Effect of sandblasting substrate treatment on single lap shear strength of adhesively bonded PEEK and its composites

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Summary

In this work, the effect of sandblasting treatment on surface morphology, wettability and the adhesive bonding strength of PEEK matrix, carbon fibres (CF) and glass (GF) reinforced PEEK substrates was studied. The adhesive was an epoxy based system. The strength of bonded joints was tested with the use of single lap shear tests.

1 General introduction

Adhesive bonding is one of the preferred methods to join composites and polymeric materials. In fact this method offers many advantages such as uniform stress distribution along the joint and minimizing stress concentration, weight reduction, compared with bolding for example.
PEEK (PolyEtherEtherKetone) is a semi-crystalline high temperature resistant thermoplastic material [1-3]. However, the low surface energy and ability to resist to chemical changes makes PEEK adhesive bonding complex compared to metals or thermosettings. As a consequence, the preparation and surface treatments of adherents are primary steps before bonding [4].
Nowadays, several techniques of surface preparation for adhesive bonding are used. These techniques can be classified in three main groups; mechanical treatments; paper abrasion and sandblasting, physical treatment; plasma and laser treatments and finally chemical treatments like solvent degreasing and chemical etching [5].
Low cost, environmental friendly and easy to implement make sandblasting treatment one of competitive methods for preparing thermoplastic material surfaces before adhesive bonding [6,7]. This technique consists of projecting fine abrasive particles at high-velocity in the aim to clean or etch a surface.
In this study, neat matrix PEEK and its composites short carbon and glass fiber reinforced PEEK were treated with sandblasting by varying experimental conditions as projection time and mean particle sizes. Investigation of changes in the experimental conditions is to understand the effect of morphologies created after treatment on wettability and adhesive joint strength. The tests were performed using single lap shear specimen in a monotonic tensile load.

2 Experimental

2.1 Materials

Injected plates of 3mm thick of PEEK (PEEK 90G), 30% by weight short carbon and glass fiber reinforced PEEK composites (PEEK 90CA30 and PEEK 90GL30 respectively) from Victrex® were used. The transversal section of composites was examined by optical light microscope after polishing by a metallographic method.

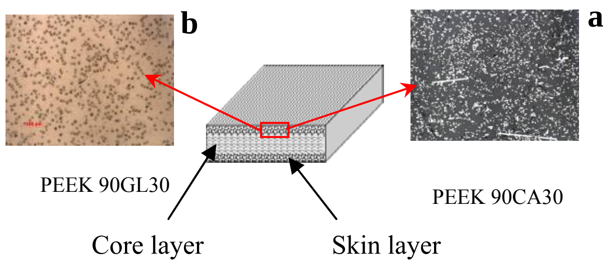

Fig.1. Optical micrographs of transversal section orthogonal to the carbon and glass fibers direction of composites: (a) PEEK 90CA30 carbon fiber Φ ≈ 7µm and (b) PEEK 90GL30 glass fiber Φ ≈ 15µm. (Φ: fibers diameter)

The observations show that the majority of fibers (carbon and glass) in the skin layer are aligned in the direction of injection (Fig.1) as a consequence of the friction between the mold and the flow in the

injection molding process. In the core of both composites the fibers are randomly oriented.
The adhesive used in this study was a two parts epoxy based 9323 B/A 3M, mixed in the following proportions; for 100% in weight for the epoxy base 27% in weight are added for the hardener part as indicated in the adhesive technical support. The curing process is defined as 65 °C for 2 hours. The glass transition temperature of the cured adhesive was determined with using differential scanning calorimetry (DSC) ($Tg_{Adhesive}$= 66°C).

2.2 Thermal characterization

The DSC analysis was carried out using the DSC STAR 01 module from METTLER TOLEDO with a 25°C to 400°C temperature range and a heating rate of 10°C/min. In semi-crystalline polymers the degree of crystallinity is an important factor, since the mechanical behavior of these materials is linked to the crystallinity degree [8]. The degree of crystallinity was calculated using the following relationship: $X_c = \frac{Q_m}{\Delta W Q_c} 100$, where X_c is The degree of cristallinity (%), Q_m is the melting enthalpy [$J.g^{-1}$], Q_c is the melting enthalpy for fully crystallized PEEK (Q_c=130 Jg^{-1} [16]) and finally ΔW is the weight content of PEEK in the composite.

Table.1. Materials characteristics.

Materials	Type of Reinforcement	Fiber weight content W_f(%)	DSC		
			Glass transition* Tg (°C)	Melting enthalpy Q_m [Jg^{-1}]	Degree of cristallinity Xc (%)
PEEK 90 G	-	-	147	49,83	38
PEEK 90CA30	Carbon	30	146	30,33	33
PEEK 90GL30	Glass	30	145	30,58	33

* Mid point.

2.3 Abrasive surface treatment (Sandblasting)

The particle projection treatments were performed in a commercial sand-blasting chamber. It is equipped with a ceramic jet nozzle with a diameter of 8 mm and linked to an air compressor.
The pressure was regulated at 5 bars and the distance between the nozzle and target was holed at constant value for all tests (L = 80 mm). The sandblasting process principle is shown in Fig.2.
The three materials, neat matrix PEEK (90G) and both composites were subjected to sandblasting with varying experimental conditions (particles characteristics, time of projection).
The impact angle was chosen at 90° to minimize its influence and to reveal the particle size effect. Projection time was varied from 5 seconds to 45 seconds.

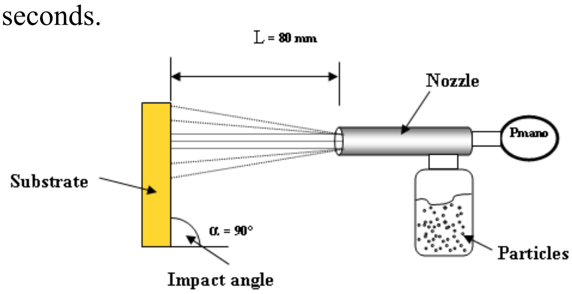

Fig.2. Principle of sandblasting device.

Three different particle sizes (50µm, 110µm and 250µm) were used to vary the level of the surface texture. These particles are commercially composed chemically at 98% of alumina (Al_2O_3) and have an irregular shape with abrasive angles.
After treatment all samples were cleaned ultrasonically with ethanol for 15min to eliminate any particles and dust present on surface.

2.4 Surface roughness measurements

The surface roughness parameters of untreated and treated surfaces were characterized by using a 3D Veeco profiler type WYCO 9300. The analyses are based on non contact Vertical Scanning Interferometry (VSI) measurement mode.

2.5 Wettability measurement

The contact angles between the liquid (distilled water) and treated materials were measured using a DSA30 apparatus-from Kruss®. All measurements were made with distilled water as a probe liquid. The materials were ultrasonically cleaned with ethanol for 15 minutes before the measurement to minimize surface contaminations.
Tests were carried out in an ambient conditioned room (T=22±1 °C, RH +40±5%). After dispensing onto the substrate, the drop shape was recorded using a camera-video. The static contact angle was taken 20 s after distilled water drop deposition.

2.6 Joint preparation and testing

Specimens for single-lap shear test were prepared with two pieces of PEEK measuring 106 mm by 25 mm and 3 mm thick bonded along 12,5 mm according to the international standard ISO 4587 :2003 (Fig 3). A specific device was realized in laboratory to make manufacturing of the bonded specimens easier. The overlap joint and the adhesive thickness (200 ± 20 µm) were controlled. After sandblasting treatment, the substrates were ultrasonically cleaned with ethanol for 15 min before bonding. The single lap shear bond strength for each material and sandblasting conditions was measured by tension loading using a MTS Bionix (Model 370.02) axial/torsion tester equipped with a 25 kN load cell and a displacement sensor at room temperature (23 ± 1 °C) and at a cross-head speed of 1 mm/min.

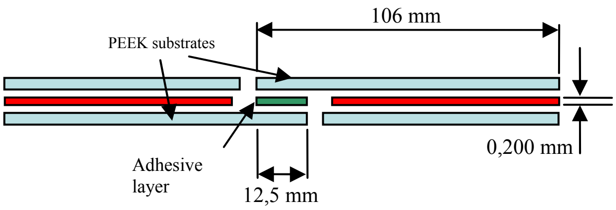

Fig.3 Diagram of single lap shear specimens.

The apparent shear strength values (τ) were calculated according to the following equation ($\tau = F/A$), where F the load at failure (N) and A represents the overlap shear area (mm^2).

3 Results and discussions

3.1 Surface analysis

The surface topography and the roughness level of all treated materials were considerably modified compared to as received specimens and depend strongly on sandblasting conditions.

Scanning electron micrographs of untreated and the more drastically treated surfaces (250 µm average particle size for 45 seconds) of the three materials are shown in Fig 4. These micrographs show that in the case of the untreated CRF PEEK (Fig 4.c) the fibers lay parallel to the mold flow in the skin layers inducing anisotropic surface roughness on the surface of molded composites. This phenomenon is less marked for GFR PEEK (Fig 4.e). Moreover, the SEM observations reveal a high level of isotropy for all sandblasted materials. Nevertheless, the SEM observations give only qualitative information on surface morphology. The quantitative surface analysis was performed using a 3D laser profilometer and two 3D roughness parameters were selected: the arithmetic average surface heights S_a (µm) defined in international standard ISO 25178 and the density of summits Sds defined in European standard (EUR 15178N). The definitions of these two parameters are:

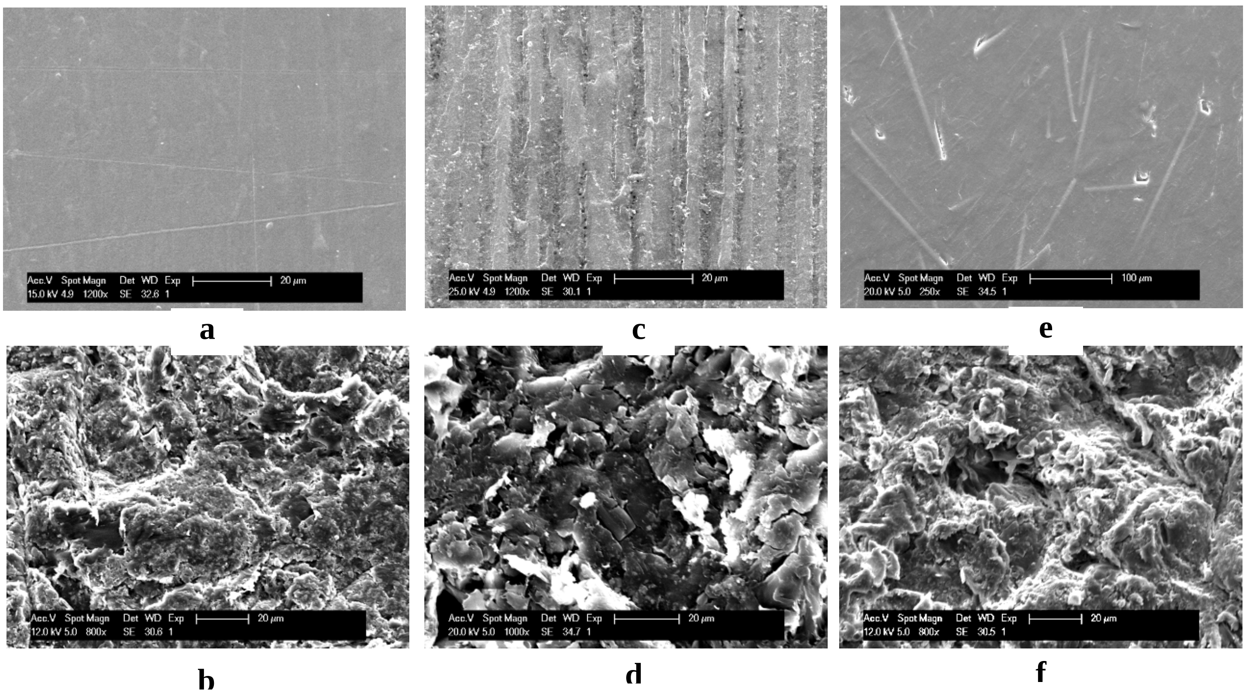

Fig.4. SEM micrographs show the untreated surfaces and sandblasted surfaces (250 µm average particle sizes for 45 seconds) for PEEK pure (a-b), carbon fiber reinforced PEEK 90CA30 (c-d) and glass fiber reinforced PEEK 90GL30 (e-f)

$$S_a = \frac{1}{A}\int_A |Z(x,y)|dxdy \qquad (3)$$

$$S_{ds} = \frac{Number \text{ of summits}}{A} \qquad (4)$$

where A corresponds to the defined area and Z(x,y) are the different heights according to x and y. Sds is the number of summits of a unit sampling area; a peak is defined if it is higher than its eight nearest neighbors [15].

The variation of the roughness parameters S_a and Sds as a function of projection time for all materials and all the particles sizes are shown in Fig.5.

The results show that in all cases the calculated S_a parameter increased significantly up to about 5 seconds of treatment beyond which a flattening of S_a is observed. Moreover, as expected, the smoother surfaces are observed for the finer particles. The both composites display the highest roughness level in all the cases (raw and treated materials for any average size) probably due to the presence of the fibers. S_a parameter gives information about the surface roughness level, but no information about the surface morphology. In fact, it is possible to observe the same or similar S_a value for different surfaces showing different peak-valley values [9].

Indeed, the summits density (Sds) evolution as a function of time reveals the morphological differences between all treated materials as shown in Fig.5.

Indeed, both composite materials treated during 5 seconds display the same S_a but different Sds for all the average particle sizes used. Therefore the Sds reveals that the sandblasting modifies considerably the surface morphology and creates peaks and valleys.

3.2 Wettability behavior

In order to investigate the wettability behavior as a function of roughness parameters two projection times were selected (5 and 45s). Fig.6 shows the apparent contact angle evolution as a function of average particle sizes.

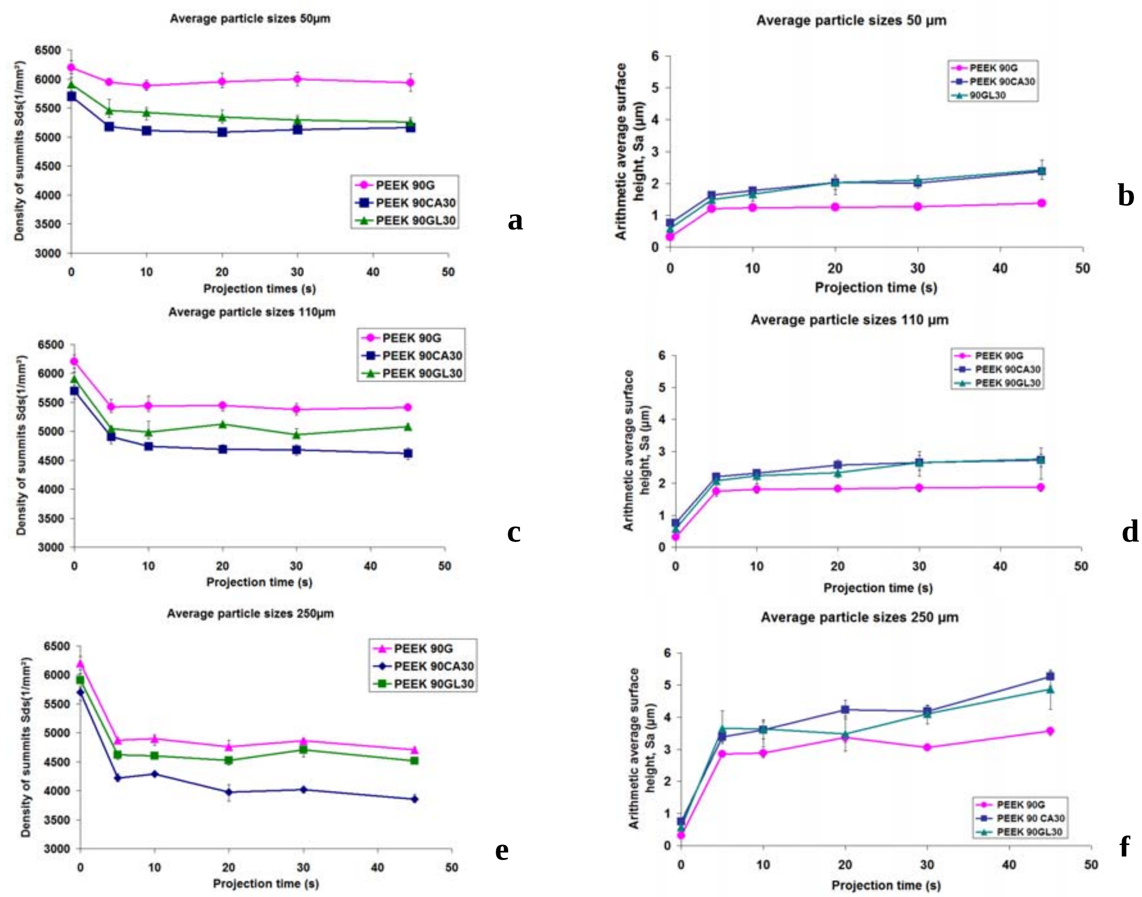

Fig.5. Sds and S_a evolution as a function of projection time and average particle sizes; (a-b) 50µm, (c-d) 110µm and (e-f) 250µm.

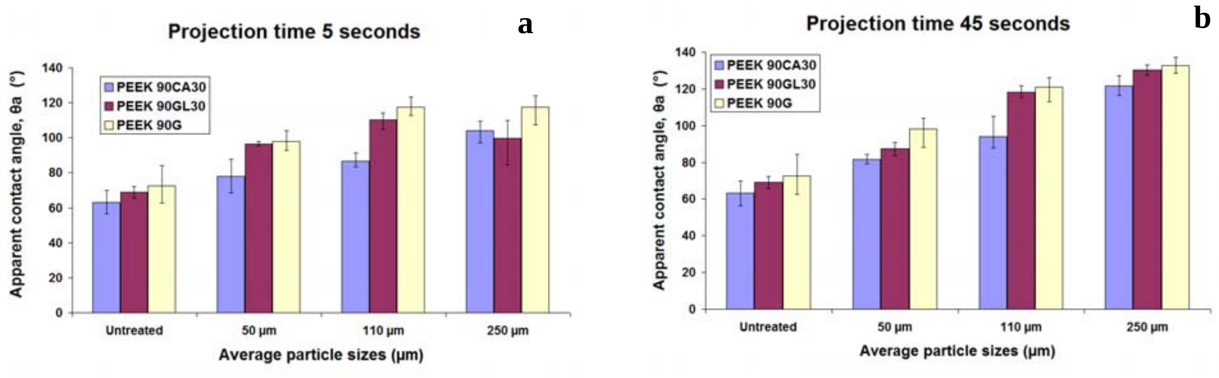

Fig.6. Measured apparent contact as a function of average particle sizes for two projection times; (a) 5 seconds and (b) 45 seconds.

The result indicates that the measured contact angles increase with the projected particle sizes. It is linked, as previously mentioned, to the creation of high peeks and deep valleys which play as a barrier to the spreading of the droplet [10, 11]. The result shows also that the CRF PEEK has the better wetting behavior in all cases (as received and treated samples). This phenomenon is due probably to the presence of fibers in surface which can promote the spreading of deposit water droplet.

3.3 Lap-shear bond strength

Single-lap shear tests were performed to investigate the bonding properties of the sandblasting treated surfaces. In the case of untreated neat matrix PEEK the bond shear stress is quite low (τ = 2±0,6 MPa) with a pure adhesive failure. For all treated specimens a substrate failure occurs at about F=2400N that corresponds to $\tau \approx 8$ MPa. Therefore it simply could be concluded that the sandblasting treatment enhances the bonding resistance.

The results obtained for composites (CF) and (GF) reinforced PEEK are presented Fig.7. Two projection times (5s, 45s) were selected to show the effect of average particle sizes on the lap-shear strength.

It can be noticed that, for composites CF and GF reinforced PEEK, the sandblasting treatment always induced a strong increase of the bond joint strength compared to the untreated materials. These changes are defined by the creation of peaks and valleys which can be considered as sites of mechanical interlocking between substrates and adhesive. On the other hand, the mechanical interlocking adhesion is highly dependent on surface morphological properties created after treatment.

In fact, the CF reinforced PEEK shows a higher joint bond strength than the GF reinforced PEEK for all particle sizes. For the both composites the sites promoting a mechanical interlocking are created but the surface morphologies are different. Moreover, the evolution of lap shear joint strength for the composites displays the same behavior. The increase of summits density enhances considerably the lap shear strength (Fig.7).

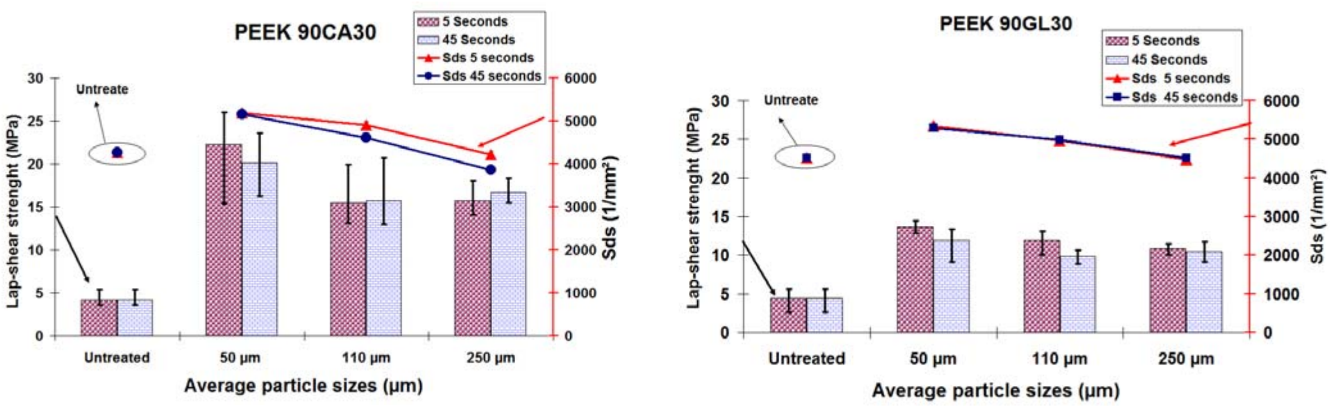

Fig.7. Lap-shear strength and Sds evolution as a function of average particle sizes and projection time (5s and 45s); (a) PEEK 90CA30 and (b) PEEK 90GL30

This is due to the distance reduction between peaks in the case of a highest summit density which increases the adhesion between the adhesive and the cavity created from a peak to another. On the other hand, the adhesive have a tendency to wet well the real surface area and go through in to the cavities which promote the mechanical interlocking and enhance the adhesive bond shear in the case CF reinforced PEEK. These assumptions are confirmed by the wettability behavior of both composite (Fig 6). The other factor that can influence considerably the adhesion strength is the nature of fiber. In the case of reinforcement by carbon fibers the strength adhesion is clearly better than in the case of reinforcement by glass fibers.

4. Conclusion

The effect of surface morphology created by sandblasting on the wetting and bond strength properties of neat matrix PEEK 90G, carbon fiber reinforced PEEK 90CA30 and glass fiber reinforced PEEK 90GL30 was investigated.
It was observed that after 5 seconds of sandblasting the roughness parameters has a tendency to stabilize whatever the particles size used. The 3D Sds parameter was chosen to describe the surface morphology. Presence of fibers has a significant impact in the result of sandblasting process. In fact, it was observed that after sandblasting for the same treatment conditions the composites CF reinforced PEEK and GF reinforced PEEK have a higher level of roughness than the matrix PEEK 90G.
Surface morphology has a significant influence in the wettability behavior for all materials. It was observed that the droplet contact angle increases with the average particle sizes. Furthermore, the nature of fiber has also an influence on the wettability behavior.
The mechanical interlocking plays a leading role in the enhancement of bond strength. However, the efficiency of this phenomenon is highly linked to the surface morphology. The suitable morphology can be defined as surface which have a maximum peaks until a level where the surface wetting remains satisfactory.

Acknowledgement

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