Adhesive bonding of ceramic-based armor system

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ABSTRACT

Today's military vehicles are relying increasingly on ceramic-based armour for ballistics protection instead of the classic metal armour. This is because ceramic-based armour is much lighter in weight than metal armour, providing vehicles with increased agility and greater fuel efficiency, while not sacrificing ballistic protection. In the ballistic behaviour of adhesively bonded mixed armours (ceramic/metal backing), the influence of the adhesive layer used to bond the ceramic to the backing materials has to be taken into account. Poor bonding and adhesive selection for ceramics-based armour systems has been attributed to the reduced multi-hit capability for both personal and vehicles armour systems. Not only does the bond affect the strength at the interface, it could also minimize the damage to the ceramic caused by reflected shock waves. Two main factors that play major roles in adhesive bonding are the adhesive type and the surface treatment technique used to pretreat the adherend surfaces. In order to obtain a higher interfacial strength which ultimately leads to stronger bond strength, it is essential to tune the surface properties in the first place in such a way that the adhesive easily marries with the final surface chemistry. The final surface chemistry and morphology, basically, controls the adhesive's wettability and mechanical anchoring capacities.

In this work, silicon carbide (SiC) tiles were bonded to aluminum sheets using two different adhesive chemistries, namely, epoxies and polyurethanes with a constant bondline. The SiC tiles were surface treated prior to bonding using pre-screened surface treatment methods, namely, atmospheric pressure air plasma, laser texturing and methyl-ethyl-ketone (MEK) wipe. The aluminum sheets used were pretreated using an aerospace method which combines a grit-blast abrasion and silanization. All treated surfaces were characterized for water wetting properties and surface energies via contact angle measurements, chemical analysis by infrared spectroscopy as well as energy dispersive X-ray analysis and morphological analysis using scanning electron microscopy in order to optimize the treatment parameters. The bonded specimens were tested for peel strengths using a floating roller peel test method. A correlation established based on the bond quality with surface pretreatment methods and parameters will be discussed in-depth in this paper.

Keywords: *Adhesive bonding, surface treatment, atmospheric pressure plasma methods, adhesives, ceramic-metal armor, floating roller peel tests, wettability, surface characterization*

1.0 INTRODUCTION

The increasing need of energy saving without compromising the ballistic protection in today's military vehicles system has led to dependency on lightweight ceramic-based armor as opposed to the traditional metal armors composed of materials such as steel and aluminum. The basic disadvantage of the classic metal armor system is their excessively high weight as they could only provide adequate ballistic/blast protection provided a sufficiently high areal density of these materials is used [1]. Although ceramic based armors feature low density and high resistance to penetration, their major disadvantage is their reduced multi-hit performance compared with metallic based armour [2]. Ceramic armour typically comprises a ceramic front strike face, which is adhesively bonded to a composite or metal backing. The influence of the adhesive used to bond the ceramic tiles to the metallic or composite backing plate has to be taken into account. In an armor, it is not only holding two materials together; employing the right adhesive can be crucial since its function includes transferring shock waves from the ceramic to the backing plate in order to minimize the damage to the ceramic caused by the reflected shock waves. Adhesive bonding is a material joining process in which an adhesive, placed between the adherend surfaces, solidifies to produce an adhesive bond between the adherends. An adhesive may have superior mechanical properties upon curing, however, a strong adhesive-surface interface can be created only if the surfaces to be bonded are properly prepared prior to bonding. Therefore, two main factors that play major roles in adhesive bonding are the adhesive type and the surface treatment technique used to pretreat the adherend surfaces. In order to obtain a higher interfacial strength which ultimately leads to stronger bond strength, it is essential to tune the surface properties in the first place in such a way that the adhesive easily marries with the final surface chemistry [3-5]. The final surface chemistry and morphology, basically, controls the adhesive's wettability and mechanical anchoring capacities.

The main objective of this work was to identify the potential adhesive-surface treatment combination to obtain a strong interfacial bond between the adhesive and the adherends. Silicon carbide (SiC) tiles have been bonded to aluminum sheets using two different adhesive chemistries, namely, epoxies and polyurethanes with a constant bondline. The SiC tiles were surface treated prior to bonding using pre-screened surface treatment methods, namely, atmospheric pressure air plasma, laser texturing and methyl-ethyl-ketone (MEK) wipe. The aluminum sheets used were pretreated using an aerospace method which combines a grit-blast abrasion and silanization. All treated surfaces were characterized for water wetting properties and surface energies via contact angle measurements, chemical analysis by infrared spectroscopy as well as energy dispersive X-ray analysis and morphological analysis using scanning electron microscopy in order to optimize the treatment parameters. The bonded specimens were tested for peel strengths using a floating roller peel test method.

2.0 EXPERIMENTAL METHODS

2.1 Adhesives

Choice of the right adhesive chemistry plays an important role in the ballistic performance of ceramic armors. Therefore, two major adhesive chemistries, namely, epoxies and polyurethanes, commonly discussed in the defense applications, were tested in this work. An exhaustive list of adhesives was recommended by suppliers and distributors from North America (including Henkel and 3M) based on the handling and physical properties of the adhesives under different environmental conditions. However, only a selected number of potential adhesives were tested in our study. The epoxy adhesives will be mentioned as E1, E2, E3 and E4 and the polyurethanes as PU1 and PU2 throughout this paper. The bondlines (adhesive thickness) used in this study were 0.010" for the epoxy adhesives and 2 mm for the polyurethane adhesives and were chosen based on the manufacturer's recommendation. In general, the polyurethane adhesives are highly flexible and require higher bondlines for better impact resistance. Epoxies, on the other hand, are rigid and require thinner bondlines for improved bondability.

2.2 Surface treatment methods

Several surface pretreatment methods including mechanical, chemical and physical as well as combination of more than one method has been used on the SiC tiles purchased from Morgan Inc. These include methyl-ethyl-ketone (MEK) wipe, grit-blast (GB, 180 grit size), glycidoxypropyltrimethoxy-silane (GPS) silane, grit-blast followed by GPS silane treatment, atmospheric pressure Openair® (OA) plasma, OA plasma followed by GPS silane treatment and the laser treatment. The as-received SiC tiles surfaces were used as baseline. Due to the numerous methods used, the experimental part will only be briefly described here. For aluminum (AA 061-T6 alloy), however, a traditional method used by aerospace industry was employed and kept constant throughout the study. This method combines the grit-blast abrasion technique using 180 grit white alumina particles followed by GPS silanization.

The GPS silane treatment was realized by wetting the SiC tiles with the silane solution pre-prepared using GPS precursor by hydrolyzing in nanopure water with a pH controlled using acetic acid. The SiC tiles sprayed with silane for 10 minutes were dried in an oven at 100ºC for 1 hour prior to surface characterization and bonding. The OA plasma treatments were carried out using a RD1004 nozzle controlled by a FG5001 plasma generator. The plasma was generated and maintained inside the Openair® rotation jet using a non-equilibrium discharge applied to air (excitation frequency of 21 kHz, operating at 300 V and 16.3 A). The 25° nozzle, in rotation at an angular speed of 2800 rpm, was filled with compressed air at an input pressure of 3 bars. The distance between the bottom part of the nozzle and the SiC surface and the exposure time of plasma by moving the SiC tiles under the plasma shower were optimized based on the surface chemical and wettability properties prior to bonding (Figure 1). The laser treatment was performed with a 10 W, 10 ps pulsed laser system equipped with a high precision, computer-controlled translation platform. The laser beam having a Gaussian beam intensity and wavelength of 1064 nm was operated at 1 MHz frequency. A 40 mm focal length lens was used with an 8x magnification resulting in a beam focus diameter of ~ 25 μm. An Argon gas pressure of ~30 psi was used for the nozzle during pattern fabrication phase which aided in removing particle agglomeration on the machined surface as well as protecting the focusing optics. The grid pattern was designed with the purpose to provide a larger contact area and the mechanical interlocking effects by retaining the adhesive in the channels. Test samples were prepared using multi-pass technique with laser beam travel in zig-zag direction for machining SiC ceramics surface. Each sample was prepared using 4 passes to cover the desired surface area.

Figure 1. Openair® plasma during the treatment of a SiC tile.

2.3 Surface characterization

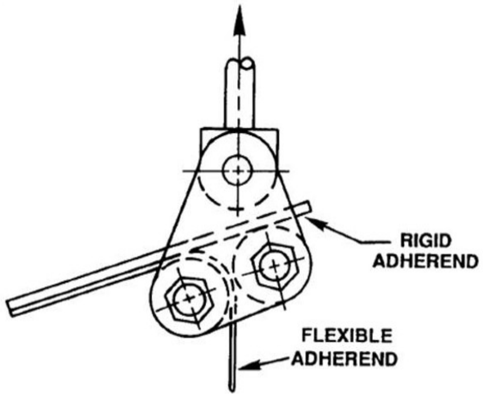
Hitachi SU-70 field emission scanning electron microscopy (FESEM) was used to study the morphological modifications of the pretreated surfaces. Infrared reflection absorption spectroscopy (IRRAS) (Nicolet 6700 FT-IR) was employed to characterize the surface chemistry of the pretreated surfaces. The spectra were recorded from 4000 to 650 cm^{-1} with a resolution of 4 cm^{-1} and 64 scans with the background subtracted using a clean gold sample. A Kratos Axis Ultra DLD X-ray photoelectron spectroscopy (XPS) was used to precisely identify the chemical composition of the treated surfaces, owing to the depth resolution of less than 10 nm for this technique. The wetting characteristics and surface energy measurements of all the surfaces were performed using a contact angle goniometer (Krüss GmbH, Germany) via static contact angle measurements of liquid drops of size ~ 4 μl using Laplace-Young method. The wettability was studied by measuring the contact angle of water on the different surfaces. The surface energies were determined using the Owens-Wendt-Rabel-Kaelble (OWRK) theory, widely used to determine the surface free energy of solid surfaces [6]. The OWRK method is based on a two-component model, i.e., the surface free energy comprises of (1) a dispersive component (Van der Waals interaction) and (2) a polar component (dipole interactions, hydrogen bonding). This method involves contact angle measurements from two different liquids of known liquid surface energies (σ_l) with polar (σ_l^P) and dispersive (σ_l^D) components and the solid surface energy ($\sigma_S = \sigma_s^P + \sigma_s^D$) is obtained from the OWRK equation (Equation (1)) for both liquids.

$$\sigma_l (1 + \cos\theta) = 2 [(\sigma_l^D * \sigma_s^D)^{1/2} + (\sigma_l^P * \sigma_s^P)^{1/2}] \qquad \text{Equation (1)}$$

In this work, water (σ_l = 72.8 mN/m; σ_l^D = 21.8 mN/m; σ_l^P = 51 mN/m) and diiodo-methane (σ_l =50.8 mN/m; σ_l^D = 49.5 mN/m; σ_l^P = 1.3 mN/m) were used for surface energy measurements and their contact angles were measured on the respective surfaces.

2.4 Mechanical testing

The mechanical tests were performed via a floating roller peel test based on the standard ASTM 3167. A schematic of the test method is shown in Figure 2. This test method covers the determination of the relative peel resistance of adhesive bonds between one rigid adherend (SiC tile) and one flexible adherend (Al) when tested under specified conditions of preparation and testing. In this method, the flexible adherend (Al) is peeled from the rigid adherend (SiC tile) at a controlled angle of peel using the test fixture shown in Figure 2. A crosshead speed was 152 mm/min on an overlap area of 1” x 4” was used. Figure 3 shows the schematic of the bonded specimen and respective dimensions.



a
Roller drum test fixture

Figure 2. Schematic of floating roller test method (ASTM 3167)

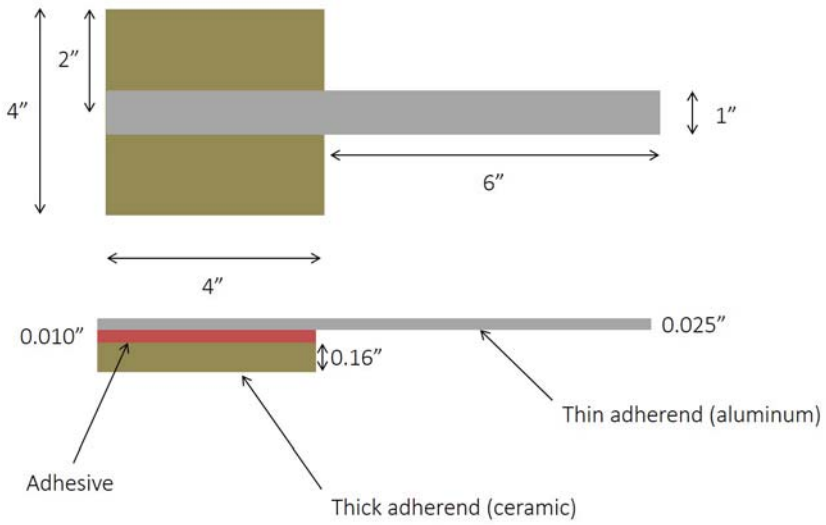


Figure 3. Schematic of the SiC-adhesive-Al bonded specimen for floating roller peel test

3.0 RESULTS

The surface treatment process parameters in OA plasma and laser techniques were optimized initially based on surface properties such as water wettability of these surfaces via CA measurements. In order to avoid repetitive contents, only the optimized results have been presented and discussed here. Figure 4 presents the water contact angle values and the total surface energies (sum of dispersive σ_l^D and polar σ_l^P components) obtained on the SiC tiles surfaces as a result of the respective surface modification. It is clear from these results that the surface treatment plays a significant role in controlling the surface energy and hence the wettability. A very low water contact angle (~ 20°) and high surface energy (~ 71 mN/m) was obtained on the OA plasma treated tiles compared to the as-received (AR) counterpart (CA ~ 67° and SE ~ 48m N/m) other techniques studied. The change in surface energy and hence wettability behaviour of the OA plasma may be attributed to the chemical changes that the plasma exposure has brought on the surface. Plasma technique is known to incorporate oxygenated functionalities on the treated surfaces and hence result in increased surface energy. This phenomenon is particularly of interest in adhesive bonding since the chemical reaction occurring during the adhesive's curing process generates hydrophilic functionalities such as hydroxyls. Engineering the surface to match the adhesive chemistry can enhance the number of chemical bonds at the adhesive-surface interface and hence providing a stronger bond strength. Mechanical abrasion such as grit-blast techniques have their own advantage in mechanically anchoring the adhesive within the texture created by the abrasion. However, a chemical interlinking may not occur in such techniques. The laser treatment of SiC tiles resulted in a complete spreading of the water droplet making it impossible to measure the water contact angle and the surface energy. This behaviour may be attributed to a combination of texturing by Laser energy used as well as the chemical modification. These results indicate that the plasma and the laser treatment methods may be promising in increasing the interfacial bond strength between SiC and the adhesive. Further investigations on the chemistry of the surface was performed by IR spectra to complement the surface wettability behaviour.

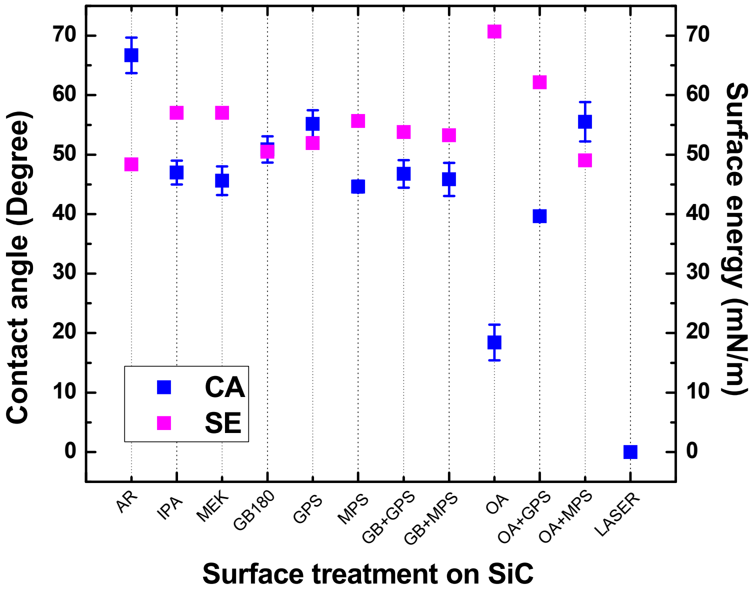


Figure 4. Contact angle (CA) of water and the total surface energy (SE) of variously treated SiC tiles surfaces

The IR spectral analyses showed a significant variation in the appearance and broadening of the Si-O/Si-N intensities between 950 cm^{-1} and 830 cm^{-1} on treated surfaces. This type of broadening has been previously reported, however, using a different kind of treatment method involving plasma [7]. Figure 5 shows the IR spectra of the variously treated SiC surfaces. The inset shows specifically the spectra of the surfaces treated with OA plasma and as-received. It is clear that the plasma treatment has incorporated oxygenated functionalities on the surface, which in turn helps increase the surface energy. Silane treatments also showed such broadening due to the formation of silanols upon silanization process. Laser treated surface was too rough to reflect IR to obtain a spectrum.

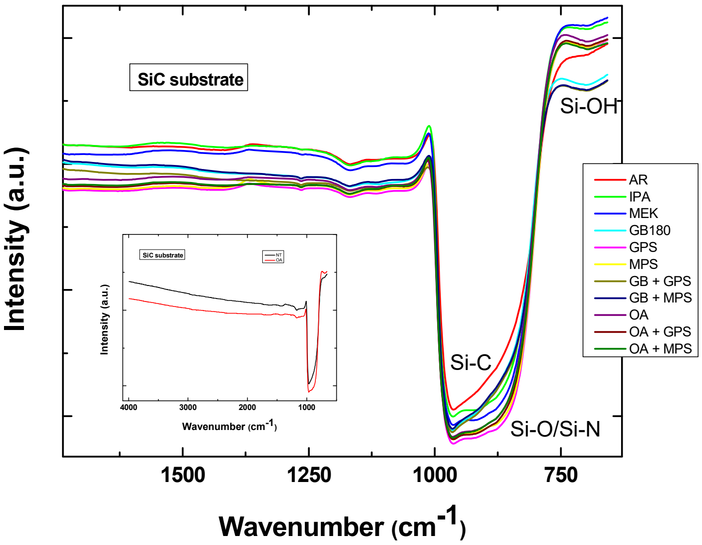


Figure 5. Infrared spectra of variously treated SiC tiles surface; Inset shows the spectra of OA treated surface versus as-received surface for specific comparison

Morphological evolution and the chemical changes of the surfaces as a function of surface treatment technique was also investigated on the SiC tiles. Figure 6 shows the FESEM images of selected surfaces of interest. The as-received SiC tiles surfaces were not completely smooth and their SEM image revealed certain texture with a root mean square (*rms*) roughness of ~ 0.5 µm as measured by an optical profilometry. The OA plasma treated tiles presented similar morphology, however, the surface roughness seems to have increased slightly to a *rms* value of ~ 1.0 µm. The plasma treatment seems to have slightly affected the texture in addition to the surface chemistry as evidenced by IR analyses. The laser treatment has in fact resulted in a significant change in morphology rendering the surface highly textured. The surface was so rough that the light was completely absorbed in it making it difficult to obtain the roughness value. As a comparison of a texturing method, the Figure 6 also shows the grit-blasted surface where a very high surface roughness was expected. However, the grit-blasted surface appeared to be very similar to that of the OA plasma treated surface. The morphological evolution indicates that an excellent mechanical anchoring of the adhesive will be expected in case of the laser treated surfaces.

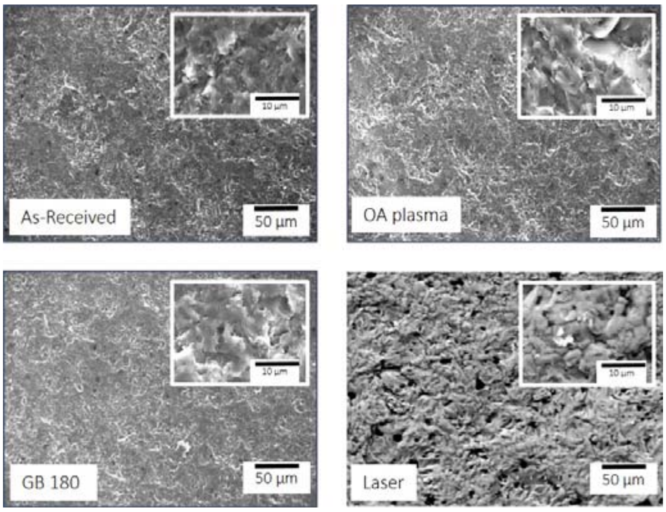


Figure 6. SEM images (inset: magnified images) of as-received (AR), OA plasma, grit-blast and laser treated SiC tiles surfaces

EDX analysis was further performed to explore the chemical modifications on all surfaces. Figure 7 (a) shows the surface treated using chemical and physical methods. Plasma treatments resulted in an increase in the oxygen content which complements the IR analyses showing possibilities of incorporation of oxygenated functionalities upon exposure to plasma. Silane treatment also showed increase in oxygen concentration which is normal due to the formation of silanols on the surface. However, the laser treatment presented twice the oxygen concentration as compared to the plasma treated counterparts. This shows that the laser treatment not only improved the texture, but also enriched the chemical nature of the surface in favour for bonding. On the other hand, the Figure 7 (b) showing the EDX oxygen concentration on the mechanically treated surfaces presented a very high oxygen content and also showed the presence of aluminum on the surface. This indicates that the loose alumina particles following blasting using alumina grits remained on the surface even after blowing the blasted surface with N_2 gas followed by a MEK wipe. This indicates that the adhesive may not bond well to the SiC surface due to the presence of an intermediate alumina particle pollution which has no chemical interaction with the underlying SiC surface.

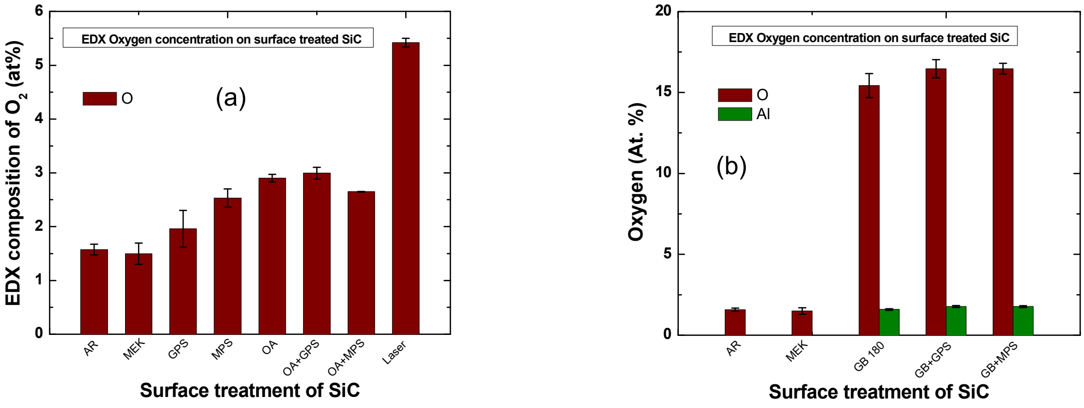


Figure 7. EDX chemical analyses of variously surface treated SiC tiles versus as-received and MEK wiped surfaces: (a) Chemical and physical methods; and (b) Mechanical methods

Owing to the surface precision of up to a depth of 10 nm from the surface an XPS can provide, selected surfaces of interest were investigated using this technique. The survey spectra presented all elements of interest, namely, the silicon, carbon, oxygen as well as nitrogen with slight intensities. High-resolution spectra of each element were obtained and investigated. The most important peak of interest, namely, the Si 2p peaks are shown in Figure 8. The Si 2p peaks were further deconvoluted into two Gaussian peaks at binding energies 102 eV and 104 eV representing Si-C and Si-O bonds, respectively. A slight increase in the atomic concentration from 21 at. % to 25 at. % was observed with OA plasma treatment for the Si bonded to O, which was 50 at. % for the laser treated surface. These observations confirmed that the physical methods have a positive influence on the surface characteristics in favour of improved bonding and the laser treatment possess higher potential.

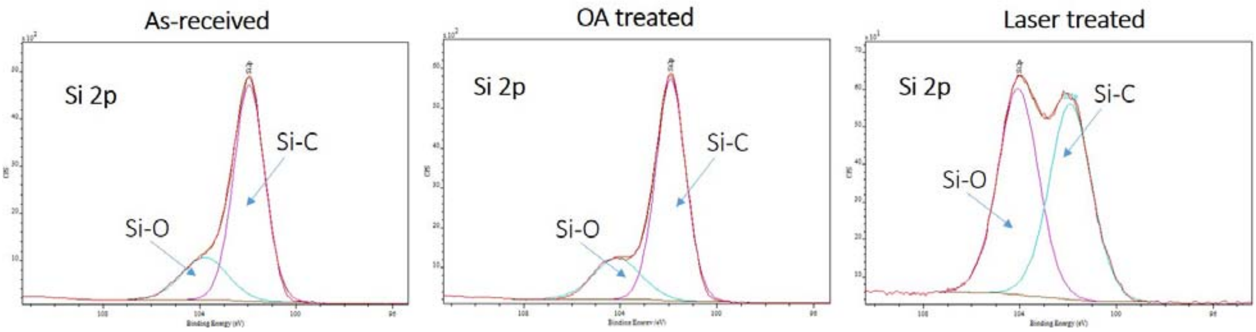


Figure 8. High-resolution Si 2p XPS spectra of SiC tiles treated by OA plasma and laser technique as compared to their as-received counterpart.

For further bonding and testing, the OA plasma and laser treatment were chosen with the as-received and MEK wiped tiles as baseline. The as-received tiles and the OA and laser treated tiles were bonded to thin aluminum sheets using adhesives, E1, E2, E3 and E4 from epoxy family and PU1 and PU2 from polyurethane family. The configuration of the bonded specimens for the floating roller peel tests has been detailed in section 2 (Experimental methods) and shown in Figure 3. The objective at this point was to evaluate the pre-screened surface treatment methods under bonded conditions leading to the identification of best performing adhesives for further testing including ballistic testing which are currently in-progress and are not included in this paper.

Figure 9 shows the floating roller peel test results for variously treated SiC tiles bonded to pretreated aluminum with different adhesives. It is clear from the results that among the same family of adhesives, the performance varied for the same surfaces. For instance, while the epoxy E4 provided a peel strength of ~ 1.5 & ~ 3.2 lbf on specimens bonded with as-received and OA treated SiC tiles, respectively; for the same surfaces, we obtained ~ 44 and ~ 49 lbf with E2. However, both adhesives showed slight improvement of performance on the OA treated specimens and the behaviour may be considered similar with all epoxy adhesives. The specimens prepared with laser treated SiC tiles were not tested on all adhesives, however, given that the adhesive E2 performed better than other epoxy adhesives, the E2 was tested with this surface treatment method. As expected, the laser treatment exhibited higher peel force (~ 53 lbf) than OA treated and baseline counterparts. Figure 10 shows the images of specimens bonded with epoxy adhesives after peel tests demonstrating the mode of failure. In all cases, only the behaviour on SiC tiles have been presented as no interfacial (adhesive) failure was observed on the aluminum side. The adhesive was found intact on the aluminum surface confirming that the aerospace method of combining grit-blasting and silanization was appropriate and maintained in all adhesive systems studied. It is clear from these images that the adhesives' adhesion to ceramic side was very poor in as-received and MEK wiped cases. Although some traces of the epoxies were observed on OA plasma treated SiC surfaces, it is very evident that the adhesive E2 showed the best failure mode upon treating with MEK wipe, OA plasma and Laser. Fully cohesive failure mode has been observed with OA plasma and Laser treatment. This adhesive also provided better peel strengths compared to other epoxies. It must be noted that all these investigations were carried out on as-bonded specimens in room temperature conditions. A strong dependence on surface treatment can be witnessed upon exposure of the peel specimens to environmental degradation conditions as high heat and humidity as well as to temperature fluctuations.

On the other hand, the PU adhesives showed a significant increase in the peel strength. In particular the PU2 adhesive showed a peel strength of greater than 75 lbf on all surfaces irrespective of the treatment method employed. This indicates that these adhesives with very high elongation (flexibility) were not greatly influenced by the surface treatment as tested under as-bonded conditions. However, it is known that extreme temperature fluctuations and humidity greatly affects the interface between adhesive and the surface. In order to understand the effect of the surface treatment, it would be essential to test these specimens under environmentally degraded conditions. Figure 11 shows the images of specimens bonded with polyurethane adhesives after peel tests demonstrating the mode of failure on the SiC side. In both the PUs, a completely cohesive failure mode has been observed. It is expected for a flexible adhesive with very high elongations to perform well under non-degraded conditions with minimal or no surface pretreatment applied. In the two cases studied, however, the PU2 provided higher peel strengths than PU1. Again, after exposure to temperature variations and high humidity, the influence of surface treatment can be revealed. The degradation studies are in progress and will not be discussed here. The PU2 adhesive in Figure 11 shows voids in the middle, which, is attributed to insufficient cure conditions. PU2 is a moisture cure adhesive candidate. The voids in the middle in PU2 indicates that on large areas, this adhesive that cures inward starting from the outer contact point forms a strong contour that turns into a barrier for moisture to penetrate further to cure the adhesive in the central regions.

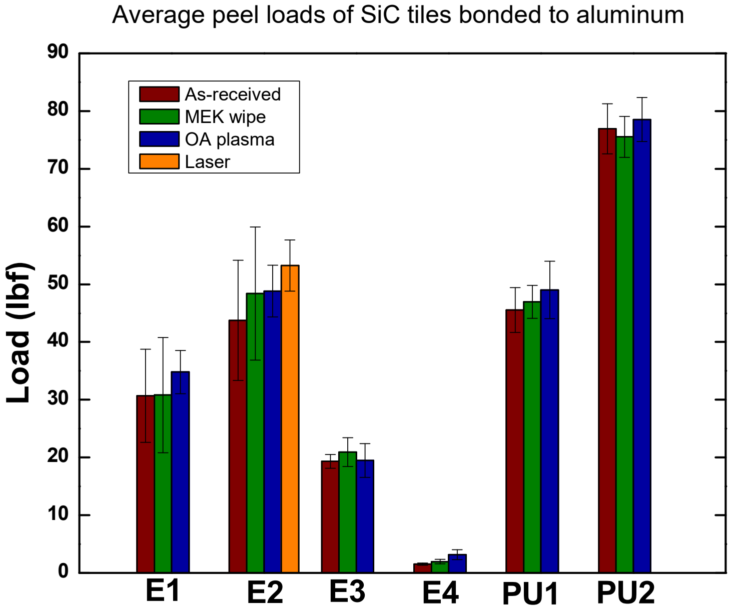


Figure 9. Floating roller peel test results of SiC-Al specimens bonded with different epoxy and polyurethane adhesives; SiC tiles were treated with OA plasma and laser techniques while as-received (AR) and MEK wiped SiC tiles served as baselines.

As far as the adhesive's role is concerned, there are mixed information on the role of epoxies and polyurethanes in the literature. A study on ceramic-steel armor bonded by polyurethane adhesive showed more resistance to spalling than those with epoxy [8], while another study by Zaera *et al.* reports less lateral damage with epoxy adhesive than that of polyurethane in ceramic-metal armors as more energy is transferred into epoxy layers due to less mechanical impedance mismatch than polyurethane [9]. These authors in favor of epoxy adhesives also suggest that the epoxy layer is stiffer and will reduce the degree of bending experienced by the ceramic layer during impact. These questions and others related to environmental issues will be answered by evaluating wide range of potential adhesive systems from both these families under impact and ballistic tests. Further work, namely, drop-tower puncture testing in accordance with ASTM D3763 and ballistic tests in accordance with NATO STANAG level 3, is in-progress in order to investigate the role of improved bonding procedures on multi-hit ballistic performance of ceramic based armour systems.

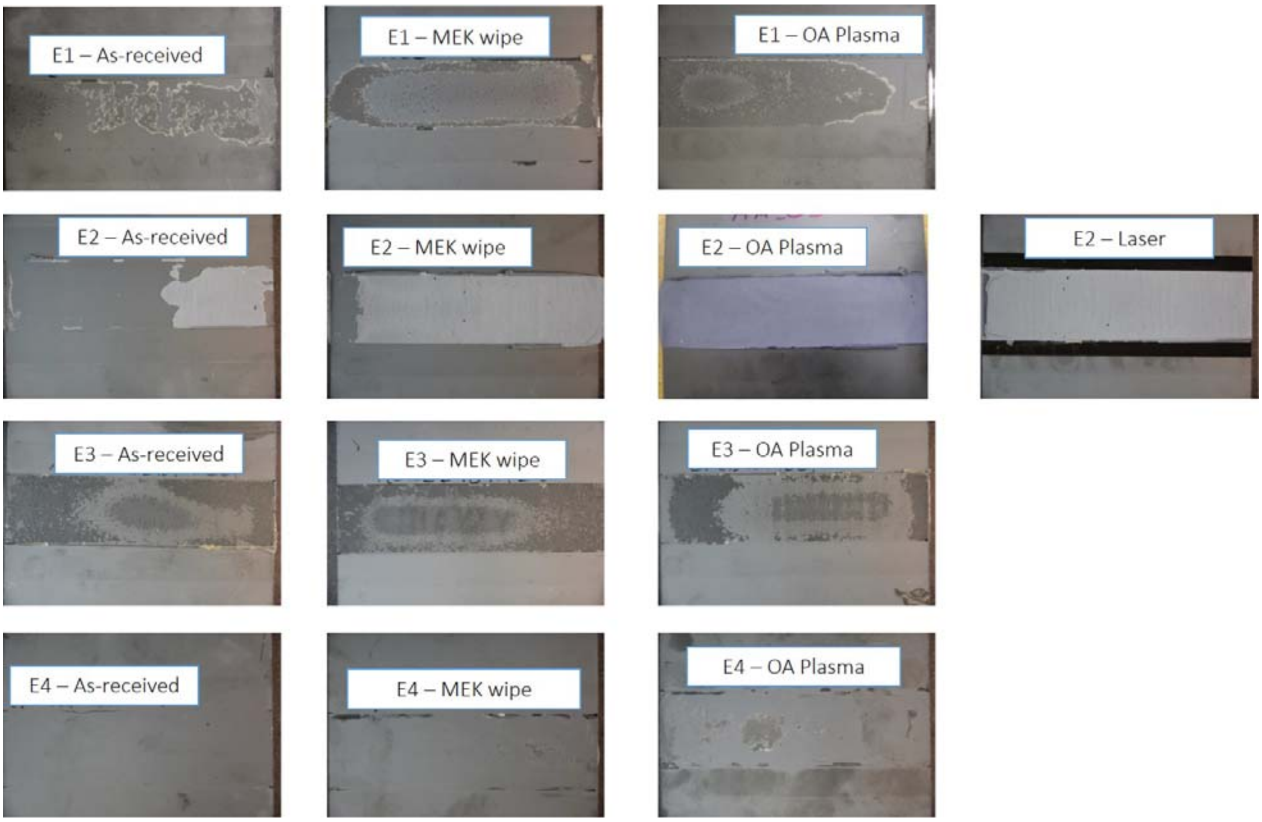


Figure 10. Images of peel tested specimens with epoxy adhesives E1, E2, E3 and E4, showing their failure modes

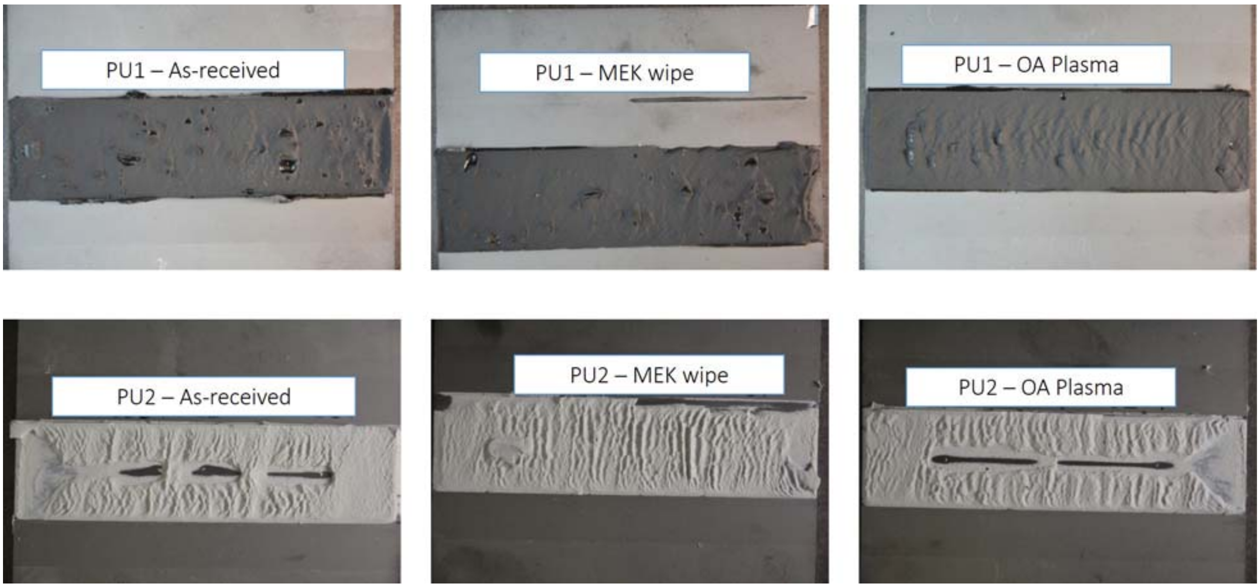


Figure 11. Images of peel tested specimens with polyurethane adhesives PU1 and PU2, showing their failure modes

4.0 CONCLUSIONS

Owing to the importance of the presence and influence of an adhesive layer in ballistic protection of ceramic-metal armors, in this work we have addressed the fundamental requirement of two important factors, namely, the selection of the right adhesive system and the use of appropriate surface treatment method. The results based on various surface characterization techniques revealing the morphological and chemical evolution as a function of surface treatment technique used showed that the surface energy responsible for improved wettability of water/adhesive as well as the surface geometry for increased mechanical anchoring of adhesive is greatly influenced by the surface treatment. The results showed that the surface treatment helped improve slightly the peel performance owing to the enrichment of surface's chemistry and texture. The epoxy adhesives particularly demonstrated this dependency as evidenced by the failure modes of the peel tested specimens where the OA plasma treated surfaces showed presence of adhesives either in traces or in bulk. The laser treatment provided slightly higher peel force (12% higher than as-received counterparts) with one of the epoxy adhesives (E2). These results indicate that the physical treatment methods showed better surface characteristics in terms of chemistry and wettability as compared to the cleaning method (MEK). PU adhesives showed little effect on the peel performance with surface treatment which is expected in case of flexible polyurethanes. However, in both adhesive systems, further work in environmentally degraded conditions are required to further conclude the effect of surface treatment on the adhesive-ceramic interfaces. Further work is also in progress to evaluate the PU adhesives with laser treatment as well as the drop tower puncture tests and ballistic performance of the ceramic-aluminum bonded system.

5.0 ACKNOWLEDGEMENT

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