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Surface preparation of silicon carbide for improved adhesive bond strength in armour applications☆

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Abstract

Surface treatments of silicon carbide have been investigated with the aim of improving the strength of the bond between the ceramic and an epoxy adhesive. Three surface conditions have been characterised; as-fired, air re-fired and KrF laser processed. A number of characterisation techniques have been used to determine the morphological and chemical changes that have occurred to the surface. Scanning electron microscopy of the re-fired and laser processed samples showed surfaces that appeared glassy, with the laser processed surface showing a different morphology. X-ray photoelectron spectroscopy indicated both treatments had oxidised the surface and the laser processed surface also had a greater concentration of hydroxyl groups. The wettability of both surfaces had improved and the laser processed surface was found to be highly hydrophilic. Mechanical testing of joints prepared with this technique showed them to have the highest strength in tension, with the locus of failure being cohesive.

Keywords: Armour; Silicon carbide; Adhesive bonding; Mechanical testing; Spectroscopy

1. Introduction

Ceramic based armour systems feature increasingly on modern armoured vehicles. The main benefit of these systems is that they combine a high resistance to penetration with a low density. These features are ideal for armour. However, a major disadvantage of these systems is the reduced multi-hit performance compared with metallic based armour.

Ceramic armour typically comprises a ceramic front strike face, which is adhesively bonded to a composite or metal backing. A ballistic impact leads to a compressive stress wave being transmitted through the ceramic to the back face of the ceramic. On reaching the free surface, the wave is reflected, leading to a tensile stress in the ceramic, which is the main cause of the failure of the ceramic by fracture. The intensity of the reflected stress wave is dependent on the amount of energy that can be transmitted to the adhesive layer,[1] which in turn depends on the acoustic impedance of the adhesive.[1]

The two main adhesive systems used with ceramic armour systems are polyurethane and epoxy. The impedance mismatch between polyurethane and ceramic is greater than for epoxy and ceramic.[1] Using epoxy results in a 13-fold increase in the amount of energy transmitted from the ceramic to the adhesive and hence the ceramic is less likely to fail.[1] However, the ability of the adhesive to absorb energy is only of practical benefit if the ceramic remains bonded to the backing; the adhesive must not fail and hence polyurethane is preferred over epoxy currently as it is less likely to fail. If the bonding between ceramic and epoxy could be improved then not only would the ceramic be more likely to survive a ballistic impact because of the reduced intensity of the reflected stress wave, but also a stiff adhesive such as epoxy would reduce the bending of the ceramic during the impact. A further consequence of reducing the effect of a single impact might be a move towards an armour that is capable of withstanding multiple hits. Thus, there are many attractions to developing a technique to improve the bond strength between a ceramic and an epoxy adhesive.

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Previous research on alumina showed that preparing the surfaces to be bonded by using a laser treatment led to an increase in the number of hydroxyl groups on the ceramic surface and an increase in the adhesive bond strength.[2] Thus, it is hypothesised that increasing the number of hydroxyl groups on the surface of silicon carbide would also lead to an improvement in the strength of the bond between the ceramic and an epoxy adhesive. In this investigation this hypothesis is tested by using two methods to change the surface chemistry of silicon carbide and then investigating the effects of those treatments on the strength of tensile and shear joints made using an epoxy adhesive.

2. Experimental methods

2.1. Silicon carbide samples

Solid state sintered silicon carbide (Sicadur F) tiles were supplied by Ceramtec ETEC GmbH. The as-received tiles were used as control samples with no additional processing other than to clean the surface using a solvent wipe prior to material characterisation and adhesive bonding experiments.

In an attempt to increase the native surface oxide, samples of silicon carbide were refired in air at 1100 °C for 1.5 h.[3] All samples were cleaned prior to adhesive bonding. In order to measure the surface contamination caused by this technique a pre-cleaned sample was characterised using X-ray photoelectron spectroscopy.

Laser ablation was performed on a third set of samples using a 248 nm krypton fluoride ultra violet excimer laser. The laser was traversed across the surface. To obtain an even coverage each raster overlapped the previous by half the width of the laser spot. The intent of this technique is to oxidise[4] and possibly introduce hydroxyl groups[2] to the surface.

2.2. Characterisation of ceramic surfaces

A Hitachi 3200 N scanning electron microscope was used with a secondary electron detector and a backscattered electron detector at an acceleration voltage of 25 kV. All samples were coated with ∼3 nm of sputtered gold prior to characterisation.

X-ray photoelectron spectroscopy was performed using a VG ESCALAB mk II–XPS using a non-monochromated Al Kα source. Using the Al Kα source there was a depth of analysis of 5–6 nm. At least two measurements were taken in separate locations on each of the surfaces that were analysed. The data were referenced to carbon at 285 eV. The data were then peak fitted to determine the silicon carbide and oxide atomic concentration using the silicon peak. The oxygen peak was also peaked fitted to determine the atomic concentration of the hydroxyl groups.

The sessile drop technique was used to measure the surface wettability and energy. A computer controlled syringe with a camera perpendicular to the surface was used to determine the contact angle of the liquid on the surface. Distilled water and glycerol were used to determine the surface energy using the Owens and Wendt method.[5]

2.3. Mechanical testing of joints

Epoxy – silicon carbide joints were prepared to measure the tensile and shear strength of the bond. The epoxy, which comprised two resin components and one hardener, was provided by Resiblend PLC. The product is referred to as ESK T + ESK LV2 + ESK LVH2.

The control sample required only cleaning prior to bonding. Cleaning was carried out using a solvent wipe of methanol and then isopropyl alcohol to remove any residue. The refired sample was also cleaned using the same method after refiring. For the laser processed sample only the face that was in contact with the epoxy was processed. The whole sample was cleaned prior to laser processing.

The butt joint for measuring tensile strength required two pieces of silicon carbide with a 0.5 mm epoxy layer bonding them together. The joints were visually inspected prior to mechanical testing to determine that the adhesive had covered the entire joint face. A schematic diagram of this joint is shown in Fig. 1. The experiments followed ASTM standard D2094-00 for the preparation of bar and rod specimens for adhesion tests. The joints were tested at a rate of 1.0 mm per minute. At least 6 samples of each surface type were prepared for mechanical testing.

Joints were also prepared for measuring the strength in shear. The experiments followed ASTM standard D3528-96, the standard test method for determining the strength of double lap shear adhesive joints under applied tensile loads. The set-up for this joint is shown in Fig. 2.

All joint samples were mechanically tested to failure using an Instron 8800 testing machine with a 50 kN load cell. The load measurement uncertainty had been confirmed by certification to be 0.22% for loads 20–100% (10–50 kN) of the load cell range. At least 6 samples of each surface type were prepared for mechanical testing.

After testing, some fracture surfaces from both tensile and shear testing were observed using SEM, with a view to identifying the failure locus of the joints.

3. Results and discussion

3.1. Observations

The as-received silicon carbide samples were a dark glossy grey colour, Fig. 3(a). Following refiring in air the appearance changed to a glossy blue colour, Fig. 3(b). Laser processing produced a surface that had silver and black stripes, Fig. 3(c).

During laser processing of the sample it was noticed that a plasma was created. This suggests that the energy supplied to the surface was sufficient to cause decomposition. Silicon carbide has been assumed to decompose at temperatures greater than 2750 °C.[6]

3.2. Scanning electron microscopy

Three samples of silicon carbide were analysed using scanning electron microscopy; control, refired and laser processed.