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Processing of CF/PEEK thermoplastic composites from flexible preforms

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Abstract—Commingled yarns and powder/sheath fibre bundles are two flexible thermoplastic composite preforms, consisting of a blended combination of reinforcing fibre yarn (e.g. carbon fibres) and a yarn spun from a thermoplastic resin or a polymer resin powder respectively. These preforms can be used as basic filament yarns that can be interwoven, braided or knitted for making two- or three-dimensional composite parts. Commingled yarns and powder/sheath fibre bundles retain the flexibility of a tow such that when a fabric is woven from these materials, the fabric can be designed to be highly conformable and drapeable. When heat and pressure are then applied, the thermoplastic yarn or powder/sheath melts, thus wetting the reinforcing fibres and forming a thermoplastic resin binder. In the subsequent cooling step, the system is transformed into a rigid composite material. In this paper, major achievements in the study of the fundamental mechanisms which govern the impregnation process, the consolidation quality and the resulting mechanical properties of CF/PEEK commingled yarn and powder/sheath fibre bundle composites are highlighted.

Keywords: CF/PEEK composites; commingled yarns; powder/sheath fibre bundles; consolidation; processing variables.

1. INTRODUCTION

The argument for the advantages of thermoplastic composites lies in their potential for rapid, low-cost mass production. During the last decade, great efforts have been made to produce innovative/cost-effective preforms for manufacturing fibre-reinforced thermoplastic composites. Commingled/hybrid yarns or powder/sheath fibre bundles and their fabrics, as illustrated in Fig. 1, representing some kind of a 'dry impregnation' have been developed and technologically demonstrated for various applications [1–7]. In both cases, the fibre yarn/bundle consists of a blended combination of reinforcing fibres and thermoplastic filaments or powder from the bulk polymer matrix. In

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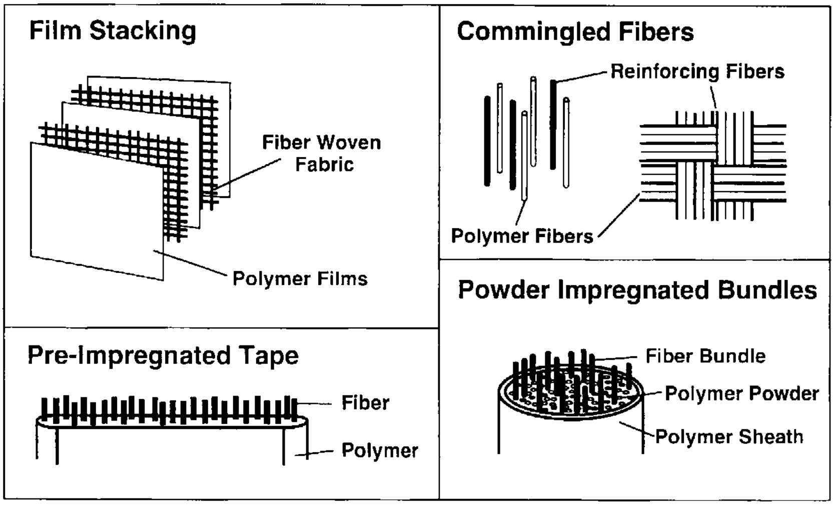

Figure 1. Innovative/cost-effective preforms for thermoplastic composites.

the commingled state, the multi-filament fibres are scattered amongst one another at the filament level; while in powder/sheath fibre bundles, the individual rovings of reinforcing fibres were mingled with very fine thermoplastic matrix powder and then bound together by a thin jacket (sheath) of the same polymer matrix. Both preforms retain the high flexibility such that when a fabric is woven from these materials, the fabric can be designed to be highly conformable and drapeable. When heat and pressure are applied, the polymer filaments or the polymer powder/sheath melt, thus wetting the reinforcing fibres and forming an amorphous resin binder. In the subsequent cooling step, the whole system is transformed into a rigid thermoplastic composite.

Nevertheless, up to now relatively little knowledge exists about the impregnation and consolidation behaviour during processing of thermoplastic composites from commingled yarn or powder/sheath fibre bundle preforms. This is the more surprising because some problems have been identified during the technological manufacturing steps of these material forms. For example, it was observed during filament winding of GF/PP commingled yarns that PP filaments in the tow can get separated from the glass filaments when tension is applied [1, 8]. In fact, the polymer fibres and the reinforcing fibres in commingled yarns can become unmingled when non-uniform tension is applied, because of the mismatch of stiffness in that direction [9]. This may result in both non-uniform distribution of fibres in final composite parts and insufficient impregnation of fibres, resulting in poor load transfer between them.

The present study is intended to provide a deeper insight into the impregnation and consolidation behaviour of these flexible preforms. Impregnation mechanisms and consolidation quality of unidirectional CF/PEEK composites were investigated using a small compression mould with a laboratory hot press at different processing conditions (i.e. applied pressure, holding time and processing temperature). Based on the investigations on impregnation mechanisms, impregnation models for qualitatively describing the consolidation processes of commingled yarns and powder/sheath fibre bundles were developed. The models simulate variations of void content during consolidation as well as the time, temperature and pressure required to reach full consolidation.

2. MATERIALS AND PROCESSING

The CF/PEEK commingled yarn was supplied by BASF, Germany, which consists of a 60 : 40 wt-% mixture of carbon (AS4, Hercules) and PEEK fibres (150G, 25–40 μm in diameter). The 'as-received' commingled yarn was applied by a proper 'sizing' to prevent the unmingling process of the fibre yarns due to electric charging or non-uniform stretching when the fibre yarn was wound onto the bobbin. AS4 (3K and 6K)-CF/PEEK (150G) powder/sheath-fibre bundles were supplied by Enichem, Italy. Before compression moulding of composites, 'flexible' unidirectional mats were produced from the 'as received' filament yarns/bundles by winding the individual yarns/bundles onto an aluminium plate. Subsequently, the aligned yarn/bundle mats were welded together with a soldering iron along two transverse lines at both ends of the mats before cutting, as described in [8]. A number of layers of unidirectional 'prepreg' mats were packed onto each other in order to reach an estimated thickness of about 3 mm of the composites after full consolidation. The latter process was carried out using a laboratory hot press with a small steel mould having a square cavity. Once the mould reached a desired processing temperature (T_p between 360°C and 400°C) within 15 min, a processing pressure was applied. Different impregnation pressures (0.5 to 3.0 MPa) and holding times (3 to 20 min) were selected to identify the impregnation mechanisms. The composite panels were then cooled under pressure from the melt to ambient temperature at an average cooling rate of 10°C/min. A typical processing cycle is illustrated in Fig. 2.

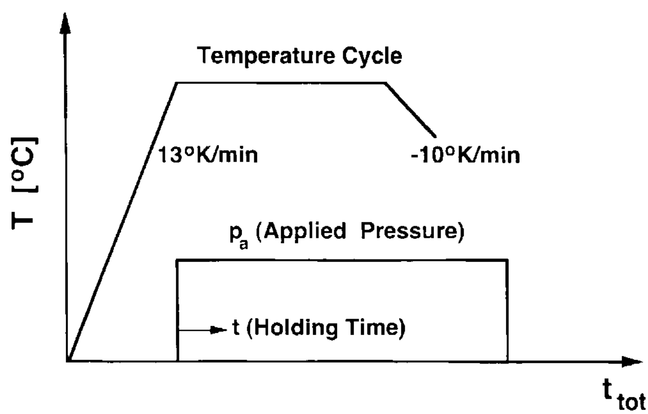

Figure 2. A typical processing cycle for consolidation of CF/PEEK composites.

3. MECHANISMS OF IMPREGNATION AND CONSOLIDATION

3.1. Commingled yarn

The impregnation mechanisms were examined using reflection light microscopy of polished cross-sections taken from consolidated composites. The consolidation process of CF/PEEK commingled yarns is quite different from that of pre-impregnated prepreg sheets (e.g. APC-2 [10–11]) or powder/sheath fibre bundles as discussed hereafter, because of the difference in distributions of matrix and reinforcing fibres in the preforms. In APC-2 CF/PEEK composites, because each individual composite prepreg sheet has been almost fully impregnated during prepreg processing, consolidation of composites can be described mainly by two regimes, namely, bulk consolidation (where intimate ply contact is developed and spatial gaps between composite layers are eliminated to form autohesion/diffusion bonding) and resin squeeze flow (where resin percolates through fibres in each composite layer to transport voids and to fill in matrix-depleted regions) [12], with the former as the dominant process. Micrographs in Fig. 3 illustrate some typical time-dependent consolidation steps in cross-sections of unidirectional CF/PEEK commingled yarn composites with respect to consolidation time. First, initially separated fibre yarns are flattened and moved towards one another when the pressure is applied. With temperature increasing above the melting temperature, T_m, PEEK polymer fibres start to melt. At this point, the molten polymer first tends to separate out of the reinforcing fibre bundles and fill the spaces between them, which is referred to as 'unmingling' [9]. At the end of this period, the border of the dry reinforcing fibre tow can still be clearly identified (Fig. 3a). In the second step, with increasing of holding time, the applied pressure drives the molten matrix to melt-impregnate the reinforcing fibres in the tows, thus pushing the air between them away. This process continues until the yarn is completely impregnated, and full consolidation is achieved (Fig. 3b–3d). It can be assumed that this sequence of mechanisms dominates the whole consolidation procedure of CF/PEEK commingled yarn composites, although it is found that not all molten polymer leaves the reinforcing fibre bundles to form a matrix pool around them in the first step. For this reason, at the final step of consolidation, autohesion of remaining molten matrix occurred in the fibre bundles at the front of melt impregnation from outside. Therefore, the front of impregnation could not be clearly identified.

3.2. Powder/sheath fibre bundle

It was found that the consolidation of this prepreg material could probably be described by two different procedures, namely, compaction and impregnation [13]. Micrographs in Fig. 4 illustrate some typical time-dependent impregnation steps in CF/PEEK-6K composites from the powder/sheath-fibre preform. During the compaction stage, initially separated fibre bundles were moved towards one another when the pressure was applied. With temperature increasing above T_m, the sheaths around the fibre tows melt first. Coalescence between melting sheaths then occurred, so that interval spaces between fibre bundles were filled by the matrix pool, surrounding the dry fibre

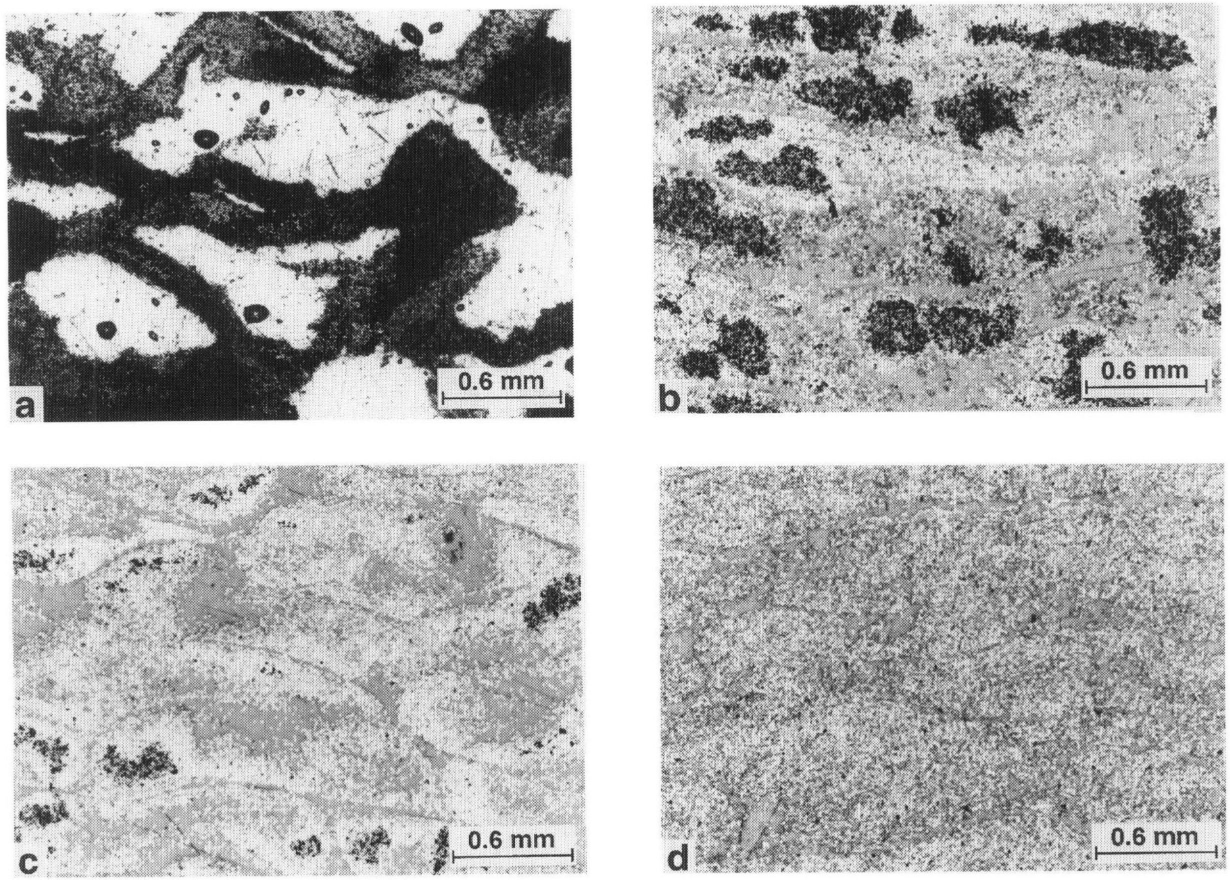

Figure 3. Micrographs for time-dependent consolidation steps in CF/PEEK commingled yarn composites.

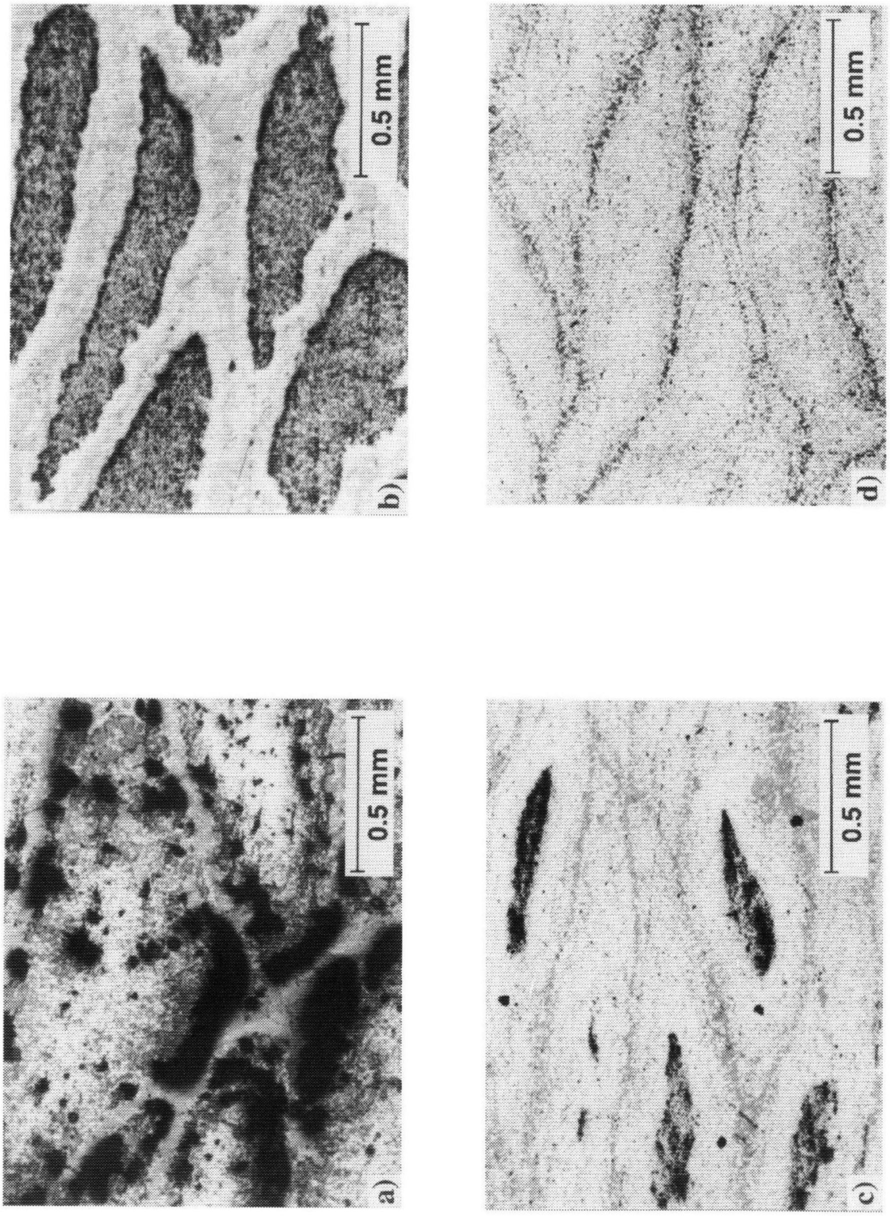

Figure 4. Micrographs for time-dependent impregnation steps in CF/PEEK-6K powder/sheath fibre bundle composites.

tow (Fig. 4a). In the second step, with increased holding time, the applied pressure drove the matrix into the reinforcing fibre tows and autohesion between the resin front and the melting powder took place, thus reducing the voids between them (Figs 4b and 4c). This process continued until the fibre tow was completely impregnated and full consolidation was achieved (Fig. 4d).

4. IMPREGNATION MODELS

Assuming that all fibre bundles undergo impregnation simultaneously and that all of them are identical in geometry, the consolidation of the entire composite can be described by the impregnation of a representative single cell (yarn/bundle) only. As the first order approach, it is further assumed here that the outer border of the fibre tow in the representative cell is fixed during the impregnation process, i.e. fibres are not moved towards each other and the fibre volume fraction is a constant and independent of the compaction. In this case, the compaction of a whole bundle and its consolidation are simply governed by the impregnation process of the molten matrix into the free space between the reinforcing fibres under a particular holding pressure. The velocity of the molten matrix passing through the aligned fibres can be defined by

$$\boldsymbol{v}_r = -\frac{K_{\mathrm{p}}}{\mu}\frac{\mathrm{d}\boldsymbol{p}}{\mathrm{d}r}, \tag{1}$$

where $\boldsymbol{v}_r$ the velocity vector of matrix impregnation [14], K_{p} the permeability of the fibre tow estimate by the Carman–Kozeny equation [15], μ the matrix viscosity, and $\boldsymbol{p}$ the pressure vector; r designates the position in the flow direction. For the commingled system, as mentioned previously, the as-received commingled status has a tendency to fade in the initial step of the consolidation process, because the molten matrix separates out of the fibre bundles. If the height of the partially impregnated area is h_{c}, a parameter, $D_{\mathrm{com}} = h_{\mathrm{c}}/h_{\mathrm{o}}$, is introduced, indicating the degree of the commingling status at the starting point of consolidation (i.e. the bundle is partially impregnated when the processing temperature is reached). D_{com} is equal to zero for a fully unmingled yarn, whereas $D_{\mathrm{com}} = 1$ stands for a fully commingled yarn. For a desired impregnation step (Fig. 5), the void content, X_{v}, in the composite as a function of the penetration distance, $r(t) = r_{\mathrm{f}}$, is therefore

$$X_{\mathrm{v}} = \frac{A_{\mathrm{m}} - V_{\mathrm{m}}b(h_{\mathrm{c}} + 2r_{\mathrm{f}})}{A_{\mathrm{b}} + A_{\mathrm{m}} - V_{\mathrm{m}}b(h_{\mathrm{c}} + 2r_{\mathrm{f}})}, \tag{2}$$

where V_{m} is the volume fraction of polymer matrix, b the bundle width, A_{b} the cross-sectional area of a fully consolidated (i.e. void free) filament yarn [9] and $A_{\mathrm{m}} = V_{\mathrm{m}} \cdot A_{\mathrm{b}}$. For the powder/sheath fibre bundle shown in Fig. 6, at a desired impregnation step, $r = r_{\mathrm{f}}$, the remaining free space in the fibre tow can be expressed as

$$V_{\mathrm{v}} = (1 - V_{\mathrm{f}} - V_{\mathrm{mp}})\pi r_{\mathrm{f}}^2 = \pi r_{\mathrm{o}}^2(1 - D_{\mathrm{imp}})(1 - V_{\mathrm{f}} - V_{\mathrm{mp}}), \tag{3}$$

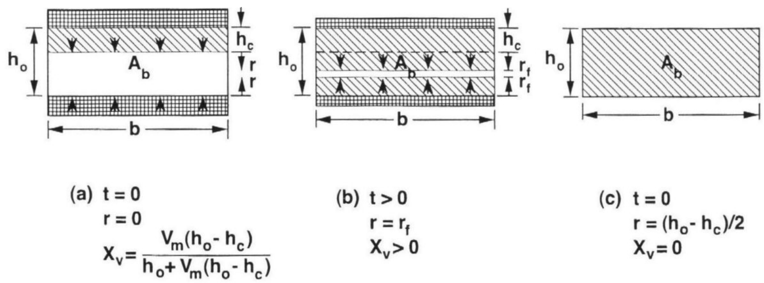

Figure 5. Schematics of consolidation steps in a representative yarn for partially commingled CF/PEEK composites.

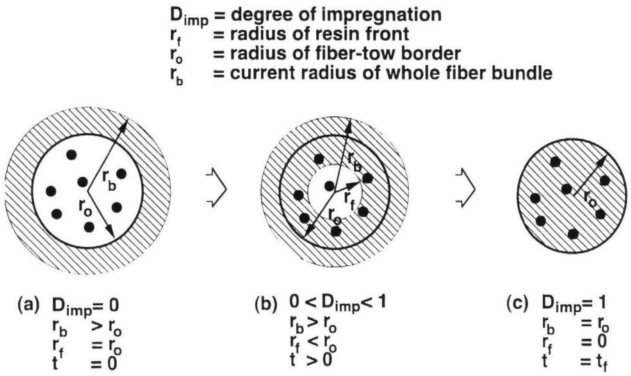

Figure 6. Schematics of consolidation steps in a representative bundle for CF/PEEK powder/sheath fibre bundle composites.

where V_f and V_{mp} are the volume fractions of the fibres and the matrix powder between them [13], r_o the equivalent radius of fibre bundle [3] and D_{imp} the degree of impregnation [16]. Hence, the void content, X_v, in a representative bundle at a specified impregnation step is

$$X_v = \frac{V_v}{\pi r_b^2}. \tag{4}$$

In this way, the consolidation quality of the entire composite can be simulated. Full impregnation is achieved when the molten matrix is totally transferred into the free space within the reinforcing fibre bundle. This represents a void free consolidated composite ($X_v = 0$), as shown in Figs 5(c) and 6(c). These approaches provide relationships between the void content and the processing variables in the consolidation process of composites. In particular, these variables are (a) viscosity as a function of processing temperature [$\mu = \mu(T)$], (b) applied pressure, (c) holding time and (d) bundle geometry. The viscosity data of PEEK matrix as a function of processing temperature in Kelvin were assumed to be the same as in the study of Kim *et al.* [16]:

$$\mu = 1.13 \times 10^{-10} \exp\left(\frac{19123}{T}\right) \quad \text{[Pa s]}. \tag{5}$$

5. CONSOLIDATION QUALITY AND MECHANICAL PROPERTIES

Density measurements were conducted in order to correlate consolidation states with apparent void contents in relation to the processing conditions. The composite density, ρ_e, for different processing conditions was determined according to ASTM-792. The theoretical density, ρ_t, of a fully consolidated composite was estimated by the following equation:

$$\rho_t = \frac{\rho_f \rho_m}{W_f \rho_m + W_m \rho_f}, \tag{6}$$

where ρ_f and ρ_m are the densities and W_f and W_m the weight fractions of the fibres and the matrix, respectively. The apparent void content, X_v, was then determined by:

$$X_v = \frac{\rho_t - \rho_e}{\rho_t} \tag{7}$$

which was used as an indication of consolidation quality of the composites [17].

Figures 7 and 8 illustrate the consolidation quality in CF/PEEK powder/sheath fibre bundle composites as a function of holding time at different processing conditions. The symbols indicate the apparent void contents, and the solid lines indicate the

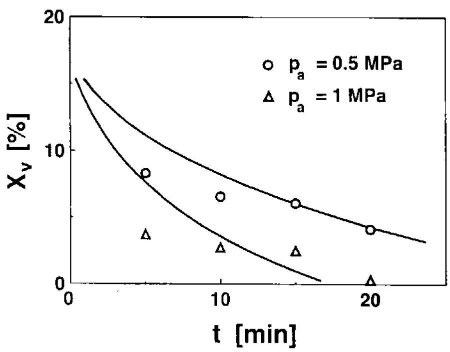

Figure 7. Void content of CF/PEEK-6K powder/sheath fibre bundle composites as a function of holding time at two different levels of applied pressure ($T = 390°C$).

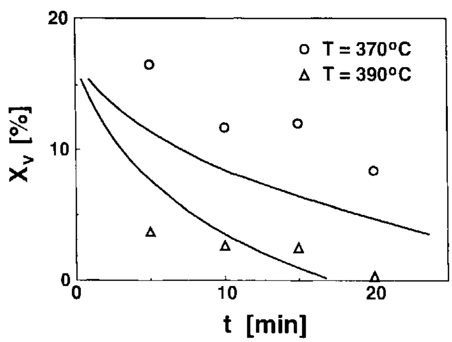

Figure 8. Void content of CP/PEEK-6K powder/sheath fibre bundle composites as a function of holding time at two different processing temperatures ($p_a = 1$ MPa).

simulations from the model. It can be seen that an increase in either holding time or applied pressure obviously reduces the void content in the composites, and produces better consolidated materials. For example, Fig. 7 illustrates void contents in the consolidated CF/PEEK-6K composites as a function of holding time at two different levels of applied pressure; the processing temperature was held constant at $T = 663$ K ($= 390°C$), i.e. $\mu = 380$ Pa s. The data indicate that the composite reaches a fully consolidated status when the holding time exceeds 20 min at $p = 1$ MPa. However, it seems that the CF/PEEK composites do not achieve a high level of consolidation at 360°C (i.e. $\mu = 1490$ Pa s).

Figures 9 and 10 illustrate the consolidation quality of CF/PEEK commingled yarn composites as a function of holding time at different processing conditions. The simulations were made from the model at $D_{com} = 75\%$. It can be seen again that an increase in either applied pressure or processing temperature clearly increases the consolidation quality of the composites. In Fig. 9 void contents in CF/PEEK composites are plotted as a function of holding time at three different levels of applied pressure; the processing temperature was held constant at $T = 653$ K ($= 380°C$), i.e. $\mu = 591$ Pa s. The data indicate that the composite has not yet reached a fully consolidated status after a holding time of 20 min, even at $p = 3$ MPa. In Fig. 11 the applied pressure was held at 1.5 MPa, whereas the processing temperature was varied between $T = 653$ K ($= 380°C$), 673 K ($= 400°C$, $\mu = 247$ Pa s) and

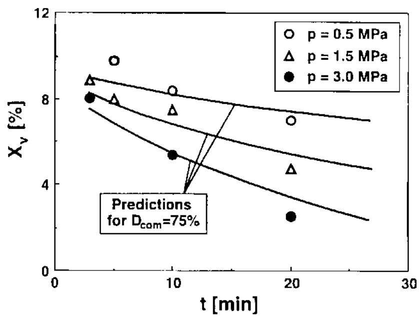

Figure 9. Consolidation quality of CF/PEEK commingled yarn composites at different processing pressures ($T = 380°C$, $D_{com} = 75\%$).

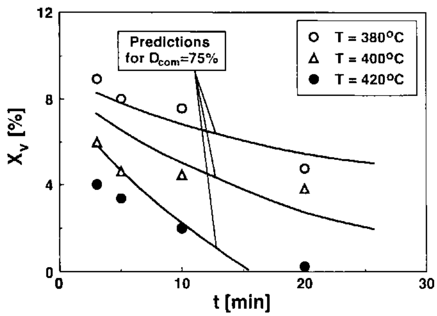

Figure 10. Consolidation quality of CF/PEEK commingled yarn composites at different processing temperatures ($p_a = 1.5$ MPa, $D_{com} = 75\%$).

693 K (= 420°C, μ = 109 Pa s). The experimental data indicate that the composite reaches a fully consolidated status when the holding time was about 20 minutes at p = 1.5 MPa and T = 420°C. From the results in Figs 9 and 10, it can be found that good correlations between experimental measurements and simulations from the consolidation model are achieved at $D_{com} = 75\%$.

From these results, it can be concluded that in spite of a good distribution of reinforcing fibres and polymer matrix in commingled yarns or powder/sheath fibre bundles (in the non-molten state), the observed 'unmingling' step at the onset of melting of the polymer fibres or a high viscosity of the matrix at low processing temperatures still obstruct fast consolidation and achieving of void-free composites.

Characterisations of mechanical properties as a function of the consolidation quality were conducted using a small transverse flexure (three point bending) testing fixture. The span of the specimens amounted to 40 mm; the width and thickness were about 10 mm and 3 mm, respectively. The cross-head speed was set to 2 mm/min. Transverse elastic constants and flexure strength were determined according to ASTM standard D-790.

Figures 11 and 12 illustrate effects of the consolidation quality on the transverse flexure strength of CF/PEEK composites. The ultimate strength, σ_{max}, is obviously reduced as the void content is increased. For example, for a change of the apparent

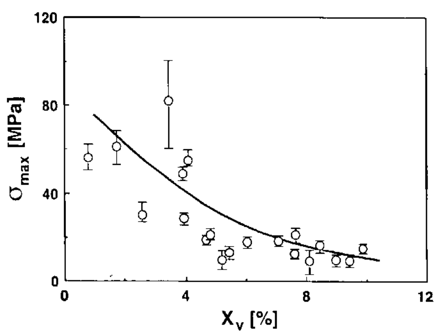

Figure 11. Transverse flexure strength of CF/PEEK commingled yarn composites versus consolidation quality.

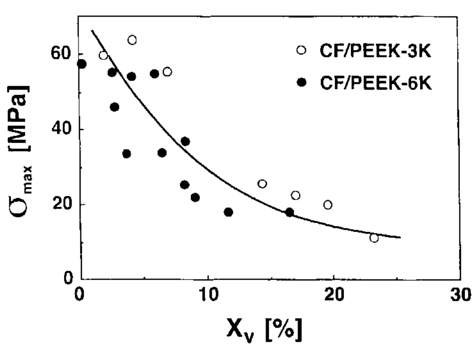

Figure 12. Transverse flexure strength of CF/PEEK powder/sheath fibre bundle composites versus consolidation quality.

void content from 23% to zero in the CF/PEEK powder/sheath fibre bundle composites, an increase in ultimate strength by, on the average, six-fold was identified. From these results, it can be stated that both transverse strength and elastic constant are highly dependent on the consolidation quality of composites, which is therefore an essential factor to be considered during the manufacture of thermoplastic composites. From Figs 11 and 12, it can be seen that the maximum apparent transverse flexure strength approaches 90 MPa for CF/PEEK commingled yarn composites and 70 MPa for CF/PEEK powder/sheath fibre bundle composites respectively. Both are significantly lower than a data sheet value of 138 MPa for APC-2 CF/PEEK composites [18]. This can be attributed to the fact that consolidation quality is not the only factor which influences mechanical properties of thermoplastic composites. In fact, the actual fibre volume fraction, matrix morphology, molecular weight, and fiber-matrix interfacial adhesion as well as kinetics of polymer matrix crystallisation also greatly affect the mechanical response of the consolidated composite parts [18]. While some good fibre/matrix adhesion has been found to be always present in the APC-2 prepreg, this interaction has to be created during the molding process of both flexible preforms used in this study. It was found [19–20] that the establishment of fibre/matrix adhesion in the commingled system was strongly dependent on processing variables.

Processing of composites can be based on a critical level of void content, e.g. $X_{vc} = 0.5\%$. The relationships between processing temperature, applied pressure, and holding time to stay below this maximum level of void content are described in Figs 13 and 14 for CF/PEEK powder/sheath fibre bundle and commingled yarn composites respectively. Two processing temperatures ($T = 380°C$ and $400°C$ for powder/sheath fibre bundle, and $T = 400°C$ and $415°C$ for commingled yarn) were selected for the model prediction of the relevant curves. Possible combinations of appropriate processing variables that lead to composite materials with the void content less than this critical value can be estimated. Hence, the mechanical property profiles of the consolidated composites will not be lower than those corresponding to this critical level of void content. From such a processing window, it can be deduced that, for example, at a processing temperature of 400°C, the holding time for high quality consolidation of CF/PEEK commingled yarn composites with a void content of less than 0.5% will be over 60 min (indicated by the point A in Fig. 14), at a processing

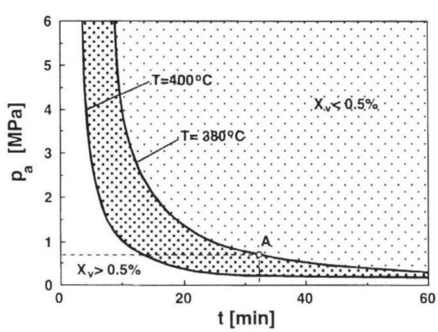

Figure 13. Optimum processing window for processing of CF/PEEK powder/sheath fibre bundle composites.

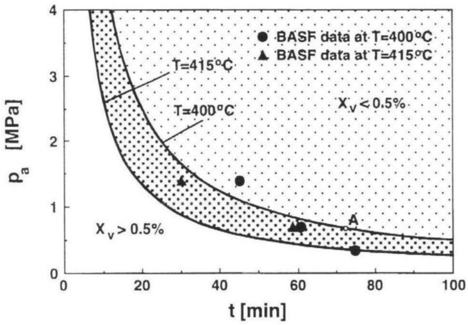

Figure 14. Optimum processing window for processing of CF/PEEK commingled yarn composites.

pressure of 0.7 MPa. This is quite close to the results published by BASF for a similar CF/PEEK commingled system [6], illustrated by the solid symbols in the window. For the CF/PEEK powder/sheath fibre bundle composites at a the processing temperature of 380°C, it is expected that the holding time for high quality consolidation with a void content of less than 0.5% will be around 32 min, at a processing pressure of 0.7 MPa.

7. CONCLUSIONS

The impregnation and consolidation mechanisms in CF/PEEK thermoplastic composites processed from commingled yarn and powder/sheath fibre bundle preforms have been investigated. Impregnation models have been developed to qualitatively describe the consolidation processes during composite processing. These models simulate void contents as a function of preform geometry and processing variables (temperature, applied pressure and holding time). Good correlations with experimental data indicate the success of these approaches. Based on a desired minimum level of void content ($X_v = 0.5\%$), optimum processing windows for CF/PEEK composites from these flexible product forms were generated. In practice, the present results would be of benefit to various processing techniques of thermoplastic composites for engineering applications, such as compression moulding, fast consolidation/or in-situ consolidation in filament winding and pultrusion manufacturing, etc.

A parameter has been introduced to describe the degree of commingled status which control consolidation of commingled yarn thermoplastic composites. However, as this parameter is dependent on the production process of commingled yarns, on proper 'sizing' and on subsequent procedure used to manufacture composites, the prediction of its value is very difficult. Further work should be done to identify this essential fact.

If comparing the impregnation and consolidation mechanisms of CF/PEEK commingled yarn composites with those in the materials processed from powder/sheath fibre bundles, longer processing times and higher applied pressures may be required to reach full consolidation. However, a direct comparison between the two types of proforms is hardly possible, because it is dependent on many variables associated with raw materials and their processing to final manufacturing processes of the composites.

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