Fatigue life analysis of adhesively bonded CFR-PEEK composites using acoustic emission monitoring

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ABSTRACT. *In this paper, experimental fatigue behavior of adhesively bonded PEEK composite was investigated. PEEK based materials are considered as a high performance thermoplastic materials due to their semi-crystalline property giving them good mechanical properties and chemical resistance for many aggressive environments. However, it is well known that the adhesive bonding assembly of this kind of materials is difficult without an appropriate surface preparation. In the objective of enhancing the mechanical properties in cyclic fatigue of adhesively joined PEEK, sandblasting surface treatment was applied. The effect of sandblasting conditions on surface morphology was presented. Joint durability and fatigue life were expressed in terms of Wöhler curves according to surface preparation condition. Finally, an energetically approach and acoustic emission (AE) monitoring were used to analyze joint microstructural evolution during monotonic and cyclic long term loading.*

RÉSUMÉ. *Dans cet article, une étude expérimentale du comportement en fatigue des assemblages par collage de composites à matrice PEEK a été réalisée. Le PEEK est un matériau thermoplastique de hautes performances ; son caractère semi-cristallin lui confère une bonne résistance chimique à différents environnements agressifs. Cependant, l'assemblage par collage de ce type de matériaux est difficile sans une préparation de surface préalable. Dans l'objectif d'amélioration des propriétés mécaniques en fatigue cyclique du joint de colle, un traitement de surface par sablage a été appliqué. L'influence des conditions de traitement de surface sur la morphologie de surface est présentée. La durée de vie et la durabilité du joint de colle sont exprimées sous la forme de courbes de Wöhler. Une approche énergétique couplée à un suivi par émission acoustique (AE) a été utilisée afin d'analyser les évolutions microstructurales durant un chargement cyclique.*

KEYWORDS: *PEEK, sandblasting, surface morphology, adhesive bonding, fatigue, acoustic emission, dissipated energy, damage.*

MOTS-CLÉS: *PEEK, sablage, morphologie de surface, collage, fatigue, amission acoustique, énergie dissipée, endommagement.*

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1. Introduction

Nowadays, adhesive bonding technology appears to be an appropriate alternative for assembling composite parts. In fact, this method offers many advantages such as uniform stress distribution along the joint and minimizing stress concentration, joining dissimilar materials and weight reduction compared with other joining methods. High performance semi-crystalline thermoplastics (PolyEtherEtherKetone (PEEK)) reinforced fibers are increasingly used in aeronautical industry due to their high toughness and recyclability. The good chemical resistance of such thermoplastic composites makes their adhesive bonding resistance poor. Therefore, surface treatment before bonding is a primary step to achieve satisfactory joints (Shmidlin *et al.,* 2010).

Various surface treatments can be used to enhance adhesive bonding strength of CFR-PEEK composite, like physical treatment (laser and plasma), chemical treatment (chemical etching, functionalization) and mechanical (abrasion). The common surface result of all these techniques is surface morphology modifications. Low cost, environmental friendly and easy to implement technique makes sandblasting treatment (mechanical treatment) one of competitive methods for preparing thermoplastic material surfaces before adhesive bonding (Ivosevic *et al.,* 2016).

For many structural systems such as adhesive bonding, fatigue behavior is certainly an important type of loading to consider. Indeed cyclic loading fatigue may produce failure of adhesive-bonded assemblies at lower stress level than they can tolerate under monotonic loading. Hence, fatigue analysis and life time service prediction are highly required especially in the case of damage tolerance design of adhesive bonded systems based on PEEK composites for aeronautic applications. For this purpose two principal approaches are used: the first is based on the measurements of crack growth rate of pre-cracked adhesive joint subjected to mode I, II (Markolefas *et al.,* 2009, Datla *et al.,* 2010 and Khalili *et al.,* 2008) or mixed-mode fatigue loading. The second approach consists in determining, the stress versus the number of cycles to failure curves usually named Stress-Number of cycle (S-N) curves or Wöhler curves.

The effect of substrate surface state and nature on the fatigue performance of adhesive joints has been investigated by different authors (Gomatam *et al.,* 2005 and Shin *et al.,* 2006). It was shown that the relationship between surface texture and adhesion is very complex. In the purpose of optimizing abrasive surface treatment processes, different mechanisms and their interactions need to be understood (Harris *et al.,* 1999 and Ourahmoune *et al.,* 2014). In this paper, the effect of surface morphology generated by sandblasting process, on the long term behavior of adhesively bonded PEEK composite in single lap-joint specimen, under cyclic loading (fatigue) was studied. In-situ fatigue cycle and Acoustic Emission (AE) monitoring allowed determining fatigue life and damage states of bonded joint. The cross correlation between interface (substrate/adhesive) properties and the fatigue performance of joints was discussed.

2. Materials and methods

2.1. Materials

Continuous carbon fibers reinforced PEEK composites as substrate were investigated. These kinds of materials begin to be widely used in aeronautical industry.

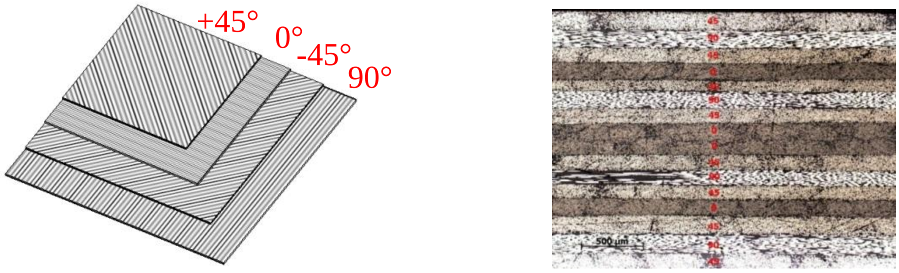


Figure 1. Schematic representation of PEEK composite with quasi-isotropic layup (a), and optical micrograph showing the stacking sequence of PEEK APC-2 (b).

The PEEK composite plate was made from 16 plies of unidirectional PEEK reinforced by high resistance carbon fibers (AS4) prepreg manufactured by Cytec® named APC-2 and was supplied by Airbus® France. The configuration of the laminates was quasi-isotropic and the stacking sequence is as follows: [+45/0/-45/90/+45/0/-45/90]s as shown in Figure 1. The PEEK matrix melt viscosity was approximately like the PEEK150G commercialized by Victrex®. It was characterized by middle molecular weight compared to other PEEK references, which facilitates fiber impregnation.

The structural adhesive used was a commercial epoxy based two parts adhesive (3M 9323-2 B/A) generally used in aeronautical applications. Curing cycle was as follows: 2 hours at 65°C, as recommended by the manufacturer.

2.2. Sandblasting treatment

The studied materials were subjected to dry sandblasting (particles projection), with varying experimental conditions (particle size, projection duration).

Schematic principle of sandblasting process is shown in Figure 2. The following results were obtained for subsequent fixed operating conditions: ceramic jet nozzle of 8 mm diameter, applied air pressure is about 5 bars, the distance L between the nozzle and target is 80 mm and the impact angle is 90°. The influence of process duration (5, 10, 20, 30 and 45s) and abrasive particle sizes (50, 110 and 250 µm) were examined.

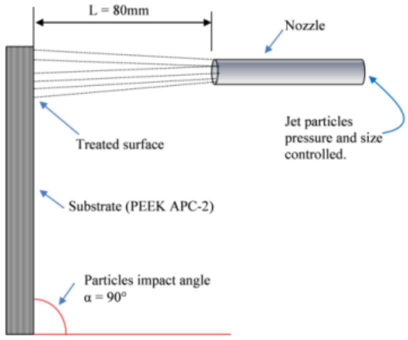


Figure 2. Schematic setup of sandblasting device.

2.3. Surface morphology analyses

The effect of sandblasting process on surface morphology was studied using two surface characterization techniques. SEM (Scanning Electron Microscopy) analysis was performed on the surface in order to point out morphological changes after sandblasting and interactions between particles and the composite. Surface roughness parameters (surface morphology) of untreated and treated surfaces were characterized by using a 3D optical white light interferometer (Mathia *et al.*, 2011).

2.4. Monotonic and fatigue testing of adhesive joints

In order to evaluate mechanical behavior of the adhesively bonded system (APC-2/Epoxy/APC-2), the single lap shear configuration test was chosen, due to its simplicity to implement and reproducibility. The single lap shear test is the most used method as comparative test in the case of adhesive bonding strength evaluation.

Specimens for single-lap shear test were prepared from two samples of PEEK APC-2 of 106 mm long, 25 mm wide and 2 mm thick bonded along 12.5 mm according to the international standard ISO 4587:2003 (cf. Figure 3). The overlap joint and the adhesive thickness (200 ± 20 µm) were carefully controlled after manufacturing. The monotonic tensile shear strength of the adhesively bonded single lap joints was measured 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) at a cross-head speed of 1 mm/min.

Tension-Tension load controlled fatigue tests were performed in the same metrological device. The frequency was maintained constant at 10 Hz for all tested configurations, and the fatigue loading ratio (R-ratio) was 0.1. However, if the failure did not occur before 10^6 cycles the test was stopped. The fatigue test criterion was assembly failure. The maximum stress level was varied from the monotonic maximum strength of the adhesively bonded single lap joints.

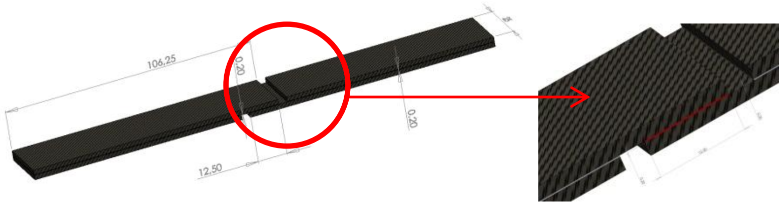


Figure 3. Schematic representation of adhesively bonded single lap shear specimen

2.5. Acoustic Emission (AE) device

Acoustic Emissions (AE) are the local dissipation of energy stored in form of elastic energy. Sources of acoustic emission can be associated to different phenomena like crack initiation and growth crack opening and closure. Therefore, (AE) is the mechanical or ultrasonic manifestation of micro-displacements in the internal structure of the material.

When the material is mechanically loaded, waves with various nature and frequency are propagated inside the material and reach finally the material surface. Surface vibration is collected by piezoelectric sensor and then amplified. Detection device of AE signal is composed with sensor coupled to the surface of material, amplifier and recorder. AE is an efficient technique for in-situ health monitoring of composite materials. This is achieved by monitoring the cumulative number of events and the analysis of AE parameters of the signal emitted by the signature waveform (hit) of internal events occurring in the material like the amplitude, the energy or the duration of the event (Figure 4).

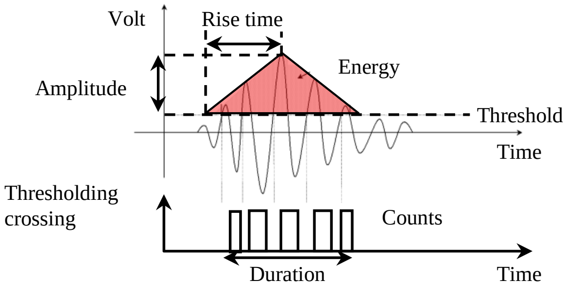


Figure 4. Schematic representation of AE wave

The amplitude of the signal is often used because it is independent on the detection threshold. Several authors (Barré *et al.*, 1994, Ceysson *et al.*, 1996, Flament *et al.*, 2016) have shown on different inorganic (glass or carbon) fibers reinforced composites that events with low amplitude (between 35–50 dB) are correlated with matrix microcracking, intermediate amplitude with delamination and microcracking coalescence (50-60dB) and matrix/fiber debonding (60-70 dB), high amplitude with fiber/matrix friction associated to pull-out (70-85 dB) and fiber breakages (>85 dB).

AE signals were acquired during monotonic and fatigue tests and processed with the AE acquisition and analyzing system Mistras 2001 from Physical Acoustics Corporation (PAC). The AE signals were detected by a piezoelectric sensor (R15) attached to the sample by silicon grease and a mechanical specific device on the sample. Two sensors were placed on the substrate on each side of the bonded joint. The signals were amplified by a pre and a post-amplifier with a total gain of 70dB. The detection threshold of AE was fixed at 35dB.

3. Results and discussions

3.1. Morphological analyses

APC-2 surfaces are characterized by a complex and anisotropic morphology as shown on SEM micrographs (Figure 5), due to the quasi-isotropic sequence stacking of the laminate, with the surface ply oriented at 45°, and the architectural morphology of the prepreg.

SEM analysis demonstrates that the sandblasting process modifies the surface morphology. However, this technique gives visual and qualitative information about surface morphological changes. Therefore and, in the aim to evaluate quantitatively surface morphological modification due to sandblasting conditions, interferometry technique was used. According to the 3D surface topographies the treated surfaces observes a complex morphology with an evident surface anisotropy following fibers orientation (Figure 6).

SEM and interferometry analyses showed that surface morphology obtained after sandblasting are complex and characterized by two distinct scales: waviness in macroscale and micro roughness in microscale. The complexity of the surface morphology is due to the architectural structure of the APC2. The ply of this composite is made from a successive horizontally assembled long fiber tow, which result on zones rich in fibers and zones poor in fibers. The material removal is easier in the area with low fiber density, which results in deeper valleys and therefore the waviness morphology. The micromorphology is due to particles impact.

In the aim to better understand surface morphological change and taking into account the surface anisotropic morphology (cf. Figure 6) and surface complexity a special surface morphological filtering is needed. In this case a Gaussian filter was applied to all surfaces allowed to separate the total surface in two distinct scales: macro-morphology describing surface waviness and micro-morphology describing surface micro-roughness, as shown in Figure 7.

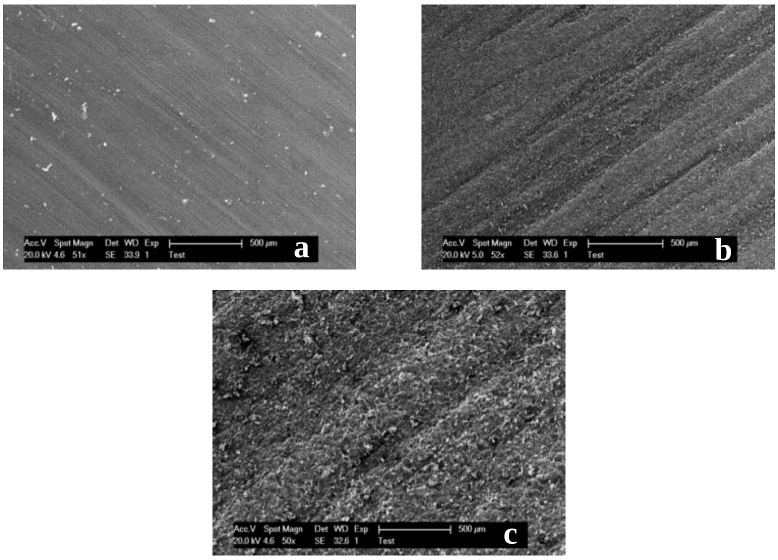
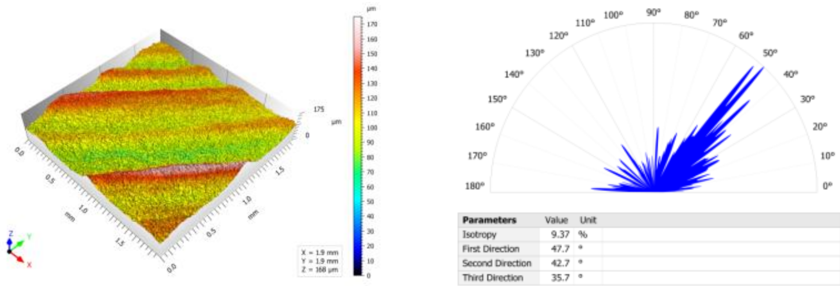


Figure 5. SEM micrographs showing APC-2 surfaces: untreated (a), sandblasted during 5 seconds and 50 µm particle sizes (b) and sandblasted during 10 seconds and 250 µm particle sizes (c)



a b

Figure 6. Surface morphology and relevant anisotropy

After surface morphological separation all morphological parameters were calculated separately for total surfaces, macro-morphology surfaces and micro-morphology surfaces. Only the pertinent parameters according to surface functionalization were taken into accountS_a average arithmetic surface heights, analogue to R_a in the case of 2D profile analysis which gives information about surface amplitudes, and S_{ds} summit density. In this case a peak is considered as a summit if it is higher than its 8 neighbour peaks and it gives information about surface asperities distribution.

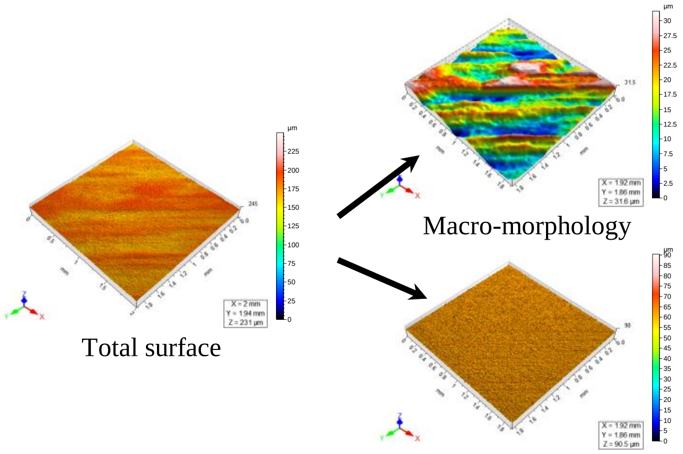


Figure 7. Surface morphological decomposition

However, if the S_a or S_{ds} are taken alone the given information is not sufficient. It is possible to have two morphologies with the same S_a but S_{ds} completely different. In this way a new morphological parameter was recently proposed by Ourahmoune *et al.* (2014) named S_r (eq. 1); this parameter takes into account height amplitudes and peaks density that can better describe surface morphology, and is defined as follow:

$$S_r = \sqrt{{S_a}^2 S_{ds}} \quad (1)$$

where S_a corresponds to the average surface heights [µm] and is analogue to R_a calculated from 2D profile roughness, and S_{ds} is the density of summits [1/mm²]. S_r was calculated for all surfaces: total surface, macromorphology and micromorphology. Figure 8 shows the evolution of S_r for all surfaces.

These figures show that in the case of total and macro surface morphology the S_r increases significantly with treatment duration for all average particle sizes. Beyond a treatment duration of 5 seconds there is no obvious correlation between the average particle size and surface morphology. However, in the case of micro-morphology S_r stabilizes after a few seconds but increases according to the higher size particles. This result demonstrates that the micromorphology gives the signature of the particle size used in the treatment.

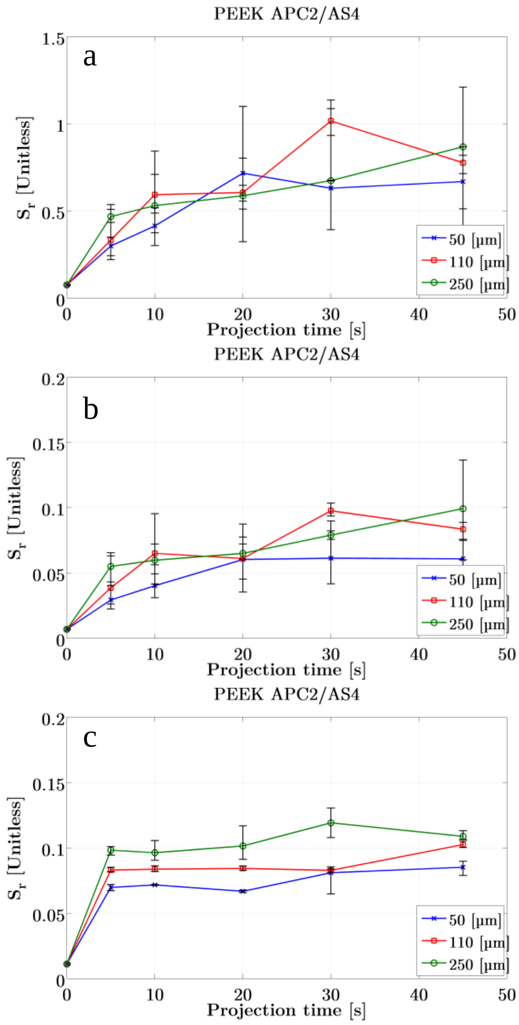


Figure 8. S_r evolution as a function of sandblasting conditions: a) total surface, b) macro-morphology and c) micro-morphology

3.2. Monotonic mechanical properties of adhesive joint

The shear strength is calculated using the following relationship:

$$\tau = \frac{F}{A} \tag{2}$$

where F (N) is the maximum load of the load -displacement curve and A (mm^2) is the overlap area. Due to the anisotropic morphology of surface, three configurations of first ply orientation according to loading direction were investigated (0, 45 and 90°). According to morphological analysis from micromorphology, treatment duration has no significant influence on surface morphology above 5 seconds, hence projection time was fixed at 5 seconds and only the average particle size was varied.

The results of shear strength are shown in Figure 9. The single lap shear strength corresponds to the arithmetic average of six tests. For treated substrate with outside ply at 90°, the values correspond to the shear strength in the joint at the failure of the substrate. Indeed, the single lap shear strength is very sensitive to substrate treatment. The average shear strength of the assembly without any treatment is below 15 MPa, whereas it increases up to 31 MPa after substrate sandblasting treatment. Nevertheless, the variation of average particle sizes has low influence on the shear strength.

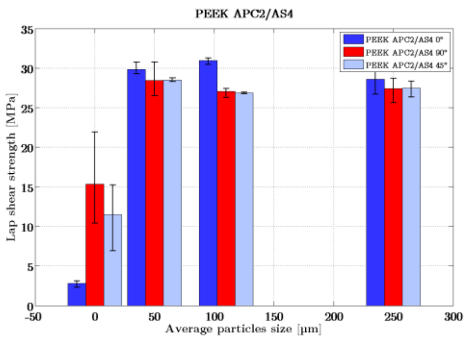


Figure 9. Monotonic average lap shear strength versus surface treatment for the different surface ply orientations

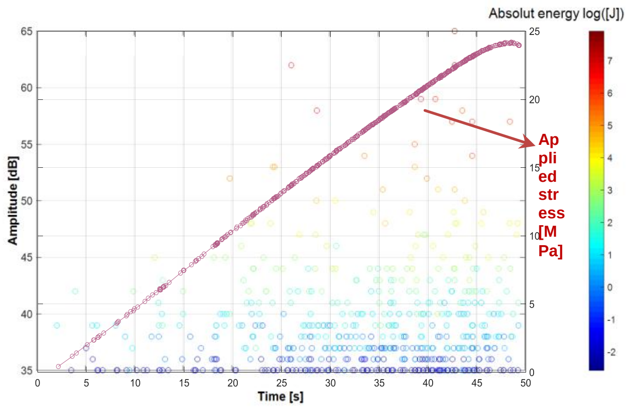


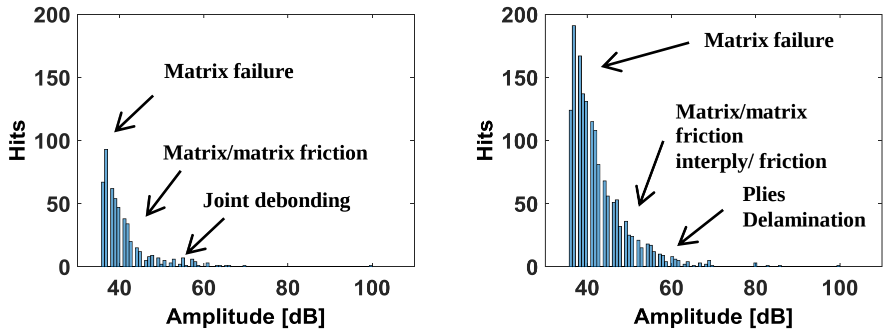
Figure 10. Acoustic emission monitoring during monotonic single lap shear test: Amplitude vs Time vs Absolute energy compared to applied stress vs time

The joints fail adhesively for untreated assembly and the failure surface features show a mixed failure mode in the case of treated substrates for surfaces oriented at 0 and 45°. However, in the case of surfaces oriented at 90° the failure takes place entirely within the substrate. Indeed, in this case, ply delamination between the first and second ply was observed for all sandblasting conditions studied.

This result is confirmed through acoustic emissions measurements. In fact, acoustic emission monitoring allowed distinguishing different damage events; liking matrix failure, matrix/matrix friction, delamination… The pertinent parameters were investigated as Amplitude and Absolute energy. Figure 10 show an example of acoustic emission activity during monotonic single lap shear test.

At a first approximation, it is possible for all the substrates to share the AE amplitudes into two distinct populations, the first one for the amplitudes ranging between 35 dB and 50 dB, and the second one between 50dB and 65 dB indicating different damage mechanisms as matrix cracking and delamination and substrate/joint debonding. Acoustic hits occur in much greater density for adherends with bonded ply oriented at 90° according to loading direction (Figure 11); this result confirms that damage mechanisms occur mainly within the substrate for this case in the form of matrix cracking followed by delamination within the composite. For the other substrates matrix breakages in the substrate yet occur but the total number of hits is much lower than for the previous case and the acoustic signals having higher amplitude is probably mainly linked to debonding at the interface joint.

The single lap test and morphological analysis results point out that the optimal sand blasting parameters are 5 seconds for projection duration and 50µm of average particle size. In fact, the increase of duration time or average particle size leads to fibers damage and therefore adhesive joint weakness.



a) b)

Figure 11. Amplitude of Acoustic Emission evolution as a function of cumulated hits, surface sandblasted during 5 seconds and 50µm of average particle sizes: a) surface oriented at 45° and b) surface oriented at 90°

3.3. Adhesive joint fatigue test

Loading levels were varied from about 30 to 70% of maximal failure stress. Six specimens were tested for each load level and surface treatment configuration. The monotonic behaviour of the adhesive joint showed that the surfaces oriented at 90° results in a ply delamination. In this case, it is not possible to have a correlation between surface morphology and adhesive joint durability. Therefore, only the surface orientation 0 and 45° were investigated. Sandblasting treatment for all the average particle sizes was applied to surfaces oriented at 45° (time = 5 seconds), and in the case of surfaces oriented at 0° only the best condition parameters were applied (5 seconds and 50µm average particle size), in the aim to compare the surface orientation influence.

S-N curves and morphology

Experimental fatigue data are represented as a plot of stress (S) as a function of the number of cycles to failure (N) or S-N curve also known as Wöhler curve. Figure12 gives the S-N curves for all the tested configurations.

The effect of substrate treatment on fatigue is more significant than on monotonic failure. The surfaces treated with an average particle size around 50 µm show the best behaviour. This trend is however attenuated as the mean stress decreases as shown by the convergence of the S-N curves at high cycles. Moreover, the results show larger scatter at high stress amplitude (low cycle fatigue) than at high cycle fatigue contrary to what is generally observed for bulk materials.

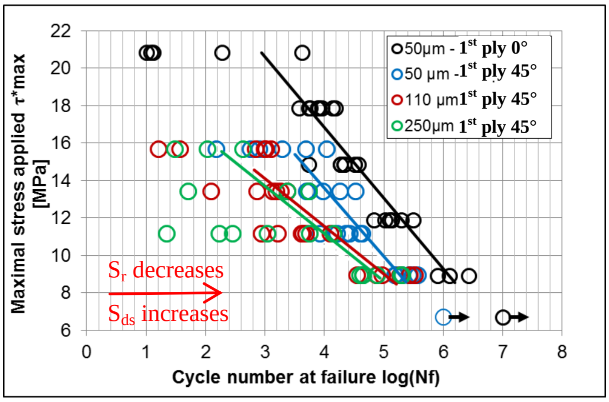


Figure 12. Wöhler curves as a function of average particle sizes

As for monotonic loading failure surfaces show a mixed mode failure, indicating high enough interfacial bonding. The functional peaks density (S_{ds}) on treated surfaces which is the highest in this case of treatment with average particle size of 50 µm is probably responsible of the best fatigue behaviour, the adhesive-substrate interface being mainly associated with mechanical interlocking. The different behaviour between monotonic and cyclic loading can be attributed to the fact that the fatigue crack growth likely took place by crack arrest and subsequent propagation for which the mechanical interlocking is a key parameter. Indeed, the first ply orientation has a considerable influence on the adhesive bonding durability. In fact assembly with bonded ply oriented at 0° present better long term behaviour. In this case fibres are oriented according to the load direction witch allow to enhance stiffness of the 1st ply in flexural behaviour.

In-situ analysis of load displacement curves

Fatigue behaviour of adhesive bonded joint was investigated by in-situ monitoring load-displacement data as a function of cycles (cf. Figure.13).

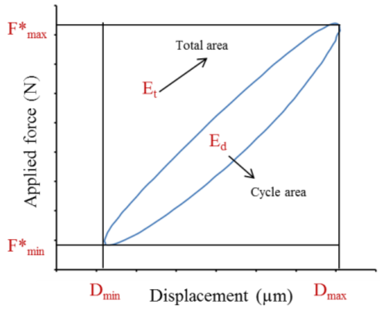


Figure 13. Fatigue cycle of adhesively bonded composites

From these cycles several physical data can be calculated (Lazan 1968):

– E_d: dissipated energy per cycle which can be quantified by the area inside the fatigue cycle.

– E_t: total energy, calculated using the following equation Et = $(F_{max}-F_{min})$ x $(D_{max}-D_{min})$.

– the system stiffness (R_s) [N/m], which is calculated by the general slope of the cycle and defined by the following equation:

$$R_s = \frac{F_{max} - F_{min}}{D_{max} - D_{min}} \quad (3)$$

Their variation may be related to a structural change at the microscopic scale in the substrate/adhesive junction or any of the components, as well as initiation and growth of microscopic cracks (Blanchard *et al.,* 1996).

Figure 14a gives an example of the changes in dissipated energy (E_d) per cycle as a function of the number of cycles until assembly failure at a loading level of about 9 MPa according to different surface morphology.

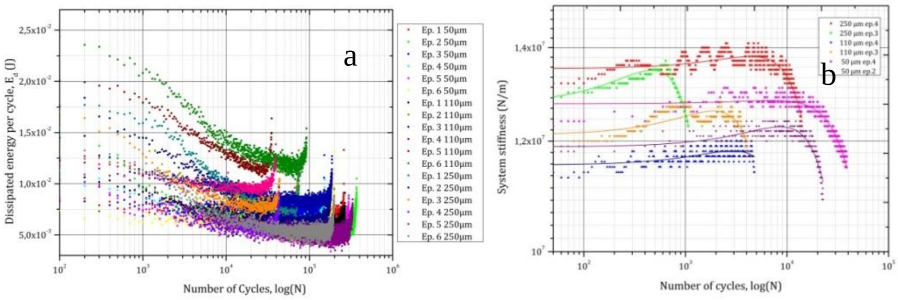


Figure 14. a) Dissipated energy evolution as a function of number of cycle (load = 9MPa) during fatigue tests. b) Evolution of the stiffness of the system during fatigue tests according to the number of cycles (load = 11MPa). The average particle size is varied from 50 to 250µm

The results show a decrease of the dissipated energy versus time until a minimum threshold where energy increases up to joint failure. This trend is observed for all stress levels studied and all surface treatments. The evolution of the stiffness against the number of cycles was plotted in Figure 14b in the case of maximum load of about 11 MPa and the three average particle sizes used. The stiffness increases slightly during fatigue up to a certain threshold where it abruptly drops up to sample failure. This result is consistent with the changes in dissipated energy.

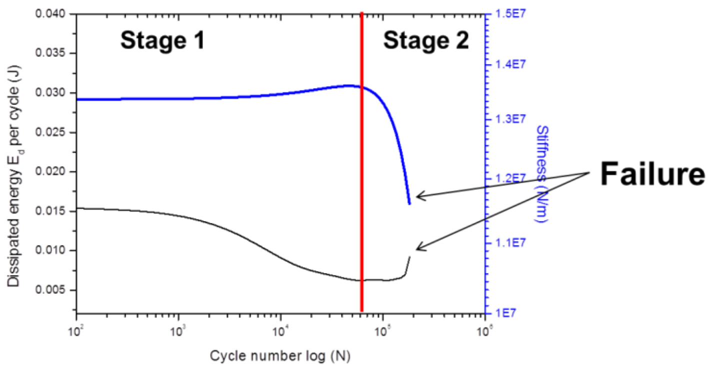


Figure 15. Fatigue stages of adhesive joint

From dissipated energy and stiffness changes of the system analysis during fatigue life, two stages of fatigue are observed (Figure 15):

• Stage I which corresponds to the drop of the dissipated energy and the increase in the stiffness of the assembly. The decrease of the dissipated energy during a fatigue test is probably associated to structural changes in the adhesive. Additional crosslinking was not considered as DSC analysis on epoxy adhesive before fatigue, shows that the adhesive is fully polymerized. This result leads us to consider the occurrence of sub-T_g structural relaxation phenomenon or physical aging observed in glasses (Perez 2001). This phenomenon will lead to compaction and densification of the network at a temperature close to the glass transition temperature, as well as changes in physical and mechanical properties. The changes in the molecular structure of epoxy adhesive may be responsible of the changes in assembly mechanical properties during fatigue test. This assumption was confirmed by the presence of an endothermic peak on a DSC thermogram of adhesive after fatigue test Figure 16 (Ourahmoune 2012). Moreover, due to loading unloading cycling the see-saw at the interface may induce complex phenomena.

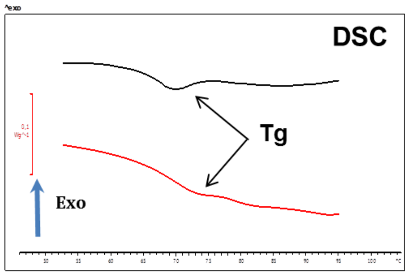


Figure 16. a) Dissipated energy evolution as a function of number of cycle (load = 9MPa) during fatigue tests. b) Evolution

• Stage II is associated to the increase in dissipated energy and a significant decrease in stiffness of the system (Figure 15). The plot of acoustic events cumulative according to time displays a sharp rise indicating a strong acoustic emission which coincides with the onset of the increase of the dissipated energy (Figure 18). Likely, this stage is linked with the high level of of micro-crack initiation and growth under the effect of the cyclic loading until assembly failure. This result was also confirmed by post-mortem SEM observation (Figure 19) showing the presence of high density of micro-cracks in the adhesive joint.

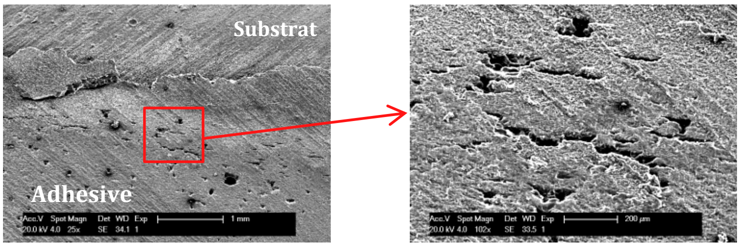


Figure 17. SEM micrographs showing adhesive joint after failure

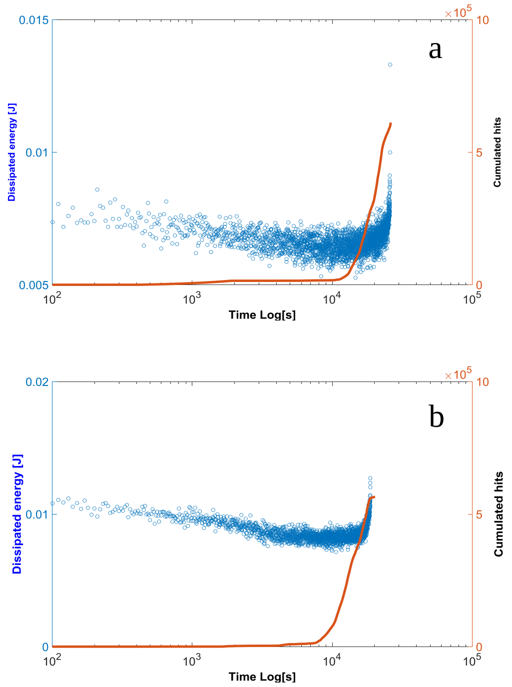


Figure 18. Dissipated energy versus acoustic emission (cumulated hits) during fatigue test. Surfaces oriented at 45°, maximal loading 40% and surface treated by a) 50 µm b) 250 µm of average particle sizes

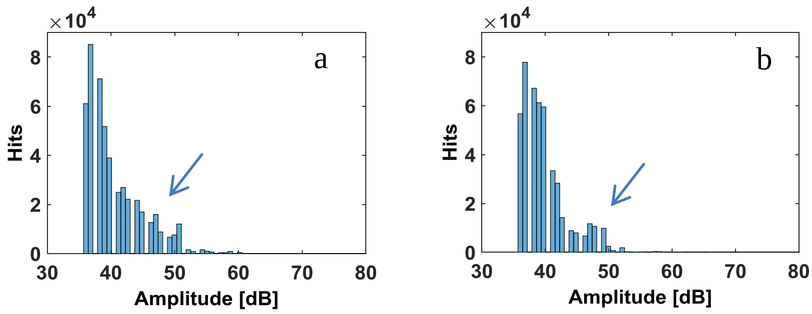


Figure 19. Amplitudes versus hits during fatigue test. Surfaces oriented at 45°, maximal loading 40% and surface treated by a) 50 µm b) 250 µm of average particle sizes

Moreover the analysis of the amplitude of the acoustic emission events (Figure 19) shows higher density over a range between 50dB and 60dB for the substrate treated with the smaller particles (50μm) suggesting progressive damage at the joint interface (debonding).

4. Conclusion

Fatigue behaviour of adhesively bonded PEEK composite was studied. The good chemical resistance of PEEK based materials makes their adhesive bonding resistance low, and surface treatment is essential to achieve structural joints. The effects of sandblasting surface treatment on lap shear strength were investigated in a first step. In a second stage in situ analysis of the joint behaviour during fatigue was examined using continuously monitoring of dissipated energy, stiffness and acoustic emission

The following conclusions can be drawn:

Using sandblasting surface treatments lead to improve significantly fatigue life of CFR (PEEK) adhesively bonded system especially for high stress level. A correlation between pertinent morphological parameters and adhesive bonding behaviour was found.

Monitoring fatigue cycle parameters lead to the identification of two successive stages in fatigue life of adhesively bonded joints: a first stage associated to the microstructural changes in the epoxy adhesive at the interface and a second stage associated to the propagation of macroscopic damage.

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