CORRELATION BETWEEN EXPERIMENTAL RESULTS AND FINITE ELEMENT ANALYSIS OF A MODEL COMPOSITE CONTAINING AN INTERPHASE WITH KNOWN PROPERTIES

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SUMMARY: This paper reports a study of the effect of an interphase on strain development in fibre fragments obtained during fragmentation. In order to form an interphase, an epoxy resin with known properties was applied to the surface of unsized reinforcing fibres and cured. These were then embedded within a matrix resin coupon, for fragmentation testing. The findings of the experimental study indicated that there was no difference in the fragment length observed in the samples when a soft interphase was present, however when a hard interphase was present, the fragment length was slightly reduced. A finite element model was used to examine the strain development in more detail. This revealed that, for a soft coating, at the interphase thickness used in the study, the effect of the interphase properties was minimal. However increasing the thickness of the coating would increase the effect of the interphase.

KEYWORDS: Fibre fragmentation, discrete interphase, finite element modelling, load transfer, interphase thickness.

INTRODUCTION

The presence of an interphase is unavoidable in the production of polymer matrix composites. It can form in a number of ways [1], for example:

i). adsorption of contaminants on the fibres prior to embedding;
ii). diffusion of chemical species to the interface between fibre and matrix;
iii). acceleration or retardation of the polymerisation at the interface;
iv). the deliberate inclusion of sizing resin at the time of fibre manufacture.

The effect of the interphase on the performance of the composite is not well characterised, since its precise nature is difficult to predict.
Mathematical analyses have been proposed, which consider the effect of an interphase on the load transferred to an embedded fibre [2,3]. One problem with these analyses is the consideration of the matrix and interphase as elastic. In practical terms, the majority of resins used in the production of polymer matrix composites can be considered to be elasto-plastic in nature, displaying a distinct yield point. It has been demonstrated that yielding has a significant influence on the load transfer between matrix and fibre and, therefore, the assumption of elasticity is misleading [4].

Finite element analysis has also been used to examine the load transfer processes between matrix and fibre in the presence of an interphase [5]. This model accurately considered the elasto-plastic nature of the matrix and interphase by incorporating the stress-strain curves obtained from real polymeric systems. It was shown that the load transferred to the fibre was dependent on the properties of both the matrix and interphase. However, the effect of interphase thickness was not examined.

Experimental techniques have been used to study the effect of an interphase on the load transfer processes between fibre fragments and the matrix resin [6]. These have shown that the properties of the interphase significantly alter the nature of load transfer, with some systems giving full debonding of the fibre, while others gave no debonding. However, while the adhesive influence of the different interphase materials was examined, the mechanical and physical properties of the interphase were ignored.

The current paper therefore examines the effect of an interphase, with known properties, on the load transferred to a reinforcing fibre. This includes a consideration of the interphase thickness, as well as the elasto-plastic nature of the resins used. The effect of the interphase is assessed using the fragmentation test geometry. Finite element analysis has been employed to provide further understanding of the experimental results.

**EXPERIMENTAL**

**Experimental study**

Three formulated epoxy resin blends were used in this study. They were produced by mixing two epoxy resins (Shell Epikote 828 and Ciba-Geigy Araldite 298) in varying proportions, each blend being named after the proportion of Epikote 828 followed by the proportion of Araldite GY298. In this way, three blends were defined, 1090, 5050 and 6040, where 1090 has 10% Epikote 828 and 90% Araldite GY298 by weight. The blends of resin were cured using a mixture of two hardeners (Henkel Capcure 3-800 and Stag Polymers and Sealants NMA), the proportions of each hardener remained constant and a stoichiometric mix of resin to hardener was employed. This ensured that chemically similar resins, with varying mechanical properties, were obtained. The chemical formulations of the blends used in this study are shown in Table I.

All of the resin blends were cured using a two stage cure process of 4 hours at 80ºC and 2 hours at 130ºC. Once the specimens had been removed from the mould, a further 2 hours at 130ºC was employed as a post-curing step. Samples consisting of resin, without an embedded fibre, were produced and their mechanical properties determined. The stress-strain curves for each blend are shown in Figure 1.
Table I. Mix ratios for the resin formulations used in this study (phr = per hundred of resin by weight).

<table>
<thead>
<tr>
<th>Constituent/Blend</th>
<th>1090</th>
<th>5050</th>
<th>6040</th>
</tr>
</thead>
<tbody>
<tr>
<td>Epikote 828</td>
<td>10%</td>
<td>50%</td>
<td>60%</td>
</tr>
<tr>
<td>Araldite GY298</td>
<td>90%</td>
<td>50%</td>
<td>40%</td>
</tr>
<tr>
<td>Capcure 3-800</td>
<td>41.5phr</td>
<td>59.42phr</td>
<td>63.9phr</td>
</tr>
<tr>
<td>NMA</td>
<td>15.54phr</td>
<td>22.26phr</td>
<td>23.93phr</td>
</tr>
</tbody>
</table>

Figure 1. Stress-strain curves for the three resin formulations used in this study.

In-order to prepare fragmentation specimens, single untreated-unsized carbon fibres (Tenax HTA) were separated from the fibre tow and mounted across a wire frame, facilitating further handling. The fibre was then coated by drawing it across the surface of a glass rod, which had been dipped into the formulated resin. This enabled a controlled coating to be applied to the fibre. The coated fibre, on its wire frame, was then suspended in an oven while the coating was cured.

Once the coating had been cured, the coated fibres were embedded in the matrix resin. In this study the 5050 blend was used as the matrix. The specimens were cast, in silicone rubber dog-bone moulds, with a gauge length of approximately 35 mm, and cured using the standard cure process.

Four matrix/interphase pairings were studied:

i). 5050/1090;
ii). 5050/5050;
iii). 5050 (uncoated);
iv). 5050/6040:
where the first blend quoted is the matrix system. The specimens which were uncoated were examined as a reference, to ensure that the coating process was not adversely affecting the result.

Optical microscopy of the cured specimens revealed that the fibres were not straight, but displayed a regular sine-wave profile along their length. This was due to the different thermal expansion coefficients of the fibre and the resins, leading to the development of curing stresses. Therefore, a post-cure step was employed, during which the specimens were first heated to 130°C (the level of the final stage of the curing process), to relieve the curing stresses. They were then restrained, preventing axial shrinkage during the cooling process. The samples were slowly cooled to room temperature, thereby relieving the curing strains by annealing the sample. Once the specimens had cooled to room temperature, they were removed from the oven and the fibres were found to be straight.

Fragmentation testing of the specimens was carried out using an automated mini-tester, whose development is discussed elsewhere [7]. This facilitated simple observation of the fragmentation process and enabled digital photographs of the fibre fragments to be readily obtained for subsequent analysis. The photographs were all captured at a fixed magnification and then the fragment lengths were measured using a custom written computer program, which was calibrated using a photograph of a graticule, at the same magnification as those of the fragments.

**Finite element analysis**

A 2D axisymmetric finite element analysis was employed, with the model being simplified further by considering quarter symmetry. A schematic of the resultant model is shown in Figure 2, with the dimensions of each constituent. The geometry was chosen to represent a fibre embedded in an infinite block of resin, the matrix dimensions were chosen to ensure that the strain along BD was less than 1% of the maximum attained strain. The model was created using ANSYS 5.4 and meshed, using axisymmetric boundary conditions, with first-order structural-solid elements (type Plane 42). The model consisted of 10320 elements and 10584 nodes. Restraint was applied along AC and AB, with a displacement being applied to CD, in-order to ensure the correct boundary conditions along sides AC and AB.

![Figure 2. Schematic illustration of the model geometry used in this study.](image-url)
The matrix properties employed in the model were those of the 6040 system, with the interphase being defined as the 5050 blend. The stress-strain curves for these blends were digitised and directly included in the FEA model. The fibre was considered to be carbon with an axial modulus of 220 GPa and a transverse modulus of 14 GPa. A small region of soft material was included between the fibre-end and the matrix to negate the effect of end-bonding. This material was defined as an isotropic elastic material with a modulus of 10 MPa, which has previously been shown to be successful in the elimination of end-bonding effects [8].

The model was then run to an applied strain of 1%, because a significant degree of plasticity existed in the interphase and matrix, due to the stress concentration at the fibre end. Since the model was not a direct representation of the experimental arrangement, it was considered that this strain level was sufficient for the comparison of trends in the data.

**Results and discussion**

The measured average fragment lengths are shown in Table II. Each value is an average of approximately 40 individual fragments. It can be seen that the 5050/1090, 5050/5050 and 5050 (uncoated) systems all have similar fragment lengths. However, the average fragment lengths obtained from the 5050/6040 system appear to be lower than the others. It was expected that the different interphases would lead to an observed variation in fragment lengths, with a soft interphase increasing the average fragment length, and a stiff interphase decreasing it. However, whereas a slight reduction in the fragment length was observed with the stiff interphase, the soft interphase did not show an effect.

**Table II.** Average fragment lengths, at saturation in the fragmentation test.

<table>
<thead>
<tr>
<th>System</th>
<th>Sample 1</th>
<th>Sample 2</th>
<th>Sample 3</th>
</tr>
</thead>
<tbody>
<tr>
<td>5050/1090</td>
<td>0.56 +/-0.12</td>
<td>0.52 +/-0.15</td>
<td>0.53 +/-0.15</td>
</tr>
<tr>
<td>5050/5050</td>
<td>0.61 +/-0.16</td>
<td>0.59 +/-0.15</td>
<td>0.56 +/-0.14</td>
</tr>
<tr>
<td>5050 (uncoated)</td>
<td>0.58 +/-0.17</td>
<td>0.55 +/-0.16</td>
<td>0.55 +/-0.16</td>
</tr>
<tr>
<td>5050/6040</td>
<td>0.49 +/-0.17</td>
<td>0.40 +/-0.12</td>
<td>---</td>
</tr>
</tbody>
</table>

In-order to further examine the load transfer processes, the birefringence patterns obtained in the photographs were studied. Examples of the 5050/6040, 5050 (uncoated) and 5050/1090 systems, at saturation in the fragmentation process, are shown in Figures 3 to 5 respectively. These photographs are presented as examples of typical birefringence patterns, they do not precisely follow the data presented in Table II as this data is the average fragment length.

Comparison of Figures 3 and 4 shows that the presence of a hard interphase decreases the area of the illuminated region around the broken fibre-end, indicating that less deformation is induced by the fibre fracture in this instance. This is a result of the stiff interphase which resists deformation, leading to an increased rate of strain development within the fibre and, thus, shorter fragments. Comparison of Figures 4 and 5, however, reveals little difference in the load transfer at saturation, when a soft interphase is present.
Examination of the average fragment lengths and the birefringence patterns therefore failed to reveal any differences when a soft interphase was present. However, comparison of the strain intervals between the onset of fragmentation and the point of saturation, for the different systems, revealed a significant difference. Table III shows a comparison of the strains at the onset of fragmentation and at saturation and also the strain interval between these two events. The strain interval is the critical parameter, as the level of the curing strains within each sample is unknown. Therefore, direct comparison of the strain levels could lead to inaccuracy, however, the strain interval should be unaffected.

Table III. Comparison of the average strains for fragmentation onset and saturation and the strain interval between them.

<table>
<thead>
<tr>
<th>System</th>
<th>Average strain at onset (%)</th>
<th>Average strain at saturation (%)</th>
<th>Strain interval between onset and saturation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5050/1090</td>
<td>2.67</td>
<td>6</td>
<td>3.33</td>
</tr>
<tr>
<td>5050/5050</td>
<td>3</td>
<td>7.33</td>
<td>4.33</td>
</tr>
<tr>
<td>5050 (uncoated)</td>
<td>2.67</td>
<td>7</td>
<td>4.33</td>
</tr>
<tr>
<td>5050/6040</td>
<td>3</td>
<td>9</td>
<td>6</td>
</tr>
</tbody>
</table>

The results in Table III reveal that the strain interval over which fragmentation occurred is reduced in the presence of a soft interphase, and increased in the presence of a hard interphase. In order to understand this data, the results of the finite element analysis were examined.

From Table III, it can be seen that the coating process did not adversely affect the results, as the 5050 (uncoated) reference system gave the same results as the 5050/5050 system.
Finite element analysis of the 6040/5050 system, with a 0.2 µm interphase thickness, satisfactorily accounted for the observation that the ultimate fragment length was not significantly affected by the inclusion of an interphase (Figure 6). The analysis revealed that the tensile strain in the fibre centre, in the presence of a soft 0.2 µm interphase, was very similar to that in the pure 6040 system, at an applied strain of 1 %. When the interphase thickness was increased to 2 µm, the tensile strain in the fibre centre for the 6040/5050 system was found to be the same as that for a pure 5050 system, at the same applied strain (Figure 6).

Figure 6. Tensile strain in the fibre centre (TSFC) profiles for 6040 matrix and interphase, 5050 matrix and interphase, 0.2 µm 5050 interphase in 6040 matrix and 2 µm 5050 interphase in 6040 matrix.

These results show that the thickness of an interphase, at a given applied strain, significantly affects the load transfer processes between fibre and matrix. Figures 7a and b show schematically the deformations taking place within the sample when a soft interphase is present, for two different interphase thicknesses. It can be seen that in the case of the thin interphase, the interphase deformation is constrained by the comparative stiffness of the matrix, reducing the overall extent of the deformed region (Figure 7a). However, in the case of a thick interphase, the deformation occurs largely in the interphase region, relatively independent of any matrix constraint (Figure 7b). The same argument can be applied in the case of a stiff interphase, although the results are reversed such that a thick interphase gives little deformation, while a thin interphase gives increased deformation. This is due to the relative stiffnesses of the interphase and matrix materials.
Figures 7. Schematic illustrations of the effect on the size of the deformed region around a broken fibre end, at a fixed applied strain when a) the coating is thin, and b) the coating is thick.

These results also give an indication of the manner in which the applied strain may influence the load transfer process when an interphase is present (Figure 8a and b). As the applied strain increases, the deformed region around the fibre-end increases. Initially this is confined to the interphase region and the load transfer processes are therefore likely to depend on the interphase properties (Figure 8a). With increasing applied strain, the deformation spreads into the matrix, leading to a reduction in the importance of the interphase properties (Figure 8b). Therefore, both interphase thickness and applied strain must be considered when an interphase is present in a composite structure.

Figures 8. Schematic illustration of the effect of applied strain on the size of the deformed region around a fibre break when a) the applied strain is low, and b) the applied strain is high.

The implications of the finite element analysis can be extended to consider the data shown in Table III, with the following results. In the case of a system with a soft interphase, it can be seen that at low applied strains, the strain experienced by the fibre will be reduced as a result of the presence of the interphase. Whereas at higher applied strains, the strain relieving effect of the interphase is negated. Therefore, fragmentation will be retarded at low applied strain,
but much less inhibited as the strain increases, leading to a shorter strain interval between the onset of fragmentation and saturation, matching the experimental observations.

The same reasoning can be applied in the presence of a hard interphase. At low applied strains, load transfer is more efficient and therefore fragmentation will start at a lower strain. However, at higher applied strains the significance of the interphase is reduced, leading to a less efficient load transfer. Therefore, the fragmentation will proceed at a reduced rate and the strain interval between onset of fragmentation and saturation will be increased. Again this correlates with the experimental observations.

CONCLUSIONS

The presence of an interphase region between fibres and matrix, in a composite system, can significantly affect the load transfer characteristics.

Experimental examination revealed that the effect of a thin interphase on the saturation fragment length was low. However, the strain interval between the onset and saturation of the fragmentation process was found to be significantly affected by the properties of the interphase region.

Finite element analysis showed that as the thickness of the interphase increased, for a given applied strain, the load transfer characteristics varied from those of the matrix to those of the interphase. This observation allowed the extrapolation that as the applied strain increased, for a given interphase thickness, the load transfer characteristics varied from those of the interphase to those of the matrix.

These results suggest that by controlling the interphase characteristics, the ultimate properties of a composite can be engineered.

REFERENCES


