry. Glass P1 was sieved to give two batches of powders designated as P2 and P3. There were thus three powders to investigate: P1 (the original as-received powder) together with the more refined P2 and P3 powders.

Combustion-flame spraying was used (Oerlikon-Metco 6P-II spray gun) as a thermal-spray technique in which the hot jet is formed by the combustion of an acetylene-oxygen gas mixture. The substrates were low-alloy steel discs of 105 mm in diameter and 2.5 mm in thickness, which were degreased and grit-blasted by using alumina grit with a pressure-operated machine to give a surface roughness (Ra) of 5–6 μm immediately before spraying. The surface roughness was measured using a Taylor Hobson instrument. Acetylene was used as the fuel gas with a flow rate of the acetylene ranging from 45 to 55 SLPM (standard litres per minute), oxygen as oxidant and compressed air as the powder-carrier gas. A spray distance between the nozzle exit and substrate surface of 250 mm was used.

The thermal history of the glasses during spraying affects the final structure and quality of the coating, and so the temperatures of the coating and substrate during processing were recorded in-situ. The temperature at the coating surface (T_s) was measured using an infrared thermometer (MX4 CF Infrared Thermometer, Raytek, UK), which has a response time of 0.1 s and data collection set at 0.125 s. The temperature at glass-steel interface (T_i) was measured using Type K thermocouples that were attached to the interface before deposition.

The thickness of coatings was measured by PosiTest DFT coating thickness gauge and subsequently confirmed by microstructural observations on the coating cross sections.

The adhesion of the glass coating to the steel substrate was evaluated using a pull-off tensile test, which conforms to ASTM D4541 and BS 3900-E10. Testing was carried out with an Avery universal testing machine (Birmingham, UK) at a crosshead speed of 5 mm/min, which measures the tensile force required to detach the coating from the substrate. Three types of failure pattern were recorded during testing. An adhesion failure refers to the fracture at the coating-substrate interface representing the bond strength of the coating to substrate, a cohesive failure by fracture within the coating itself and an adhesive failure by fracture within adhesive or at the adhesive-coating interface. Five specimens from the each powder coating were tested.

The microhardness of the coating was measured on the through-thickness cross-sections of the coatings with a Vickers microhardness tester (Struers Duramin-5, UK) under an indentation load of 1 to 5 N for a fixed loading time of 15 s.

The fracture toughness of the glass coating was estimated using the Vickers indentation test in which indentations were made on polished cross sections of the coatings under an applied load of 5 N.

The microstructure of cross sections of the feedstock powders and coatings were examined using scanning electron microscopy (S-4300SE, Hitachi Co. Ltd) with energy dispersive spectrometer (Oxford instruments, UK). The coating specimens were cold mounted using epoxy resin and prepared by sequential grinding with 120–2500 mesh grade SiC abrasive papers. Final polishing was carried out using 1 μm and 0.25 μm diamond pastes.

Image analysis software (ImageJ) and 200× back-scattered SEM images were used to perform porosity measurements. At least seven images were examined for each type of coatings. The particle size and size distribution of each powder were measured by Laser Diffraction Particle Size Analyzer (LS 200, Beckman Coulter).

3. Results and discussion

3.1. Delamination of coatings

The combustion-flame spray deposition conditions were directed at ensuring the glass particles were sufficiently molten but not degraded in the flame. To produce a double-layered structure, 150–200 μm of Glass G was at first deposited directly on the steel substrate that had been pre-heated to 300 °C immediately prior to deposition to promote bonding of the glass to the steel. This pre-treatment was accomplished by scanning the flame torch over the surface of the substrate. 800 μm of Glass P1 was then immediately deposited on Glass G as a topcoat in order to form the required double-layered coating system. The thickness of Glass G and Glass P were controlled by spraying passes during

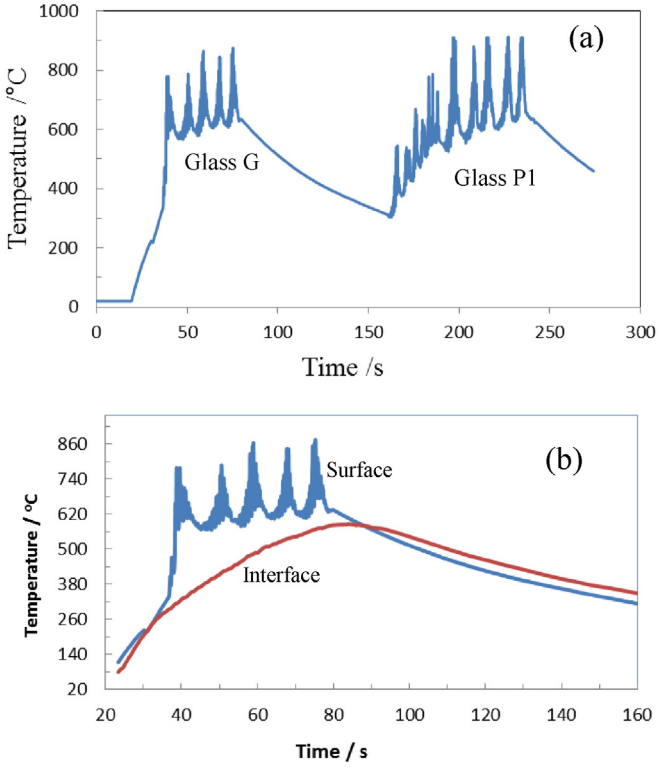

Fig. 1. Measured thermal histories of coatings and interface during thermal spraying. (a) Temperature trace of the surface of Glass G coating and the coating-substrate interface; (b) temperature trace of Glass G coating surface followed by that of Glass P1 coating surface.