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# Matrix Modification for Improved Reinforcement Effectiveness in Polypropylene/Glass Fibre Composites

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**Abstract.** The influence of matrix modification on the interfacial shear strength (IFSS) and the mechanical performance of polypropylene/glass fibre composites is investigated. Two different modifiers were used: a highly reactive hyperbranched polymer grafted polypropylene (HBPgPP) and a maleic anhydride grafted polypropylene (MAHgPP). The interfacial shear strength increased with the addition of the modifiers, with HBPgPP giving the highest values. To evaluate the effects of the matrix modification on the composite strength, a method to normalise the composite strength with respect to fibre orientation and fibre concentration is presented. The normalised strength values followed the same trend as the measured IFSS values, namely that the HBPgPP modified composite displayed the highest strength and the unmodified material the lowest.

**Key words:** short fibres, mechanical properties, interface, PP composites.

## 1. Introduction

Composite materials based on polypropylene (PP) and glass fibres (GF) offer a very competitive property/price ratio. Their comparatively low cost, fast processability and recyclability motivate their use in a range of applications, most notably in the automotive industry. Depending on the manufacturing process chosen, a range of material configurations is possible. These material configurations differ principally with respect to fibre length, content and architecture.

Polypropylene/glass fibre composites are generally produced in two steps; namely the fibre/matrix impregnation and then composite forming. The most common impregnation processes are extrusion (using chopped fibres), strand impregnation (using fibre filaments) and compression moulding (using glass-mats) [1]. The extrusion process is relatively straightforward and cost-effective but tends to damage the fibres, which reduces the composite properties. Strand impregnation is the most expensive of the three and yields material with continuous fibres, whereas

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the compression moulding process has the advantage of being cost-effective and does not harm the fibres.

The composite forming step is carried out by injection moulding, using granules of extruded or strand impregnated material, or by stamping, using extruded, compression moulded or strand impregnated material. Extrusion–injection moulding is cost-effective and allows for a high design freedom but, on the other hand, yields the lowest intrinsic properties of all the processes. Strand impregnation combined with injection moulding yields composites with longer fibres and, hence, better mechanical properties. The stamping process allows for less design freedom compared to the injection moulding process but gives composites with much longer fibres and higher intrinsic properties. However, the manufacturing process and the fibre length are not the only parameters influencing the intrinsic properties of PP/GF composites. The fibre/matrix adhesion plays also an important role.

For adhesion to occur it is essential that intimate contact is established between the matrix and the fibre. The ability of the matrix to wet the fibre during processing depends on the relative surface tension between the fibre and the matrix. If the matrix surface tension is lower than that of the fibre, spontaneous wetting may occur as long as the viscosity of the matrix is favourable [2]. However, to ensure good interfacial adhesion and stress transfer across the interface, chemical or physical interactions between the matrix and the fibre need also to be formed. The potential for physical or chemical interaction is limited in polyolefin resins such as PP due to their inert nature [3]. To overcome this, chemically reactive groups can be grafted to the non-polar resin. The grafted PP migrates to the fibre surface and forms chemical bonds during processing. This, however, is only possible if the fibre sizing contains chemical groups able to react with the grafted PP molecules. Thus, to ensure a good fibre/matrix adhesion, both the fibre and the matrix have to be chemically adapted [2].

The influence of matrix and fibre modification on the interfacial properties and final composite properties of a range of composites has been extensively studied [4–7]. Maleic anhydride (MAH), which is grafted onto the PP backbone by reactive extrusion [8, 9], is the most common chemical modifier for increasing the reactivity and polarity of PP. Earlier work has shown a significant increase of both fibre/matrix adhesion [2] and composite properties with the addition of MAH grafted PP (MAHgPP) [4, 10]. The level of improvement has been shown to depend on the concentration of MAHgPP and on the type of fibre used, in particular on its chemical reactivity [6, 10, 11]. A question, however, still remains as to the influence of the MAH concentration in the MAHgPP. This material parameter could play an important role in the fibre/matrix adhesion since the probability that MAHgPP reacts at the fibre surface should increase with an increasing MAH concentration. On the other hand, since the MAH grafting reactions involve chain scission, the grafting of more MAH molecules onto each PP chain results in a lower molecular weight of the MAHgPP. This will decrease the ability of the MAHgPP molecules to entangle and to cocrystallise with the PP matrix and, hence, decrease

the MAHgPP/PP adhesion [12]. A compromise must therefore be made between the MAH grafting level and the molecular weight of the MAHgPP.

In this paper a new approach for increasing the functionality of the modifier has been investigated. Multifunctional reactive hyperbranched polymers (HBP) have been grafted to the MAHgPP in order to form an HBPgPP modifier. This increases the modifier reactivity without decreasing its molecular weight. This novel approach to matrix modification has already been shown to be effective for PP/PA6 blends, for which a significantly higher interfacial adhesion was achieved at lower modifier concentrations than with MAHgPP [13].

The goal of this paper is to determine the effect of HBPgPP on the fibre/matrix adhesion and the related strength of the composite. For this purpose, the mechanical response of composites is first analytically described in order to assess the contribution of the fibre/matrix adhesion. Secondly, the performance of two modifiers, MAHgPP and HBPgPP, is experimentally determined and compared.

## 2. Influence of Interfacial Strength on Mechanical Properties

The stiffness and strength of fibre-reinforced composites are strongly influenced by the fibre length and their orientation relative to the applied load. In this section, analytical expressions are introduced to describe this dependency.

### 2.1. STIFFNESS

The expression in Equation (1) has been proposed as an engineering approximation for the stiffness of a composite having almost randomly oriented reinforcements [14, 15]:

$$E_c = V_m E_m + k_{SF} k_{FOD} V_f E_f, \tag{1}$$

where $E_c$ is the modulus of the composite, $V_m$ the matrix volume fraction, $E_m$ the modulus of the matrix material, $V_f$ the fibre volume fraction and $E_f$ the modulus of the fibres. The parameters $k_{SF}$ and $k_{FOD}$ are reinforcement efficiency factors related to the fibre aspect ratio (length to diameter ratio) and the fibre orientation distribution respectively. The values of each of these factors range from 0 to 1, where 1 is the maximum efficiency. This expression allows the fibre orientation and fibre length terms to be decoupled. It should be noted, however, that Equation (1) does not take the fibre reinforcement in the transverse direction into account and should thus slightly underestimate the stiffness of the composite. The efficiency factor $k_{SF}$, and thus the stiffness of composite, depends on the fibre aspect ratio and can be estimated with the Cox shear lag model. It was shown by Thomason et al. that the fibre length required for an asymptotic value of the modulus for a glass mat reinforced composite (GMT) with dispersed random fibres and a fibre content of 30% (by weight) was less than 0.5 mm [15]. The fibre lengths in glass mat reinforced composites are in the range of several millimetres to centimetres,

so $k_{SF}$ can be assumed to be equal to unity. In injection moulding grades, on the other hand, the fibre lengths are often in the range of tenths of millimetres and $k_{SF}$ is thus below unity.

The factor $k_{FOD}$ in Equation (1) describes the orientation of the fibres with respect to the applied stress field. It can be estimated using Equation (2) [16] for any known fibre orientation distribution function, $f(\theta)$,

$$k_{FOD} = \int_{-\pi/2}^{\pi/2} f(\theta) \cos^4 \theta \partial\theta, \tag{2}$$

where $\theta$ is the fibre orientation with respect to the stress field. If the fibre orientation distribution can be assumed to be random, $f(\theta)$ is a constant, which results in a $k_{FOD}$ of 0.38. If the orientation is unidirectional, $k_{FOD}$ becomes 1 in the direction of fibre orientation and 0 in the transverse direction. The fibre orientation distribution function, $f(\theta)$, is dimensionless and must fulfil the normalisation condition, namely

$$\int_{-\pi/2}^{\pi/2} f(\theta) \partial\theta = 1. \tag{3}$$

It can be seen from Equation (1) that the fibre orientation and the fibre length both have a strong influence on the stiffness of the composite while the interfacial strength should have no influence. However, the latter is only true if the interfacial shear strength is high enough to transfer all acting stresses. It is assumed here that the IFSS always is higher than the stresses acting at the interface since the deformations are very small when measuring the modulus.

### 2.2. STRENGTH

The strength of a non-continuous fibre composite can be calculated using the method presented by Hull and Clyne, in which the Kelly–Tyson equation is used to calculate the reinforcing efficiency of the fibres [16–18]:

$$\sigma_c = V_m \sigma_m + k_{FOD} \left( \sum_{L_i=0}^{L_i=L_c} \frac{V_i \tau L_i}{D} + \sum_{L_j=L_c}^{L_j=\infty} \sigma_f V_j \left( 1 - \frac{L_c}{2L_j} \right) \right), \tag{4}$$

where $\sigma_c$ is the composite strength, $\sigma_m$ the matrix strength, $\sigma_f$ the fibre strength, $\tau$ the interfacial shear strength, $D$ the fibre diameter and $L$ the actual fibre length. The indices $i$ and $j$ represent subcritical and supercritical fibres, respectively. The critical fibre length, $L_c$, which corresponds to the fibre length required for the fibre strength to be reached over its length without interfacial failure occurring, is given by:

$$L_c = \frac{D\sigma_f}{2\tau}. \tag{5}$$

Equation (4) is based on the assumptions that subcritical fibres fail at the interface and that supercritical fibres break after plastic deformation of the matrix material at fibre ends. It can be seen from Equation (4) that the strength, in contrast to the stiffness, is influenced by the quality of the fibre/matrix adhesion, which can be expressed by the interfacial shear strength (IFSS). It should, therefore, be possible to improve the composite strength by the addition of reactive modifiers that enhance the fibre/matrix adhesion. However, Equation (4) also shows that the fibre length has a pronounced influence on the composite strength. In order to determine the fibre length for which matrix modification has the most pronounced effect on the composite strength, the relative strength increase resulting from an increased IFSS is investigated as a function of fibre length below.

### 2.3. PREDICTIONS OF STRENGTH SENSITIVITY

The strength of a composite having random in-plane oriented fibres of monodispersed length was calculated as a function of the fibre length and IFSS using Equation (4). The parameters used for the prediction were a fibre strength of 2000 MPa and a matrix strength of 31 MPa. In Figure 1, the strength increase

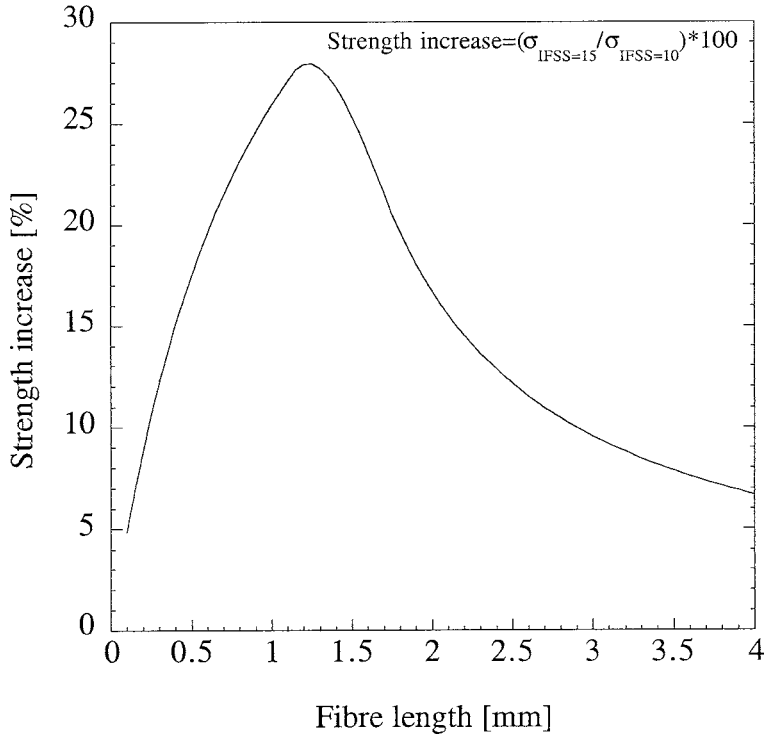


*Figure 1.* Predicted strength increase resulting from an IFSS increase of 10 to 15 MPa, as a function of the fibre length.

resulting from an IFSS increase of 10 to 15 MPa is shown as a function of the fibre length. The resulting curve is thus an expression for how effective the interfacial modification is for different fibre lengths. It can be seen in Figure 1 that there is a maximum at a fibre length of approximately 1.2 mm, at which the interfacial shear strength has a maximum effect on the strength. Below this optimum the influence of fibre reinforcement on the composite strength becomes less important and therefore so does the influence of the IFSS. Above the optimal fibre length, on the other hand, the fibre strength is more important than the IFSS in determining the composite strength. Therefore, matrix modification would be most beneficial for composites containing fibres with a length around 1.2 mm. It should be noted, however, that the critical fibre length for which IFSS has a noticeable effect on the composite strength depends on the level of the IFSS. A higher IFSS shifts the critical fibre length to lower values.

## 3. Materials and Experimental

### 3.1. MATERIALS

A heterophasic blend of PP and ethylene propylene rubber (Moplen EPN 31MA, Montell) was used as the matrix material. The PP was modified with 2% by weight of either MAHgPP (Polybond 4000, Uniroyal) or HBPgPP. The HBPgPP was produced by melt blending MAHgPP and HBP in a weight ratio of 93 : 7 using a twin screw extruder. The HBP (Boltorn$^{TM}$E2, Perstorp AB) used has a dendritic structure with a polyester-based core and approximately 28 epoxy groups on the shell. During melt blending with MAHgPP, the epoxy groups of HBP react with the MAH groups of the MAHgPP and HBPgPP molecules containing on average one HBP molecule and 4 PP molecules are formed.

Six different glass fibres from various suppliers were used. All the fibres had a surface treatment suitable for use with a PP matrix, but the exact composition of the surface treatments was not made available.

### 3.2. CHARACTERISATION OF THE FIBRE/MATRIX ADHESION

The fibre/matrix adhesion was characterised using the microbond pull-out test [19]. This consists of pulling a fibre, on which a droplet of matrix material has been placed between two fixed jaws, as schematised in Figure 2. The distance between the jaws is only slightly wider than the fibre diameter and is smaller than the diameter of the droplet. The interfacial shear strength (IFSS) is calculated by dividing the required force to detach the microdroplet by the area of the fibre-matrix interface.

The microbond samples were prepared by placing polymer fibres perpendicular to glass fibres placed on a metallic frame. The frame was inserted into an oven at a temperature of 190°C for 5 minutes. This allowed the polymer fibres to melt around the glass fibres and form axisymmetrical droplets with a length of 150–300 μm, as

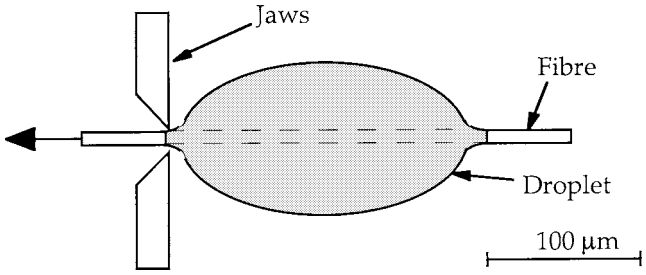


*Figure 2.* Schematic of the microbond pull-out test.

measured by optical microscopy. The IFSS was measured in a UTS load frame equipped with a 1 N load-cell using a cross-head rate of 1 mm/minute.

### 3.3. DETERMINATION OF AMINE CONTENT OF FIBRE SIZING

Amino-silane is often used as the reactive component in the fibre surface treatment and has the potential to react with MAHgPP or HBPgPP. The presence of amino-silane is, therefore, crucial in order to achieve a good fibre/matrix adhesion. The amino-silane contents of the fibres were measured using the Kjeldahl method [20]. The fibres are firstly boiled in acid in order to decompose fibre sizing. In doing so, the substances containing nitrogen, such as the amino-group of the amino-silane, are transformed into ammonia, the yield of which indicates the total nitrogen content.

### 3.4. COMPOSITE MANUFACTURING

Composite specimens were produced by an extrusion and compression moulding process. The fibres were cut to 12 millimetres length and were dry mixed with the PP matrix in powder form. The mix was melt blended in a single screw extruder at 230°C and 50 bar. The molten extrudate was taken directly from the die, placed into a flat mould of $250 \times 250$ mm$^2$ held at 70°C, and was compression moulded at a pressure of 12 MPa. To minimise physical ageing effects and residual crystallisation, the moulded plates were kept at least one week under ambient conditions before mechanical testing. Specimens were cut from the plates both parallel and perpendicular to the direction of the extrudate as placed in the mould.

The tensile strength of the composites was characterised by tensile tests using a Zwick load frame equipped with an extensometer. The tests were performed at ambient temperature at a rate of 5 mm/minute. At least 8 specimens were tested for each composite.

### 3.5. DETERMINATION OF FIBRE LENGTH DISTRIBUTION

The fibres were first separated from the matrix by incineration in a furnace maintained at 600°C for 2 hours and then transferred to a glass slide. The lengths of

approximately 500 fibres were measured using an optical microscope in order to determine the fibre length distribution.

## 4. Results and Discussion

### 4.1. INTERFACIAL PROPERTIES

Results from the microbond pull-out test using different modified matrices are shown in Figure 3. It can be seen that modified PP has a higher interfacial shear strength (IFSS) than pure PP, due to improved fibre/matrix adhesion. The IFSS of unmodified PP is, however, higher than expected, considering that no chemical interactions at the interface are possible due to the inert nature of PP. This may be due to high frictional forces caused by matrix shrinkage upon crystallisation [19] and/or to the affinity of the glass fibre treatment to the PP matrix. The increase in IFSS for the modified composites is attributed to the grafting of PP molecules at the fibre surface. Assuming a direct correlation between the IFSS increase (5 MPa for the HBPgPP and 4.1 MPa for the MAHgPP) and the amount of PP molecules grafted at the fibre surface, the results in Figure 3 indicate 22% more PP molecules have reacted at the fibre surface in the case of HBPgPP in comparison to MAHgPP. This can be attributed to the higher diffusitivity of the HBPgPP modifier, leading to more grafted molecules locating at the matrix/fibre interface during the annealing of the microbond samples. The higher diffusitivity of HBPgPP, which has been observed in PP/PA6 blends [21], is due to the solubility difference between the PP matrix and the relatively large polyester based structure of HBP inducing a high diffusion driving force. The IFSS increase as a result of modifier addition

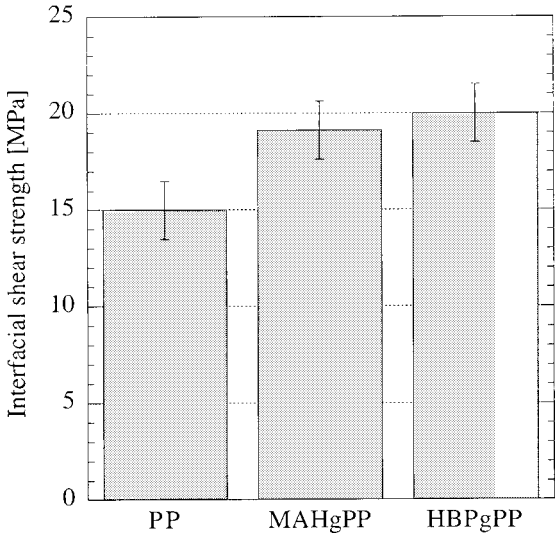


*Figure 3.* The interfacial shear strength (IFSS), as measured by the microbond pull-out test, of unmodified and modified PP matrix materials.

will, however, not only depend on the ability of the modifier to diffuse to the fibre surface but also on the reactivity of the fibre surface, namely on the type and density of reactive groups on the fibre surface. This is illustrated in Figure 4, which shows the influence of the amine content of the fibre surface treatment on the IFSS of HBPgPP modified PP. The IFSS increases with the amine content, indicating that the adhesion improvement with the addition of HBPgPP originates from epoxy-amine reactions. As a result, the amine content determines the amount of PP molecules that can be grafted at the fibre surface. The IFSS is thus shown to be slightly influenced by the type of modifier and to be strongly dependent on the number of reactive groups at the fibre surface.

### 4.2. MECHANICAL PROPERTIES

The measured stiffness of non-modified and modified composites is shown in Figure 5. The difference in modulus in different directions of the moulded plates results from the high anisotropy of the fibre structure, which is attributed to the flow induced orientations during the extrusion-compounding of the composite. For the different composites, small variations of modulus are also observed. These should result only from variations in fibre orientation and/or fibre concentration since the influence of fibre/matrix adhesion on the composite stiffness is negligible. The strength, on the other hand, is measured at high deformations at which the fibre/matrix adhesion plays an important role. Indeed, as shown in Figure 6, the strength is significantly improved with the addition of modifiers. However, consid-

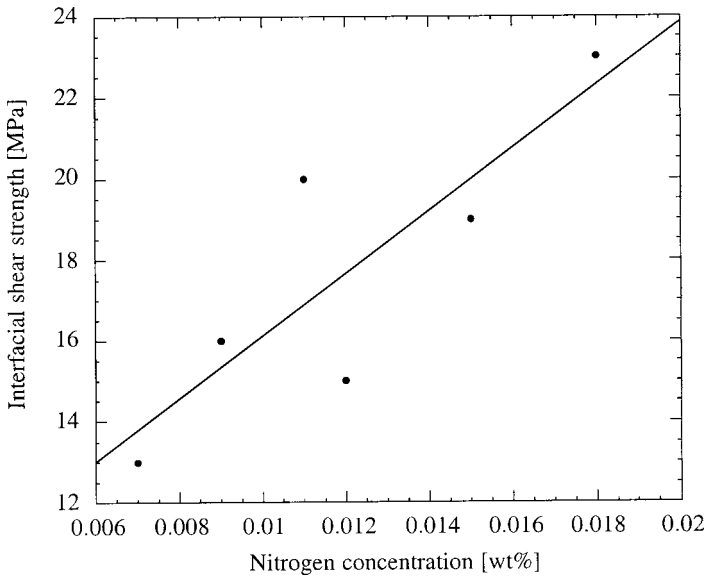


*Figure 4.* The interfacial shear strength (IFSS) of HBPgPP and six different grades of commercial fibres as a function of the nitrogen content in the fibre sizing.

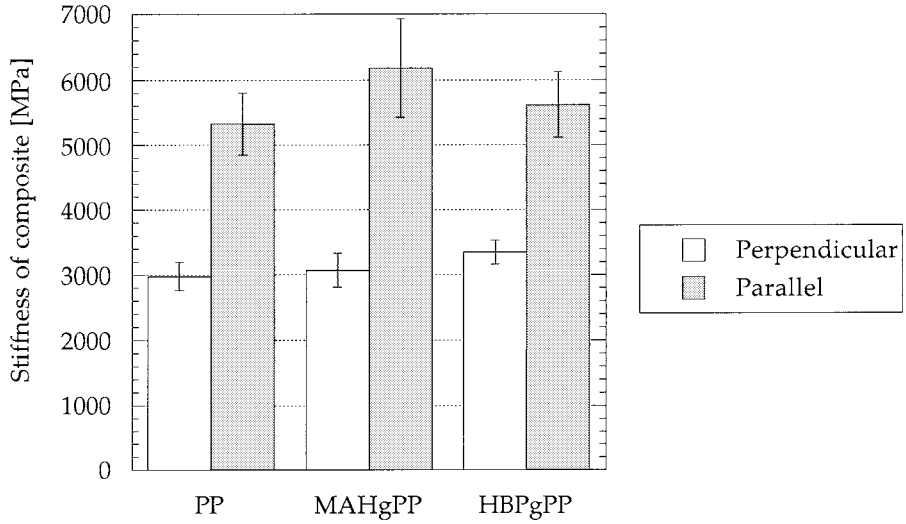


*Figure 5.* The measured stiffness parallel and perpendicular to the preferential fibre orientation of unmodified, MAHgPP modified and HBPgPP modified composites.

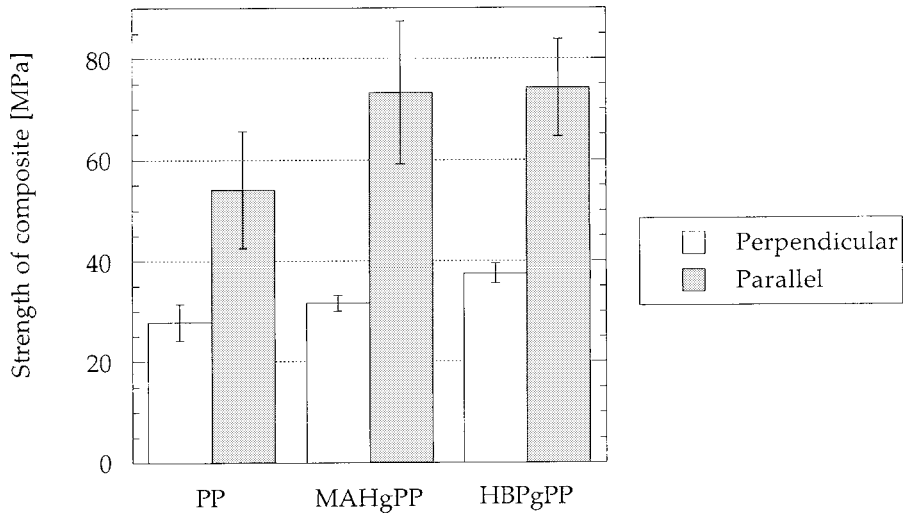


*Figure 6.* The measured strength parallel and perpendicular to the preferential fibre orientation of unmodified, MAHgPP modified and HBPgPP modified composites.

ering the results of the IFSS measurements above, a more pronounced difference between HBPgPP and MAHgPP modified composites was expected.

To evaluate more precisely the effects of matrix modification, the influence of fibre/matrix adhesion and the effects of fibre orientation and fibre concentration must be separated. Using Equation (1), the local state of fibre orientation in a specimen, as expressed by $k_{\mathrm{FOD}}$, can be calculated by inserting the measured stiffness. The fibre efficiency, as expressed by the summation in Equation (4),

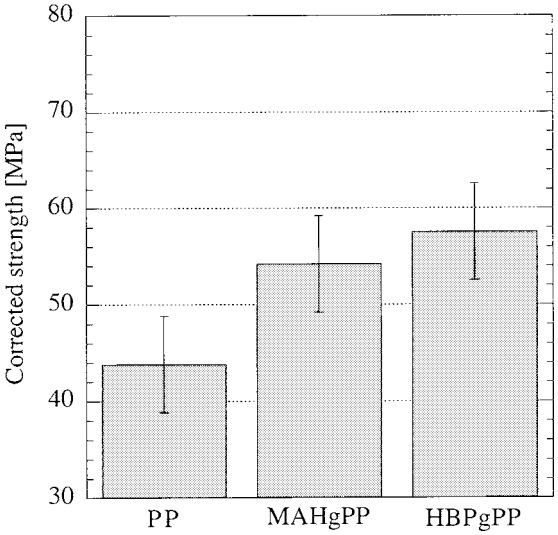


*Figure 7.* Measured strength values of unmodified, MAHgPP modified and HBPgPP modified composites, normalised to in-plane random values.

can equally be calculated by inserting the measured strength and the calculated local fibre orientation ($k_{FOD}$) into Equation (4). By reinsertion of the expressions for the measured fibre orientation factor and the measured fibre effectiveness into Equation (4), the following expression for the corrected composite strength can be derived. This expression thus uses the "measured" fibre effectiveness in conjunction with a predetermined fibre orientation factor to predict a corrected strength value for a composite with a given fibre orientation distribution:

$$\sigma_c^{corr} = \sigma_m V_m + k_{FOD}^{nom} V_f^{nom} E_f \frac{(\sigma_c^{meas} - \sigma_m V_m)}{(E_c^{meas} - E_m V_m)}, \tag{6}$$

where $\sigma_c^{meas}$ is the measured composite strength and $E_c^{meas}$ is the measured stiffness. The average corrected strength values of the composites with a $k_{FOD}^{nom}$ of 0.38 and a $V_f^{nom}$ of 0.13 are shown in Figure 7. In comparison to the measured strength values, a more pronounced difference between the composites is observed. The higher values for HBPgPP compared to those for MAHgPP are attributed to its higher functionality and to the fact that HBPgPP contains several PP chains. This leads to the grafting of more PP chains at the fibre surface and subsequently a higher IFSS and composite strength.

The composite strength can also be determined analytically using Equation (4), if the IFSS and the fibre length distributions are known. The measured fibre length distribution of the materials is shown in Figure 8. The fibre lengths are in the range of the critical fibre length and both terms in Equation (4) must thus be taken into account. The interfacial shear strength in the composite is assumed to be equal to the IFSS value measured by the microbond pull-out test. The results from this

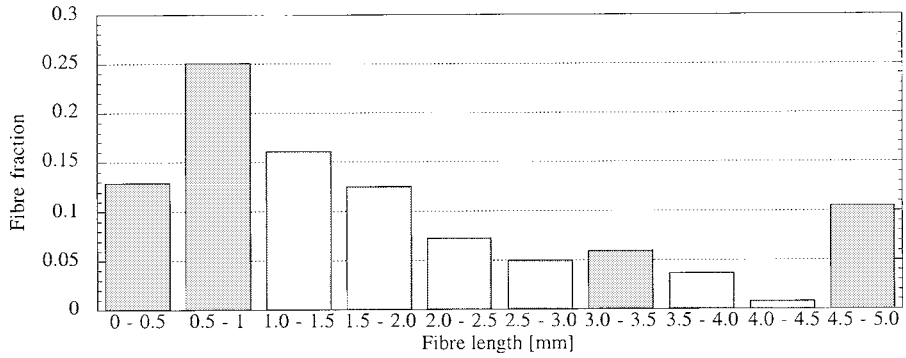


*Figure 8.* The measured fibre length distribution.

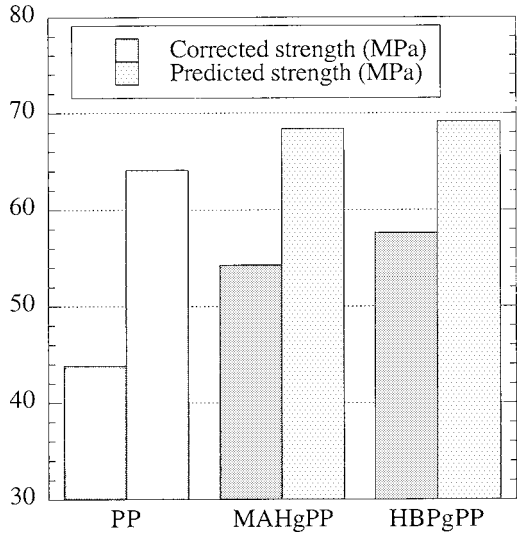


*Figure 9.* The predicted strength of composites, having an in-plane, random fibre orientation distribution, based on the measured IFSS and fibre length distribution.

analysis are shown in Figure 9. It can be seen that the strength values predicted using the microbond IFSS results are higher than the previously determined strength values of Figure 7. This is partly attributed to defects originating from the composite manufacturing process, such as voids, fibre clusters and fibre flaws, which substantially decrease the composite strength. Another factor could be that the true IFSS in the composites is lower than those measured during the microbond pull-out test due to differences in cooling rates. This has earlier been observed by Moon, who studied the influence of cooling rate on the IFSS between PP and glass fibres using the same microbond pull-out technique [22]. Samples cooled at slow rates yielded a 40% lower IFSS than samples cooled in air. The composites in this study were cooled in a mould at a temperature of 70°C while the microbond pull-out samples were cooled in air. Hence, the composites had a much slower cooling rate

and should therefore, according to the results of Moon, have a lower IFSS. The absolute IFSS difference between the microbond test and the composites should be equivalent for unmodified and modified materials, since it is only due to cooling rates effects. An equal decrease of the IFSS, which could thus be as high as 7 MPa here for all composites, would lead to an increased relative difference between the unmodified, MAHgPP-modified and HBPgPP-modified microbond samples. This explains the more pronounced effects of PP-HBP in the composites as compared to the microbond measurements.

## 5. Conclusions

The interfacial shear strength (IFSS) between Polypropylene (PP) and glass fibres (GF) can be substantially improved by adding reactive modifiers to the matrix. Hyperbranched polymer grafted PP (HBPgPP) was shown to be more effective in improving the IFSS compared to maleic anhydride grafted PP (MAHgPP). This was attributed to the fact that HBPgPP grafted more PP chains to the fibre surface due to a higher diffusitivity. The IFSS of HBPgPP modified PP/GF systems was shown to strongly depend on the amine content of the glass fibre surface treatment, indicating that the fibre/matrix adhesion originates from amine-HBP reactions.

It has been shown that the composite strength depends on four factors; the IFSS between the matrix and the fibre, the fibre length, the fibre orientation and the fibre content. The two latter factors vary strongly from sample to sample. Hence, to evaluate the true effects of matrix modification and fibre surface modification these two factors have to be normalised. The normalised composite strength followed the same trends as the IFSS, with HBPgPP modified composites having the highest strength and unmodified composites having the lowest strength. The normalised composite strengths were lower than those predicted using the measured fibre length distribution and IFSS values. This was attributed to defects, such as voids, fibre clusters and fibre flaws originating from the composite manufacturing process, as well as to differences in the IFSS in the composite compared to those obtained by the microbond pull-out tests. Indeed, the measured composite strength values suggested lower IFSS values but larger relative differences between unmodified, HBPgPP modified and MAHgPP modified composites. This was due to higher cooling rates resulting from the microbond pull-out manufacturing process compared to the composite manufacturing process.

Finally, it was shown that the effect on the composite strength of an IFSS increase by matrix modification strongly depends on the fibre length. For very short fibres, their contribution to the composite strength is reduced and thus the IFSS is also lower. For very long fibres, on the other hand, the effect of fibre strength is more important than that of the IFSS. It is thus only over a certain range of fibre lengths that the IFSS plays an important role.

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