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Manufacturing Process of S-Glass Fiber Reinforced PEKK Prepregs

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***Abstract*—**The aim of this study is to investigate the fundamental science/technology related to novel S-glass fiber reinforced poly-ether-ketone-ketone (GF/PEKK) composites and to gain insight into bonding strength and failure mechanisms. Different manufacturing techniques to make this high-temperature pre-impregnated composite (prepreg) were conducted i.e. mechanical deposition, electrostatic powder deposition, and dry powder prepregging techniques. Generally, the results of this investigation showed that it was difficult to control the distribution of the resin powder evenly on the both sides of the fibers within a specific percentage. Most successful approach was by using a dry powder prepregging where the fibers were coated evenly with an adhesive that served as a temporary binder to hold the resin powder in place onto the glass fiber fabric.

***Keywords*—**Dry powder technique, PEKK, S-glass, thermoplastic prepreg.

I. Introduction

THE future aircraft, such as Airbus A350XWB and Boeing 787, will likely use far more than 50% (by weight) advanced composite components. Thus, the manufacturing process of these composites needs to be improved to satisfy the future requirements of the new generation of aircraft. Reinforced composites made with thermoplastics (TP) as matrix materials in conjunction with glass fiber (GF), carbon fiber (CF), natural fibers (NF) have increasingly found their uses in the aerospace, automotive and renewable energy sectors due to their nature properties such as light-weight construction potential, integral design and good impact properties [1]-[5]. These advanced materials were introduced in the form of prepregs (pre-impregnated fiber or fabric in a flat form). Prepregs have been used extensively due to their low cost, ease of processing, consistent quality and higher volume capability. The mechanical properties of prepregs over a wide range of temperature are better than those from wet layups, as the resin is applied in uniform and exact quantities and an optimum fiber/resin ratio is attained. In addition, prepregs reduce the health and safety risk associated with handling resin. Thermosetting (TS) prepregs such as epoxy prepregs are used in a wide variety of applications such as automotive, construction and aerospace industries. However, these prepregs have limited shelf life due to their cross-linking degree in B-stage. These materials have to be kept in freezers at around -18°C [6]-[9] to prevent the cross-linking polymerization reaction taking place at ambient temperature.

The major problems in the aerospace applications where FMLs are extensively employed for fuselage, currently utilizes thermoset epoxy based prepregs. Low viscosity, low fabrication temperature and good resin/fiber wettability are the major reasons for use the epoxy-based composites. However, these kinds of prepregs exhibit poor hot/wet stability, high cost of manufacture, low combustibility leading to fire at elevated temperatures, generating smoke and toxic fumes which pose a serious health hazards [10]-[14].

Thermoplastic matrix is good alternative for advanced structural composite system due to their substantial advantages over thermoset matrixes, such as improved fracture toughness, fire/smoke resistance, high hot/wet stability, recyclability and rapid processing due to shortened curing cycles. No specicila facilities are nedded to store thermoplastic prepregs and it can can be kept at room temperature with infinite shelf life [4]. Thermoplastic matrix systems also present a stimulating and promising opportunity for the automotive sector and have a potential to bridge between the lightweight and reprocessable high strength composite materials in mass production [15].

The most attractive feature of fiber reinforced thermoplastic matrixes is their ability to welding at elevated temperatures thus, making them suitable for high speed production such as thermoform and allowing the applications of novel joining techniques such as ultrasonic and induction welding [16]. However, many challenges still remain in these materials that need to be solved to realize their ultimate potential. Despite the several advantages offered by the traditional thermoplastic matrixes, their usage has been limited due to their low moduli, poor chemical resistance, low glass transition temperature and low thermal stability at elevated temperatures [17].

The development of multi-functional thermoplastic matrix based on aromatic polymer has an ability to address all these limitations. The use of matrix materials, such as Poly-ether-ketone-ketone PEKK, and ply-ether-ether-ketone (PEEK), have

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demonstrated excellent mechanical barrier, exceptional impact resistance, vibration damping and thermal properties at high temperatures, especially when reinforced with high performance fibers [18]. As high temperature ─ high performance (HTHP) thermoplastics, PEKK and poly-ether-ether-ketone (PEEK) [19], [20] are considered to be the most favorite matrix systems in the aerospace industry to satisfy the need of light-weight, low cost primary load bearing structures and high temperature FMLs [21]-[23].

The impregnation of thermoplastic matrix is crucial due to their higher viscosity and processing temperature, resulting poor composite properties. Normally, thermoplastic resin is combined with fibers to make prepregs throughout a variety of manufacturing processes, which can be divided into the following categories, i.e. (1) solution dip prepregging, (2) hot melt prepregging, (3) film calendaring, (4) dry powders, and (5) aqueous suspensions. Each process has its own requirements and process variables that affect the quality of the finished product. In the solution dip prepregging, the solution is obtained by dissolve the thermoplastic resin with solvent in order to reduce the viscosity enough to allow impregnation of the polymer. Although high pressure facilities are not required in this technique, the relatively high solvent resistance of advanced composites makes it difficult to remove toxic and organic solvents with high boiling points from the composite during consolidation [24], [25]. In the hot melt method, the fiber bundles are impregnated with molten polymer. Once a molten thermoplastic polymer is provided by an extruder to a melt pool, the fiber bundle is pulled into the molten over number of spreader pins [26].

High melt viscosities are the main problem of the hot melt impregnation which leads sometimes to fiber breakage [24]. The film stacking is a standard technology for producing thermoplastic composites for a long time, in which different layers of reinforcing fibers are laminated between layers of thermoplastic polymer film by applying heat and pressure. The pressure needs to be sufficient to force the polymeric melt to flow into the reinforcement. Although the difficulty of forcing a highly viscous resin through the gaps between fibers that are normally in micron is overcome by applying pressure, this also forces the fibers together, packing them in such a way as to make infiltration by the resin more difficult [27], [28]. The difference between these methods is the way of matrix particle deposition on the fibers and the bonding force between particle and fiber, which is responsible for their adhesion to each other [29]. Several advantages can be obtained using the dry powder prepregging including wide range of high melt viscosities used with elimination of the solvent removal or hot melt problems, thus providing an increase in polymer selection [30].

Due to the advantages of dry powder prepregging technique over other methods, this method is utilized in the current work. PEKK is one of the future polymers that can be used for structural applications in high temperature environment. In addition, the processing temperature of PEKK is lower than PEEK and its mechanical properties are higher than those of PPS and PEI [3]. Therefore, a novel high temperature ─ high performance glass fiber reinforced thermoplastic prepreg was manufactured using a dry powder technique. Here, S glass woven which is known for its high strength and impact resistance properties is used as a reinforcement and PEKK as matrix. From the review, it can be seen that there is a limited data for the S-glass fiber reinforced PEKK, and these is a lack of information to manufacture and evaluate these materials under various test conditions. Therefore, the primary aim of this work is to investigate the fundamental science/technology related to novel GF/PEKK composites and to provide experimental data for numerical modelling validation and further for assisting design prepreg materials.

II. Experimental Procedure

A. Poly-Ether-Ketone-Ketone Thermoplastic Resin (PEKK)

Poly-ether-ketone-ketone (PEKK) is a high temperature high performance thermoplastic material that belong to the poly aryl ether ketones (PEAKs) family in which family members are different from each other according to the ratio of ketone-ether groups. These resins are semi-crystalline aromatic which exhibit low melt viscosity, excellent thermal stability, low moisture absorption, high toughness and tensile modulus, good chemical resistance and good flammability resistance. The glass transition temperature (Tg) of PEKK polymer is 165 °C. In this research, PEKK resin was provided by ARKEMA France in a powdered form with particle size of 50 microns. The product is marketed by the trade name of Kepstan.

B. Reinforcement

S- Glass fibers have high tensile and compressive strengths, high temperature resistance, and good impact resistance. S-Glass woven type (124 gsm (4oz) plain weave 30 Aerialite was used in the present work. The S glass fibers were obtained from East Coast fiberglass Supplies (UK).

C. Manufacturing of S-Glass Fiber Reinforced PEKK Thermoplastic Composite

The first part considers the preliminary manufacturing processes have been undertaken to make a thermoplastic prepreg of poly-ether-ketone-ketone (PEKK) reinforced with S-glass fiber using a prototype lab scale prepregging equipment which has been developed in the school of engineering, center for material and structures laboratory at University of Liverpool as shown in Fig. 1.

D. Dry Powder Prepregging Technique

In dry powder prepregging, the resin powder is deposited directly on the woven glass fiber. The resin powder can be applied on the fibers in different techniques as following;

1. Mechanical Technique

Fig. 2 shows the setup of this technique in which PEKK powder is deposited on woven glass fiber using metal bars. These bars were designed to have depths with different dimensions in order to control the powder proportion on the fibers. The advantage of this technique is that uniform powder distribution can be obtained by using a proper depth. However, it was too difficult to coat the rear fiber face. Moreover, the penetration of the PEKK resin within the fibers caused a severe damage in the fiber as a result of the high viscosity of PEKK resin, as shown in Fig. 3.

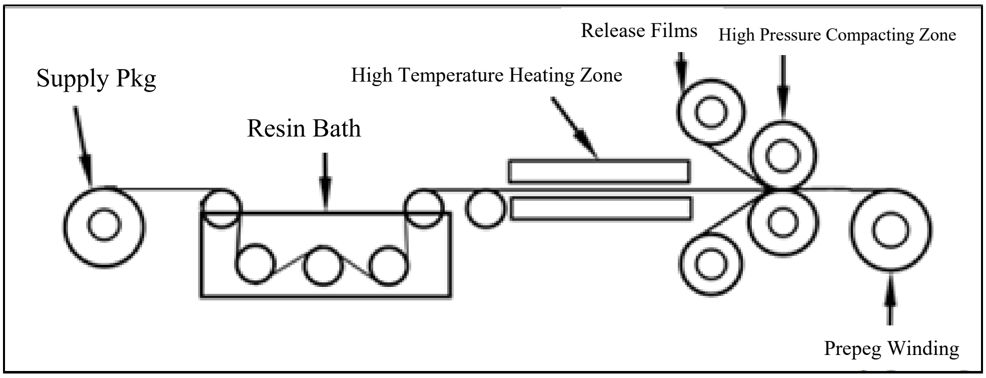

Fig. 1 Thermoplastic prepregging rig

Fig. 2 Powder deposition setup

Fig. 3 Fiber damage

2. Electrostatic Powder Deposition

An electrostatic power deposition technique is also utilised in this project as a developed powder impregnation process to charge and deposit PEKK powder onto a woven S-glass. In this process PEKK powder is charged by using high voltage source up to (30 KV) in order to deposit on grounded part which is woven glass fibre tape. Preliminary work has been done to make the prepregs as shown in Fig. 4. The results showed that the deposition of PEKK powder on woven s-glass fibre tape was not sufficient and the amount of deposited powder was not enough to give prepreg with good quality.

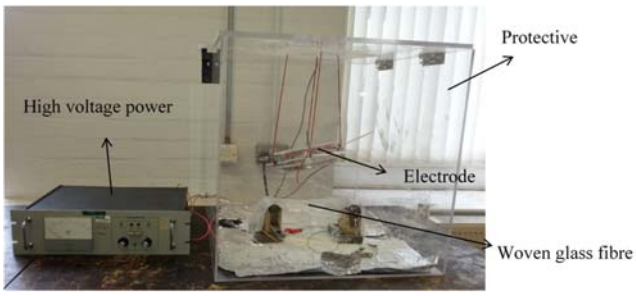

Fig. 4 Electrostatic setup

Therefore, the prepregs of the PEKK resin and woven s-glass fiber were made manually as an alternative approach.

E. Sample Preparation

The manufacturing process of S-glass fiber reinforced PEKK resin can be divide into two parts. The first one was to manufacture the prepregs, after that these prepregs were stacked together to make the composite panels.

1. Prepregs Manufacturing Process

Here, a dry powder prepregging technique was utilized to make a prepreg of PEKK and woven S-glass fiber, in which dry powder of PEKK resin was deposited onto the fibers as shown in Fig. 5. The manufacturing processes of the GF/PEKK prepreg are listed in the following steps:

- The woven S-glass fiber was cut into square pieces (250 mm x 250 mm). Then, the glass fiber was weighed using a high-resolution scale.
- Adhesive of 3M Multipurpose Spray was sprayed evenly on the fiber and the weight of the fiber plus the adhesive was recorded. Here, the adhesive works as a temporary binder to hold the resin powder on the glass fiber.

- The fibers were then dipped inside a powder tank, and the weight was recorded at each dipping time until the target percentage was obtained.
- The glass fiber with desired amount of powder attached was placed between two molds (300 mm x 300 mm) and heated to 330 °C inside the hot press. A high temperature release agent (Frekote) was used between the mold and the prepreg to ensure easy removal after the consolidation. The processing cycles in terms of holding time and pressure to make these prepregs were 10 min and 6 bar, respectively.

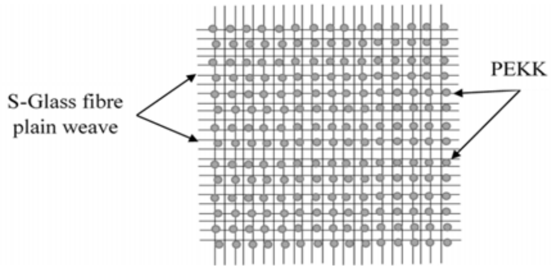

Fig. 5 Schematic diagram showing the deposition of the thermoplastic resin (PEKK) on the plain weave S-Glass fiber

F. Preparation of Composite Laminates

In this part, the composite laminates were manufactured by stacking an appropriate number of approximately 0.125 mm thick ply in a mold. Then, the resulting stack was heated to a temperature of 330 °C at an approximate heating rate of 5 °C per minute. The laminates with dimensions (125*125 mm) were cured under a pressure of 3 bars for 30 minutes prior to cooling at rate of 2 °C/ minute. The processing parameters of the composite laminates are illustrated in Fig. 6. After cooling, the pressure was released and the laminates were removed from the mold and inspected for defects.

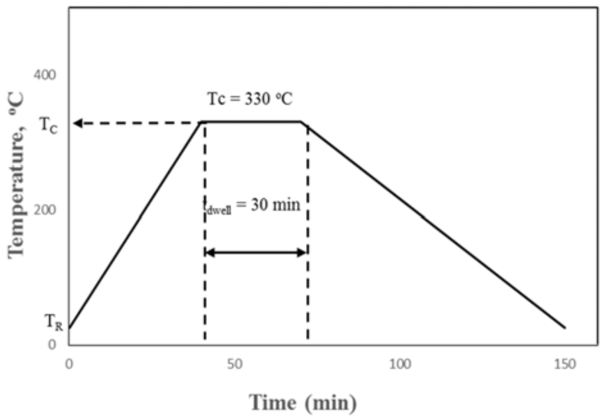

Fig. 6 Curing process curve and stacking configuration of GF/PEKK

III. Result and Discussion

A. The Effect of the Binder between the Thermoplastic Powder and the Fiber on the Perforation Resistance of the Laminated Composites

The investigation was initially focused on the influence of the binder between the thermoplastic powder and the fibers on the mechanical properties of the GF/ PEKK laminates. The investigation based on making four ply composite panels with and without binder. Fig. 6 shows load-displacement traces following quasi-static perforation tests on panels with and without the binder. An examination of the figure indicates that both panels exhibit the similar trend at the beginning. However, the critical force value of the latter laminated panels is much higher than the former panels, i.e. more than twice. It could be argued that the higher value of the critical force for the treated panels is due to the better adhesion between the resin powder and glass fibers, which leads more PEKK power attached to fibers with more uniform distribution.

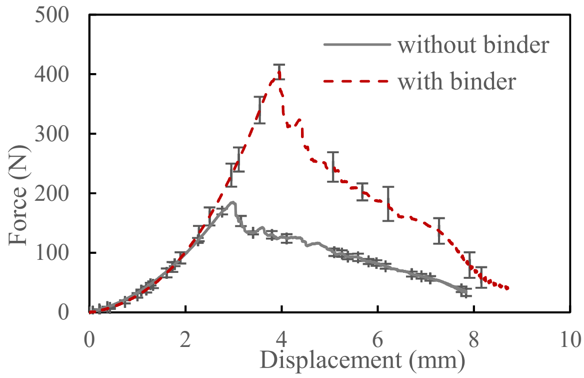

Fig. 7 Load-displacement traces following perforation tests on the 4-ply PEKK/GF composite with and without adhesive applied to the fibers

B. The Effect of the Binder between the Thermoplastic Powder and the Fiber on the Tensile Properties of the Laminated Composites

Further investigation was undertaken to assess the influence of the fiber treatment with the binder spraying on the tensile strength of GF/PEKK samples. The effect of the fiber treatment on the tensile properties of GF/PEKK laminates with a fixed thickness of 0.47 mm (4 ply) is illustrated in Fig. 7. As noted previously, the treated laminates have over twice tensile strength of the untreated ones. To summarize that, the mechanical properties of treated laminates with binder was higher than those untreated ones due the powder distribution associated with good bonding strength of the treated laminates.

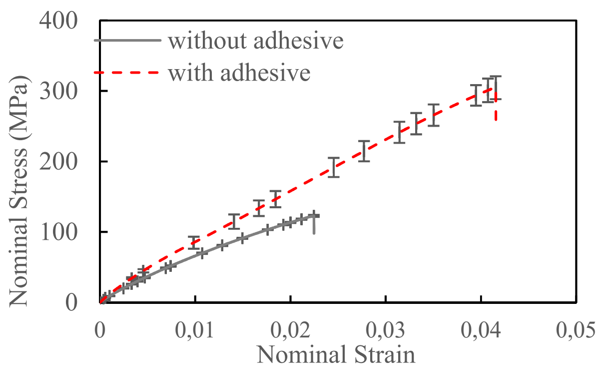

Fig. 8 Nominal stress-nominal strain curves for the 4 ply PEKK/GF composites (with and without fiber adhesive treatment)

IV. Conclusion

The possibility of making a novel prepreg of woven s-glass fiber as reinforcement and poly-ether-ketone-ketone as matrix have been investigated. The results of the preliminary manufacturing processes have shown that more experimental works are needed to improve the depositions of the thermoplastic powder resin on the fiber.

The results of the mechanical properties such as tensile tests and quasi-static perforation tests on the result prepregs have shown that the binder between the fiber and the power resin play a vital role in improving the quality of the prepregs.

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