



Ceramic particulate thermal barrier surface coatings for glass fibre-reinforced epoxy composites



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ABSTRACT

This study investigates the thermal barrier efficiency of five commercially available ceramic nano and micro particles deposited on the surfaces of glass fibre-reinforced epoxy composites (GRE). Two approaches of application of deposition of ceramic particles have been undertaken, firstly where the ceramic particles were dispersed in a phenolic resin binder and applied on a GRE surface by a K-bar coater and the second where extra ceramic particles were sprayed on the first coating while the resin was partially cured to enable the surface to be completely covered by ceramic particles, leaving no resin exposed. The thermal barrier efficiency of these coatings was evaluated from the cone calorimetric parameters at incident heat fluxes of 35 and 50 kW/m² as well as from temperature gradient through the samples' thicknesses, measured by inserting thermocouples on the exposed and back surfaces during the cone tests. The morphology and durability of the coatings to water absorption, peeling, impact and flexural loading were also studied. The results showed that the surface layers of all coated samples were uniform and there was a strong adhesion between the coating and the substrate. Moreover, they did not adversely affect the mechanical properties of GRE composite while improving the mechanical property retention of GRE composites after exposure to heat.

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1. Introduction

Fibre-reinforced polymer (FRP) composites produced by embedding structural fibres (of high strength and stiffness) in thermoset polymers (resins), are becoming popular alternatives to metals due to their low cost, lightweight, resistance to environmental variants and excellent mechanical properties. Unfortunately these composites cannot compete with metals for resistance to heat and fire because of the organic nature of the matrix [1]. Their response to heat depends upon the type of the resin and intensity of the heat source. At temperatures below the glass transition temperature, the resin matrix system is unaffected by heat and there is little effect on mechanical properties. On reaching the glass transition temperature of the resin (temperature (150–220 °C, depending upon the resin type), the resin softens and the composite laminate starts losing its mechanical properties, which can be approaching 50% of the original value [2,3]. On reaching the decomposition temperature of the resin and eventual ignition of the resin there is a complete loss of mechanical integrity of the composite [1–7]. The most effective technique of protecting these materials against heat and fire is the use of surface coatings,

which can inhibit or reduce the heat transfer from the fire/heat source to the underlying structure [6,8–10]. The coatings can be of polymeric nature where flame retardant additives, usually intumescent systems, are dispersed in a resin binder [8–10] or ceramic and glass fibrous mats, sometimes containing exfoliated graphite [11,12]. These conventional coatings however, have certain disadvantages for instance, the intumescent coating requires a thick coating layer (>1 mm coating thickness) [13], which may not be applicable in some applications. Thermal insulative mat/sheets, such as intumescent mats, may add to weight to the composite [1,11,12] and could absorb fuel in case of an accident [1].

Ceramic particles of low thermal conductivity such as yttria stabilised zirconia are commonly used to protect metallic surfaces for excessive heat cycles [14]. These particles are applied on metal surfaces using techniques such as electroplating [15], electroless-plating (e.g. hot dipping, metal spraying) [16], electrogalvanising, PVD (physical vapour deposition), CVD (chemical vapour deposition) [17] and plasma spraying [18]. These are all high temperature processes and are suitable for substrates that can sustain temperature higher than 600 °C, such as metallic substrates. The application of these coatings on polymer substrates is extremely limited due to the low thermal stability of organic resins, which require processing at temperatures below glass transition (T_g) and decomposition temperatures of the polymer component.

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A polymeric coating formulation consists of a polymeric binder, a filler (or particle), a solvent/carrier, pigments, and additives [19]. The binder material provides a matrix in which all other constituents are dissolved/dispersed. Most binders are thermoset type polymers because of their viscosity and reactive structure [20]. In this work both of the above mentioned approaches have been combined into one coating type, i.e., the ceramic particles are dispersed in a phenolic resin and applied on the surface of a composite. In our recently published work we have explored applying three types of ceramic particulate (yttria doped zirconia, a low melting silicate glass and alumina tintannate) coatings using an epoxy resin binder [21]. While the thermal barrier efficiency of these coatings was evident, some of the coated samples ignited when the surface was not completely covered by ceramic particles, leaving gaps where the resin was exposed to the surface from where heat could penetrate through the surface. This work takes this concept forward. Here the epoxy resin binder has been replaced by an inherently flame retardant phenolic resin. In addition to the three types of ceramic particles previously studied, two new particles namely nanoclay and nanosilica have also been investigated. Two sets of samples were prepared, one as previously [21] but with a different binder (phenolic replacing epoxy) and the second where ceramic particles were sprayed on the coating while it was not fully cured so that the surface was completely covered by ceramic particles and no resin is exposed as was the case in the previous study. Apart from thermal barrier effect of these coatings, the resistance to peeling, water, impact, flexural tension etc have also been studied in this work.

2. Experimental details

2.1. Materials

2.1.1. Glass fibre-reinforced epoxy (GRE) composite

Epoxy resin system: epoxy phenol novolac resin (Araldite LY5052, Huntsman) and cycloaliphatic polyamine-2,2-dimethyl-4,4-methylene bis cyclohexylamine hardener (Aradur HY 5052, Huntsman).

Glass fibre: Woven roving glass fibre of E-glass type (300 g/m², Glasplies).

2.1.2. Ceramic micro-particles for surface coatings

Nanoclay (30B): Cloisite 30B (Southern Clay Products), a commercially available alkyl quaternary ammonium modified montmorillonite clay.

Fumed nanosilica (Si): Aerosol 200 (Evonik Industries), a high purity amorphous anhydrous colloidal silicon dioxide with a specific surface area of 200 m²/g.

Glass flake (Flek): Flekashield (NGF Europe, UK), platelets of E-glass of ~5 µm thickness and 10–4000 µm width.

Aluminium titanate (Re): Recoxit (Ohcera. Co., Ltd., Japan), a ceramic powder composed of aluminium titanate (Al₂TiO₅).

Zirconia (Zr): Aqueous dispersion of yttria doped zirconia, consists of 91–93% zirconium oxide and 7–9% yttrium oxide (XZO1357, Mel Chemical, UK). The particles were obtained by evaporating water at 60 °C in an oven for 24 h. The residue was grinded with mortar and pestle.

2.1.3. Binder

Phenolic resin: DUREZ 33156 (Sumitomo Bakelite Europe) a modified liquid phenolic resin containing phenol (polymer with formaldehyde) (58–78 wt%), ethanol (20–29 wt%) and water (3 wt%).

2.2. Sample preparation

2.2.1. Glass fibre reinforced epoxy (GRE) composite laminate

Eight pieces of 300 mm × 300 mm woven E-glass fabric were used for composite laminate preparation, with the ratio of 50 wt% glass fibre and 50 wt% resin matrix. The GRE composite laminate was fabricated using a hand lay-up method by impregnating each glass fabric layer with the resin, vacuum bagging and curing at room temperature for at 24 h, and then post-curing at 80 °C for 6 h.

2.2.2. Ceramic particle coatings using a phenolic resin as binder

Five types of ceramic particles: nanoclay (30B), nanosilica (Si), Flekashield (Flek), Recoxit (Re) and zirconia (Zr) were used to prepare coatings of approximately 100–650 µm thicknesses. The ceramic particle and phenolic resin ratio for each coating was different depending upon the type of each ceramic particle as presented in Table 1. The ceramic to resin ratio of each ceramic coating type was established based upon the maximum amount of particle component that can be added into the phenolic binder. It was not possible to increase the level of ceramic particles in the coatings higher than those given in Table 1 without adversely affecting processability of the coating, which becomes too viscous and restricts uniform application on the GRE composite laminate. Two sets of coated GRE composite laminates were prepared: (a) ceramic particle with resin binder and (b) ceramic particle coatings with an additional particle layer on the surface of (a).

2.2.2.1. Ceramic coatings on GRE composite using a phenolic binder.

These ceramic particle coatings were prepared by dispersing the ceramic powders in a phenolic resin binder using the proportions given in Table 1. Firstly, the ceramic particle and a phenolic resin were dispersed in ethanol (10 wt% w.r.t. mixture of ceramic particles and phenolic resin). The suspension was stirred with a mechanical stirrer for 15 min. A 200 µm spirally wound K-bar (R.K Print-Coat Instruments Ltd) was used to apply the coating formulation of 30B, Si, Flek, Re and Zr on the GRE composite laminates (size 75 mm × 150 mm). The coated laminates were then cured at room temperature for 12 h and further post-cured at 80 °C for 24 h. This coating procedure is presented in Fig. 1(a). These samples are identified in this manuscript as GRE-P/CP (P = phenolic, CP = ceramic particle, e.g., 30B, Si etc). Similarly, a sample with only phenolic resin coated on the GRE laminate was also prepared in order to compare the effect of the binder on these ceramic particle coatings. This sample is identified in this manuscript as GRE-P.

All samples were weighed before and after coating application and the wt% ceramic deposited on the surface was calculated as:

$$\text{Ceramic particle deposition (wt\%)} = \frac{W_{\text{Ceramic}} - W_0}{W_0} \times 100 \quad (1)$$

where W_0 is weight of the virgin laminate and W_{Ceramic} is weight of ceramic particle in the coated samples, which is calculated from the ratio between ceramic particle and resin binder. For instance, in case of the zirconia coating, $W_{\text{Ceramic}} = 0.7 \times \text{total weight of coating}$ (30 wt% of phenolic binder in the coating).

Table 1

The ceramic particle: phenolic resin ratio of different coatings on laminate specimens.

| Sample | Ceramic type in the coating | Ceramic: phenolic resin mass ratio |
|------------|-----------------------------|------------------------------------|
| GRE-P/30B | Nanoclay | 25:75 |
| GRE-P/Si | Nanosilica | 10:90 |
| GRE-P/Flek | Flekashield | 20:80 |
| GRE-P/Re | Recoxit | 70:30 |
| GRE-P/Zr | Zirconia | 70:30 |

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