PROCESS-INDUCED INTERFACE AND INTERPHASE EXPERIMENTAL CHARACTERIZATION BY THERMOSET-THERMOPLASTIC COMPOSITE CO-CURING

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ABSTRACT

In this study, an aerospace-grade epoxy composite plate was co-curing welded by a unidirectional PEEK thermoplastic carbon fibre tape to develop advance composites joints. To account the surface roughness for the weldability of carbon epoxy/carbon PEEK composites, plasma treatments were performed. The co-curing was conducted by the following steps: each treated or non-treated thermoplastic tape was first placed in the mould and followed with six layers of dry woven carbon fabrics. The mould was sealed by vacuum bag and a bi-component thermoset (RTM6-2) impregnated the preform. To understand the role of curing kinetics, post-curing, curing temperature and dwell time on the quality of joints, five cure cycles were programmed. The strengths of welded joint were investigated by both interlayer peeling and single lap shear test. Furthermore, cross-sections of welded zones were assessed by scanning electron microscopy in terms of morphology of PEEK/epoxy interphase after co-curing. The preliminary results showed that cure cycle is an important controlling parameter for the crack propagation and degree of crystallization.

1 INTRODUCTION

There has been enormous attention from researchers and the aerospace industry alike for the use of thermoplastic composites (TPCs). This is primarily in part due to the advantage of reversibly bending or stretching, weldability and undergoing multiple mechanical deformations (post formability) as compared to thermoset counterparts. An example of this are the thousands of press-formed carbon fibre/polyetheretherketone (CF/PEEK) clips that were previously built into the A350 and Boeing 787 aircraft. The CF/PEEK are now attached to the CF/epoxy fuselage skin via mechanical fastening, which is well-established technique for aircraft metallic structures [1]. However, mechanical fastening for a composite joining is associated with stress concentration and requires an additional step of drilling hole through the fibre reinforcement composite for fastener installation. The weldability advantage of TPCs allows them to be welded or fusion bounded an efficient way and several techniques for the welding of thermoplastic composites have reached a high level of maturity [2]. A relevant question is how the existing techniques can be applied for the welding of dissimilar composite, i.e., TPC and thermoset composite (TSC) combinations.

In the last years, several welding strategies have been developed and examined for dissimilar composites. The first challenge is to improve thermoplastic-epoxy interfacial adhesion. One of the most common solutions to help the occurrence of interfacial adhesion using a thermoplastic interlayer which is compatible with the thermoset resin in TSC. Some examples of such interlayer thermoplastic resins are polyetherimide (PEI), polysulfone (PSU), and polyethersulfone (PES), which are in an amorphous nature[3]. In addition to the compatibility of the inter-layer thermoplastic resins with the thermoset matrix, a few other factors must also be considered to select the appropriate inter-layer thermoplastic resin: mechanical and environmental performance in applications, techniques used to incorporate the interlayers, and weldability of the interlayer thermoplastic resin without the thermoset degradation. To achieve the sufficient high strength bound without influencing the thermoset cure cycle, the chemical and physical properties of the interlayer and thermoset must be also considered. Moreover, the issues such as the resistances to aircraft solvents and moisture must be also addressed [3].

Alternatively, the surface of the TPC can be treated by the atmospheric plasma to generate a certain surface roughness that will cause mechanical interlock between the TSC and TPC during the welding process[4]. This solution does not require the interlayer and make the possibility of direct welding of a TPC on the treated surface of TSC. Understandably, some of the main points of concern in this solution are the weld strength and durability of the thermoplastic and thermoplastic connection. This study proposed a new technique to achieve high strength connection of the hybrid interface, by co-curing the unidirectional carbon fiber reinforced thermoplastic tape with the thermoset composite without the requirement of an interlayer thermoplastic. This enable us to directly test the interface by peeling test, without an additional welding process, like infrared welding, which could be responsible for thermal degradation of the TSC substrate.

2 EXPERIMENTS

2.1 Material and method

The selected thermoplastic tape is a 0.17×25.4 mm tape made of PEEK-150 with 55% AS4 carbon fibers/C in volume fraction, supplied by SUPREM. Plasma treatments were performed using an atmospheric pressure dielectric barrier discharge (DBD) reactor. Plasma discharge was generated by an AC power supply set at 450W and 6 kHz, with a flow of 80% N_2 and 20% O_2. A pressure-controlled FEI Quanta FEG 200 scanning electron microscope (SEM) from FEI Company was used to obtain information about the surface modification of the thermoplastic tape after treatment (Fig.1) and the microstructure in cross sections of the samples (Fig.2).

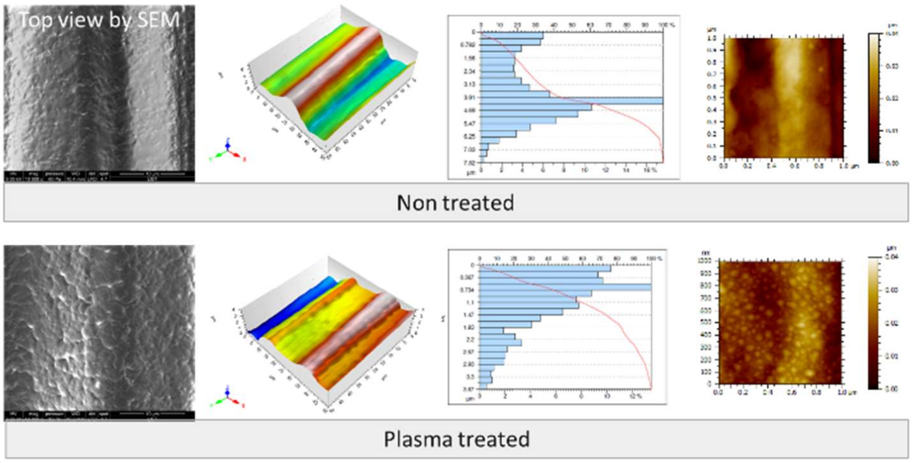

Figure 1: Top view by SEM (left), 3D topology (middle), AFM top view (right) of a PEEK/C tape as received and with a plasma treatment.

2.2 Co-curing process

The liquid resin infusion (LRI) process was used to co-cure the thermoplastic composite tape onto the TSC surface. Six treated and non-treated thermoplastic composite tape are placed in the mold, with a tape length of 350 mm, and width of 25.4 mm. One composite plate is injected per cure cycle conditions. The selected thermoset resin is RTM6–2, a bicomponent supplied by Hexcel. As showed in the Fig.2, no interphase is visible at the micro scale in case of PEEK, counter to PEI polymer presenting large interphase, from 20 to 40 µm thick [5].

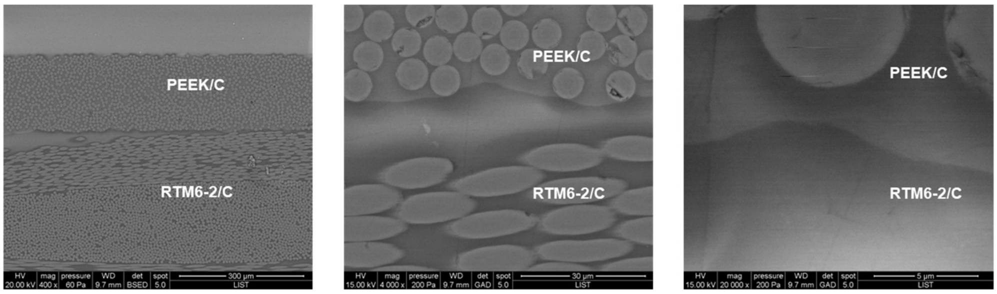

Figure 2: Hybrid join cross section obtained by SEM

step, 5 different cure cycles were proposed. It should be noted that the nontreated PEEK/C tape configuration presents a weak adhesion after demolding, which is why peeling tests weren't possible. The crystallization state of the PEEK/C, initially amorphous, evolve during the co-curing, with various kinetics as function of the cure cycle, to reach a full crystallize state for each curing conditions.

2.2 Mechanical testing

The 90° peel adhesion test is used to determine the force required to de-bond the two components joined by co-curing. The test result, like the bond strength, is represented as N (force to de-bond) / 25.4mm (tape width). The test speed is 300 mm/min. The free moving table is attached to crosshead using a pulley and rope mechanism, such that the traverse movement of table is at the same rate as the crosshead movement, thus maintaining a constant 90° angle between the two components.

2.2 Interphase investigation

One of the main challenges by diffusion-based joining is the characterization and control of the interphase, bringing a physical view of the joint quality. This represent a strong issue as the potential interphase is certainly thinner than the micron scale (Fig.3).

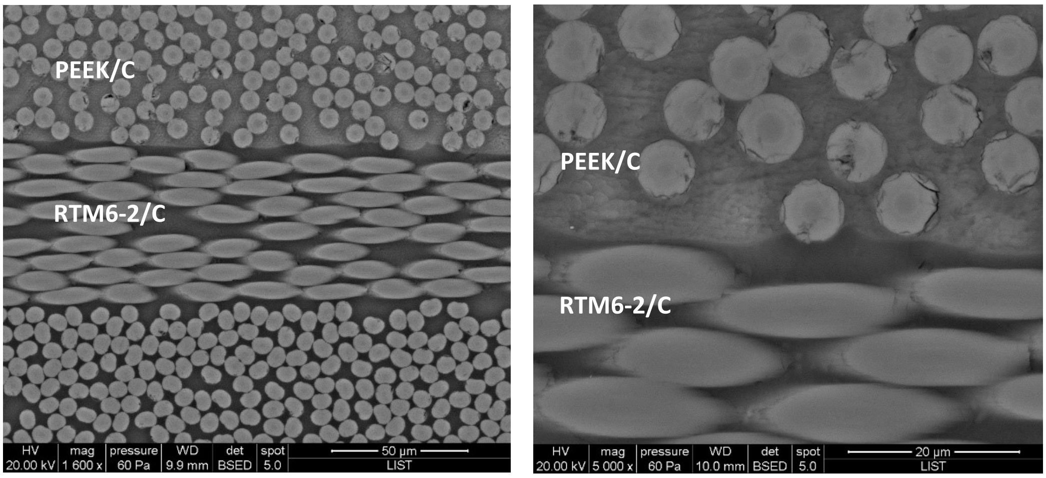

Figure 3: PEEK/C & RTM6-2/C interphase characterization by SEM after chemical etching

3 RESULTS AND DISCUSSIONS

The welded joint performances were investigated by both interlayer peeling and single lap shear test on hybrid joint. Diffusion time of the thermoset precursor into the thermoplastic have a major influence on adhesion, inducing respectively a very poor to a strong adhesion. Failure analysis by SEM were performed and contribute to increase understanding of adhesion induced by diffusion. To increase the understanding of the diffusion driven parameters, a dedicated study was performed, based on the co-curing of the thermoset composite laminate onto a plasma treated thermoplastic unidirectional tape. 90° peeling test were performed as function of several cure kinetics both as co-cured and after severe hydrothermal ageing conditions. As expected, cure kinetics are a key parameter. Observed failure mode are multiple: from adhesive failure, cohesive failure at the co-cured interface, up to the thermoplastic tape failure in one cure kinetics condition. Such results open promising perspectives enabling the assembly by fusion or co-curing of hybrid structure. Driven physical mechanisms responsible for the submicron interdiffusion must be further investigated. The interphase experimental investigation is still on-going, by reproducing the diffusion phenomena without carbon fiber, enabling a more appropriate sample preparation for AFM sub-micron observations.

4 CONCLUSIONS

This paper presents a new technique through the co-cure bonding of dissimilar carbon/epoxy (C/Ep) and carbon poly-ether-ether-ketone (C/PEEK) composites. To improve the mechanical interlocking, C/PEEK tap surface was treated through an atmospheric plasma treatment. The main features of the presented procedure are: (i) One step co-cure bonding without degrading the thermoset counterparts, (ii) such process of welding is simple without the requirement of an interlayer thermoplastic. (iii) Acceptable treatment durability of PEEK tape after 7 days. (iv) high interface quality without visible microscale interphase. The main conclusions from this research study are:

- The mechanical performance is not affected by the ageing.
- Curing cycle is very important controlling parameter and hence crack initiation is highly influential by the thermal curing even below T_g of PEEK
- The thermoplastic composite join, made of PEEK/C TPC tape and a PEEK/C laminate present a high initiation crack force, *c.*43 N followed by an average peeling force of 27 N with a highly instable crack propagation.

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