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The incorporation of carbon nanofibres to enhance the interlayer adhesion of hot compacted single-polymer polypropylene composites

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ABSTRACT

The current work is a major extension of two very different studies carried out previously to investigate factors that affect the peel strength of single-polymer composites produced by the Leeds hot compaction process. First, it was found that the peel strength was significantly increased by introducing interleaved films, of the same polymer, between the layers of woven oriented tapes that make up the composite. Secondly, it was shown that incorporation of carbon nanofibres (CNF) into the oriented tapes prior to hot compaction could also increase peel strength.

In the present study we have investigated the amalgamation of these two approaches, in particular to see if there are synergistic advantages in the combination. Samples were produced with and without interleaved films, and with and without carbon nanofibres, located either in the oriented polypropylene tapes, in the interleaved film or in both. Maximum peel strength was achieved with the combination of the interleaved film and the incorporated nanofibres, but importantly this could be achieved with the CNF located only in the film. This has significant processing and performance advantages as the incorporation of CNF into the oriented tapes tends to limit the drawability of the polypropylene due to internal voiding around the particles.

Scanning electron micrographs of the hot compacted composites show a strong correlation between the observed damage on the peel surfaces and the measured peel loads. It is shown that the peel load is dependent on the fraction of melted matrix at the interface and hence the interleaved films give additional matrix material at this point. It is also shown that the incorporation of CNF promotes fibrillation, and so increases the amount of energy absorbed during peeling.

1. Introduction

The Leeds hot compaction process can be widely applied to oriented polymers for the production of polymer/polymer composites [1,2]. In this process, an assembly of oriented fibres or tapes (often in the form of a woven cloth) is taken, under a small constraining pressure, to a critical temperature, where a thin skin on the surface of each oriented element melts. On cooling, this skin recrystallises to form the matrix of a single-polymer composite, with the remaining oriented fraction acting as the reinforcing phase. The virtue of this technique is that the matrix phase is produced around each fibre by melting and recrystallisation, negating the need for infiltration of a second matrix phase.

Capiati and Porter [3] first described the concept of single-polymer composites, using polyethylene filaments and polyethylene powder with different melting points. Other techniques reported in the literature include film stacking, powder and solvent impregnation [4–8], bi-component tapes with an outer, lower melting point layer [9] and pressure controlled melting [10]. The hot compaction process is distinct from the majority of these methods, in that only a single component is used.

In typical fibre reinforced composites, the volume fraction of the reinforcement phase is normally a maximum of 50–60%. In the hot compaction technique, since the matrix phase is produced from the reinforcement fibres, a higher volume fraction of reinforcement is achievable. Analysis of the structure [1,2] has shown that the optimum properties of the final composite are achieved at 30% of melting, i.e. 70% oriented phase remaining.

A sample is produced by compacting layers of woven PP tapes in a laminate-type structure. Whilst there is excellent bonding within each of the woven layers, since only the outer skin of the tapes is melted, there is only a thin layer of matrix material, a few microns thick, between each woven layer. Thus the interlayer adhesion, which can be quantified by measuring the force required to peel apart the layers, is of crucial importance.

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It was previously reported that the incorporation of Carbon Nanofibres (CNF) into hot compacted polypropylene (PP) (without interleaved films) resulted in an improved peel strength [11]. Another study [12] has shown the advantage of using unfilled thin film layers, of the same polymer, interleaved between the woven tape layers, resulting in greatly enhanced peel strength since the film layer introduces more matrix material at the point required – the interlayer region.

The thrust of the present paper is to report the enhancement of peel strength by combining these two approaches and show that there is a synergistic effect from doing so. Hot compacted samples have been produced to include possible permutations of incorporating the CNF into the drawn tape phase, interleaved film layers or both. It is shown that major improvements in the interlayer adhesion are achieved by incorporating the CNF. Crucially, it is shown that the high peel loads can be attained by incorporating the CNF into the interleaved film layers, but not necessarily into the oriented woven tapes, allowing potentially higher drawn tapes to be used. Scanning electron microscopy (SEM) has also been used to investigate the correlation between measured peel load and damage to the sample.

2. Experimental

2.1. Materials

The polypropylene (PP) used in this work was a homopolymer with a weight average molecular weight (M_W) of 360,000 g/mol, number average molecular weight (M_n) of 78,100 g/mol, density of 910 kg m^{-3}, and a peak melting point of 163 °C.

Vapour-grown carbon nanofibres (CNF) were obtained from Pyrograf Products Inc, a division of Applied Sciences Inc, Cedarville, OH, and are designated as Pyrograf III™ PR-19-PS-LD by the manufacturer. These are pyrolytically stripped fibres, which removes polyaromatic hydrocarbons from the surface of the fibre.

CNF were blended into PP using a twin-screw extruder, with length to diameter ratio of 40. A loading of 10% w/w CNF, with a blending temperature of 200 °C and a screw speed of ~70 rpm was used. These blending conditions were shown to be the optimal for the PP and CNF used, ensuring good dispersion of the CNF throughout the PP matrix, whilst retaining maximum fibre length [13]. Both the PP and blended PP/CNF material were melt-spun into tapes and nominally drawn to 11.5λ at a drawing temperature of 170 °C, in an air oven. Higher draw ratio CNF-filled tapes could not be produced continuously due to failure of the tapes during the drawing process. Thin films of PP and PP/CNF, blended in the same way, were produced in a small-scale blown film line at the University of Bradford.

2.2. Hot compaction

To obtain an initial assessment of the hot compaction process, in terms of mechanical properties and optimum compaction temperature, unidirectional samples (~55 mm^2 and 0.5 mm thick) were made by winding the tapes around a metal picture frame, which was then placed under pressure in a hot press and taken to the compaction temperature for a set dwell time (5 min) before fast cooling. These unidirectional samples allowed the optimum compaction temperature to be determined (Fig. 1).

For the purposes of the present study, which was directed at improvements in peel strength, it was necessary to produce hot compacted samples with balanced properties, as used in the commercial process to produce these polypropylene composites (Curv™) [14]. This was achieved using a flat (no crimp in the cloth) hand-woven fabric which was produced on a hand loom. The woven cloth was layered, and the assembly was then placed under pressure in a hot press and taken to the compaction temperature for a set dwell time (5 min) before fast cooling. A compaction pressure of 4.9 MPa was used throughout for both the unidirectional and woven samples. Woven samples were approximately 80 mm × 80 mm and 0.5 mm thick, and were produced both with and without interleaved film layers between the woven tape layers.

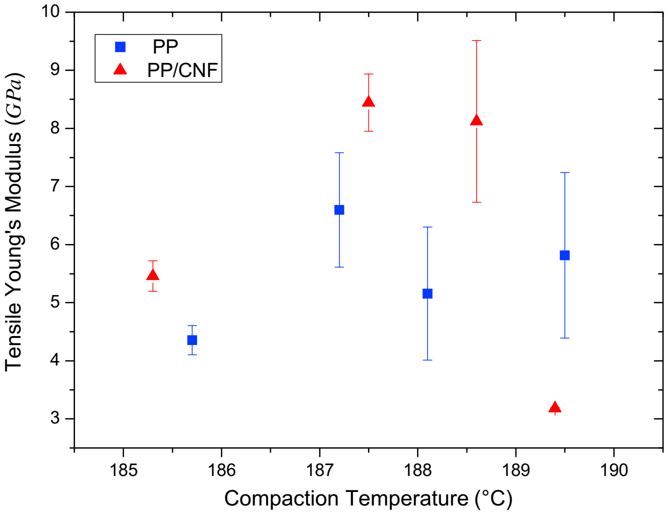

Fig. 1. Variation in tensile Young's modulus of hot compacted uniaxially aligned PP and PP/CNF tapes with increasing compaction temperature. The optimum compaction temperature is defined as the compaction temperature that yields the maximum tensile modulus.

2.3. Mechanical testing

The Young's modulus of the hot compacted sheets was determined by static tensile testing on an RDP-Howden servo-mechanical tensile testing machine. Dumbbell shaped samples were cut from the nanocomposite sheets, consistent with ASTM D638. Samples were tested at 21 °C, 50%RH, and at a nominal strain rate of 5×10^{-3} s^{-1}.

Sample strain in the tensile tests was determined using a Messphysik video extensometer to track targets painted onto the sample surface. To avoid sample straightening affecting the measurement of Young's modulus, a pre-load of approximately 1.5 MPa was applied.

T-Peel tests (after ASTM D1846) were performed on an RDP-Howden servo-mechanical tensile testing machine. Aluminium foil was placed along one edge of the hot compacted sample, located between the central two layers of fabric, prior to compaction and was used as a starting crack in order to test the sample. Samples were cut directly from the hot compacted sheet in 10 mm wide strips, using a sharp scalpel blade to prevent delamination of the samples, which would lead to potential crack points along the edges of the sample. Peel tests were performed at a rate of 80 mm/min (rate is determined by the length of the peel sample and from previous work [11,15]), at a room temperature of 21 ± 1 °C and RH of 50%. The peel load was determined as the average force required to propagate the delamination of the hot compacted layers for the 10 mm wide strip.

3. Results and discussion

3.1. Mechanical properties of hot compacted sheets

3.1.1. Unidirectional sheets

Fig. 1 shows the variation in tensile Young's modulus of the unidirectional hot compacted sheets, produced from both the

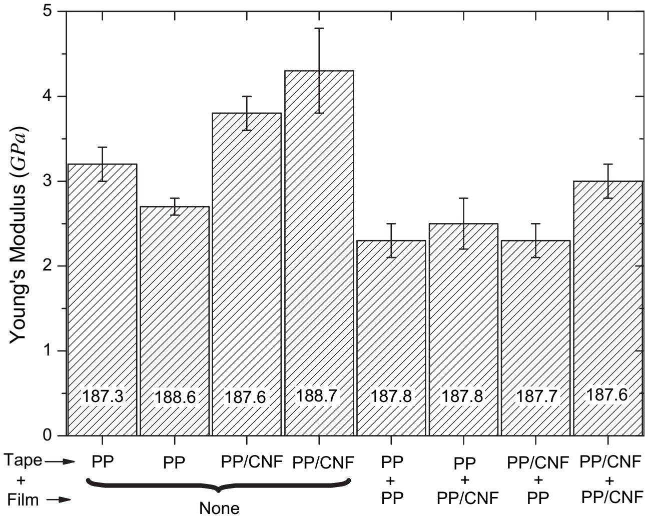

Fig. 2. Tensile Young's modulus for woven hot compacted samples with and without interleaved films. The compaction temperature of each of the samples is indicated.

wound PP and PP/CNF tapes. The optimum compaction temperature, the temperature at which the Young's modulus is at a maximum, for both the PP and PP/CNF tapes, is seen to be approximately 188 °C. This temperature was used as the estimated optimum compaction temperature for the woven samples, where it was not possible to produce enough material to investigate a range of compaction temperatures. However, for the PP and PP/CNF woven tape samples (without interleaved films) samples were produced at ~187.5 °C and ~188.5 °C in order to confirm this optimum compaction temperature. Subsequent samples with interleaved films were produced with a target compaction temperature of 187.5 °C. Fig. 1 indicates that the CNF also have a reinforcing effect on the hot compacted sheets, as has been observed in isotropic material [11,13]. It does appear that the processing window for compaction of the CNF-filled tapes may be wider than that for the unidirectional pure PP samples.

3.1.2. Woven sheets

Hot compacted samples were produced to cover all possible combinations of the woven PP and PP/CNF tapes and interleaved PP and PP/CNF films. The Young's modulus of all the hot compacted samples is shown in Fig. 2.

As expected, the incorporation of the films reduces the Young's modulus of the hot compacted sheets, due to the increased fraction of isotropic material in the hot compacted sheet from the interleaved film. Because the incorporation of the CNF into the PP increases the Young's modulus of both the isotropic and tape phases, the modulus of the compacted sheet is a function of both the Young's modulus and the volume fraction of each phase. In order to understand this, a laminate model has been used to help interpret the Young's modulus results.

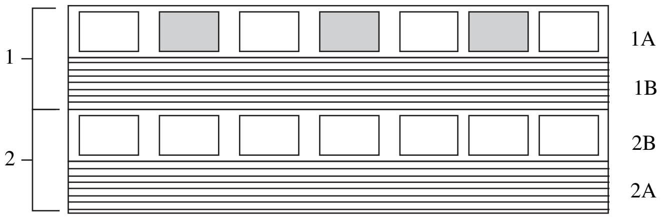

Fig. 3. Schematic representation of a woven hot compacted sheet (unbalanced weave). Each woven layer is split into two layers – warp (A) and weft (B), layer 1A shows the warp tapes in the direction out of the page, 1B is the transverse tapes associated with layer 1A. Layer 2B is the weft tapes, again the direction out of the page, and layer 2A is the transverse tapes associated with layer 2B. The grey tapes indicate the difference from a balanced weave, where the number of tapes in each direction is equal, and an unbalanced weave where the grey tapes are not present, i.e. there is a gap.

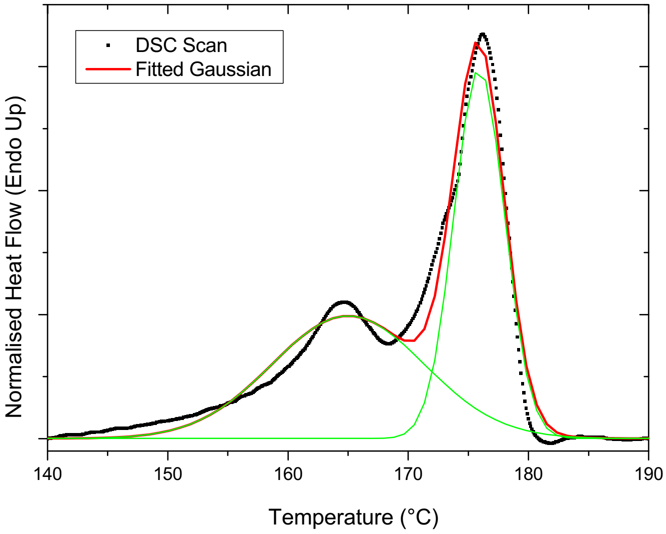

Fig. 4. DSC scan of woven PP/CNF tapes with interleaved PP/CNF film compacted at 187.6 °C. The isotropic melting peak can be observed with peak melting temperature ~163 °C. Gaussian peak analysis was used to fit the DSC scan (■), composed of two Gaussian peaks (🟩), to fit the whole scan (🟥).

Modulus measurements were made to confirm that the excellent properties of hot compacted PP were not lost due to the incorporation of the film layers. Previous work [16] has shown that it is possible to obtain high peel strengths by using very high compaction temperatures, however this results in significant loss of properties, since a greater proportion of the oriented tape core is lost. The aim of this project, therefore, was to obtain high peel strength, without undue loss of mechanical properties.

3.2. Laminate model

The Young's Modulus of these sheets can be predicted from the modulus of the composite layers. Although this could be done using the properties of the individual tapes, this may over-predict the modulus of the compacted sheet since it does not take into account any morphological changes due to the compaction process. The method which has therefore been adopted is to use the properties of a uniaxially aligned composite ply made under the same conditions (i.e. same temperature and pressure to obtain the same percentage of melting), as determined in Fig. 1.

Because the fabric used in this study was hand-woven, the number of tapes per unit area in the warp and weft directions was not equal, i.e. the fabric was unbalanced. This arises because the fabric used had gaps between the warp tapes (the grey areas in layer 1A), whereas the weft tapes were tightly packed, and hence

Table 1
Comparison of measured Young's modulus and that predicted using the laminate model.

Tape	Film	T_C (°C)	Predicted Young's modulus (GPa)	Measured Young's modulus (GPa)
PP	None	187.3	3.4 ± 0.4	3.2 ± 0.2
PP	None	188.6	3.3 ± 0.4	2.7 ± 0.1
PP/CNF	None	187.6	4.5 ± 0.6	3.8 ± 0.2
PP/CNF	None	188.7	4.0 ± 0.5	4.3 ± 0.5
PP	PP	187.8	2.9 ± 0.4	2.3 ± 0.2
PP	PP/CNF	187.7	3.0 ± 0.2	2.5 ± 0.3
PP/CNF	PP	187.8	3.0 ± 0.3	2.3 ± 0.2
PP/CNF	PP/CNF	187.6	3.3 ± 0.3	3.0 ± 0.2

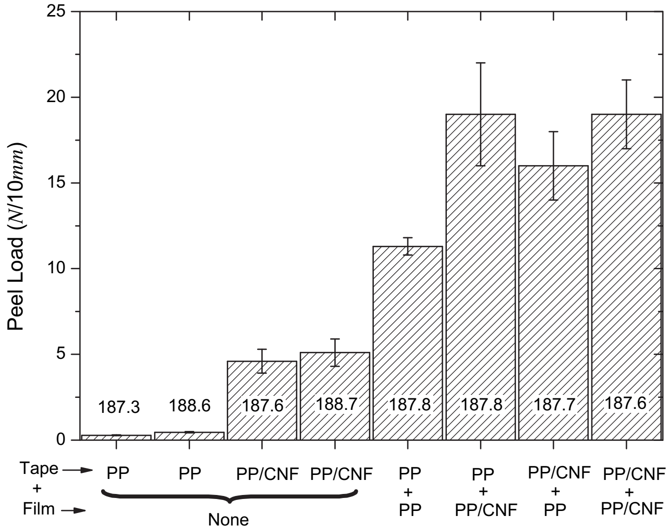

Fig. 5. Interlayer adhesion (peel strength) for woven hot compacted samples with and without interleaved films. The compaction temperature of each of the samples is indicated.

there were more tapes per metre in the weft direction than the warp. During the compaction process the woven layers were stacked in a 0–90 configuration, so that the mechanical properties were balanced in the plane of the compacted sheet. When modelling the Young's modulus of the sheet, it is important to take this imbalance into account. Fig. 3 shows a schematic of the layered compacted sheet. In this figure, each woven layer is split into two component parts – that of the warp tapes (A) and that of the weft tapes (B).

For a *balanced* woven hot compacted sheet, the composite Young's modulus would be:

$$E_C = \frac{E_{\text{long}}}{2} + \frac{E_{\text{trans}}}{2}$$

where E_{long} is the Young's modulus of a uniaxially aligned compacted sample and E_{trans} is the transverse Young's modulus of the aligned layer.

In the woven hot compacted sheets produced in this study, the number of tapes in the warp direction was approximately half that of the number of tapes in the weft direction. Therefore, in layer 1A, if the Young's modulus is measured in the direction of the warp tapes (out of the page), then there are only half the number of tapes taking the applied load. However, the layer must have the same cross-sectional area as layer 2B, where there are double the number of tapes in the direction of the applied load, thus the tapes shaded grey are in fact matrix material in between the tapes. Therefore the Young's modulus of layer 1A is given by

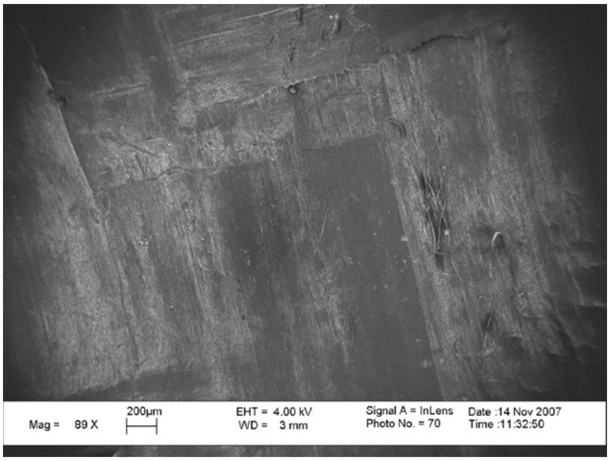

Fig. 7. SEM micrograph of peel surface for hot compacted woven PP/CNF tapes at a compaction temperature of 187.6 °C. There was no interleaved film in this sample.

$$E_{1A} = \frac{E_{\text{long}} + E_{\text{matrix}}}{2}$$

Since the ply deforms under isostrain conditions, the Young's modulus of the entire composite (all four layers) follows the rule of mixtures:

$$E_{\text{ply}} = \frac{1}{4}(E_{1A} + E_{1B} + E_{2B} + E_{2A})$$

and

$$E_{\text{ply}} = \frac{1}{4}\left(\frac{E_{\text{long}} + E_{\text{matrix}}}{2} + E_{\text{trans}} + E_{\text{long}} + E_{\text{trans}}\right)$$

The Young's modulus in layers 1B and 2A is the transverse modulus of the tapes and layer 2B is a complete layer of tapes. To a good approximation, the transverse modulus of the tapes is the same as the isotropic modulus of the polymer [17], hence

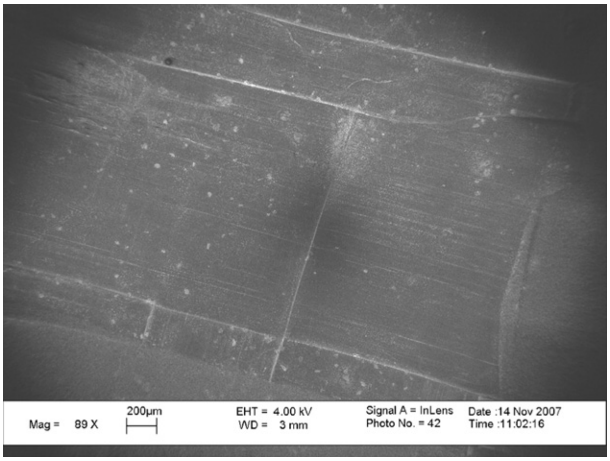

Fig. 6. SEM micrograph of peel surface for hot compacted woven PP tapes at a compaction temperature of 187.3 °C. There was no interleaved film in this sample.

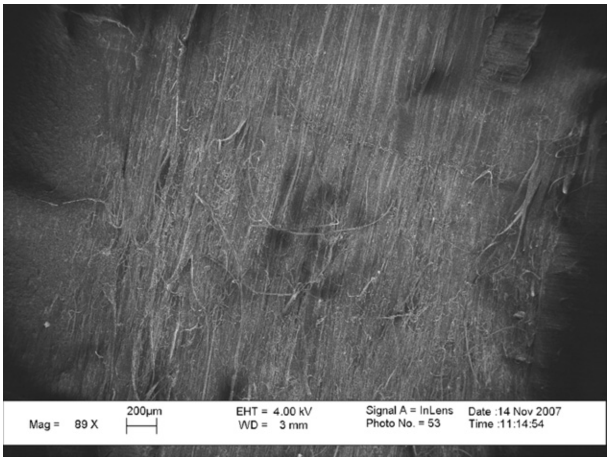

Fig. 8. SEM micrograph of peel surface for hot compacted woven PP tapes with interleaved PP film, compacted at 187.8 °C.

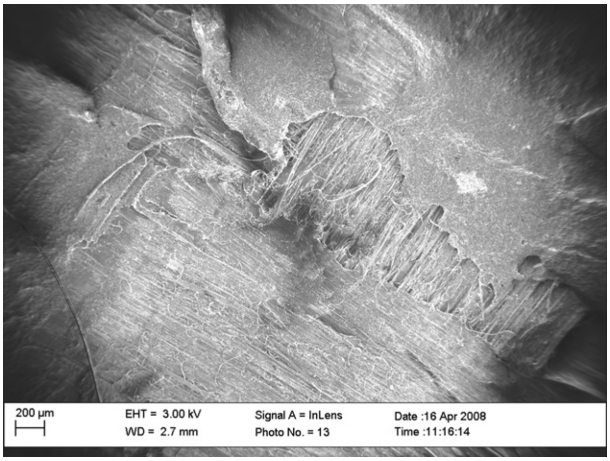

Fig. 9. SEM micrograph of peel surface for hot compacted woven PP tapes with interleaved PP/CNF film, compacted at 187.7 °C.

$$E_{\text{ply}} = \frac{3}{8}E_{\text{long}} + \frac{5}{8}E_{\text{matrix}} \tag{1}$$

where E_{long} can then be directly determined from the tensile Young's modulus of unidirectional hot compacted sheets (Fig. 1).

Equation (1) therefore predicts the Young's modulus from a woven ply within the hot compacted sheet. This can then be used to determine the *effective* Young's modulus of the oriented tapes ($E_{\text{oriented,eff}}$) in the ply, by equating with the more familiar laminate model, Equation (2).

$$E_C = E_{\text{oriented,eff}}\frac{V_{\text{oriented}}}{2} + E_{\text{matrix}}\left(1 - \frac{V_{\text{oriented}}}{2}\right) \tag{2}$$

where V_{oriented} is the volume fraction of material in the oriented phase. This was determined by using differential scanning calorimetry, and then fitting the oriented phase peak with a Gaussian, as shown in Fig. 4. The volume fractions for the oriented phase from the optimally compacted PP and PP/CNF samples at 187 °C (without interleaved films) were then used in Equation (2) to determine the effective oriented tape modulus ($E_{\text{oriented,eff}}$), where E_C was set equal to E_{ply}. This was then used in Equation (2) with the fitted volume fractions for all other samples to determine the composite Young's modulus. The film modulus was assumed to be the same as that of the matrix (isotropic) material.

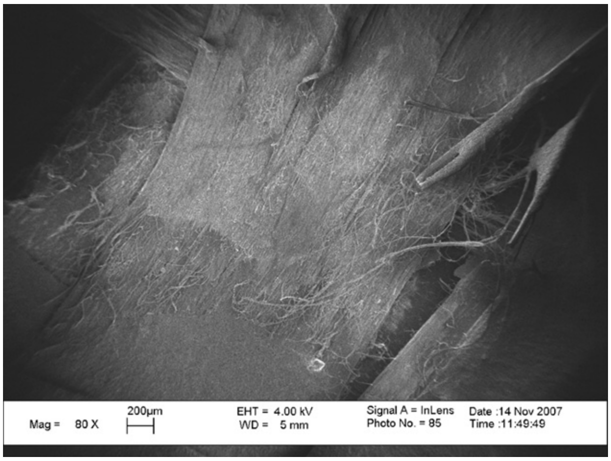

Fig. 10. SEM micrograph of peel surface for hot compacted woven PP/CNF tapes with interleaved PP film, compacted at 187.8 °C.

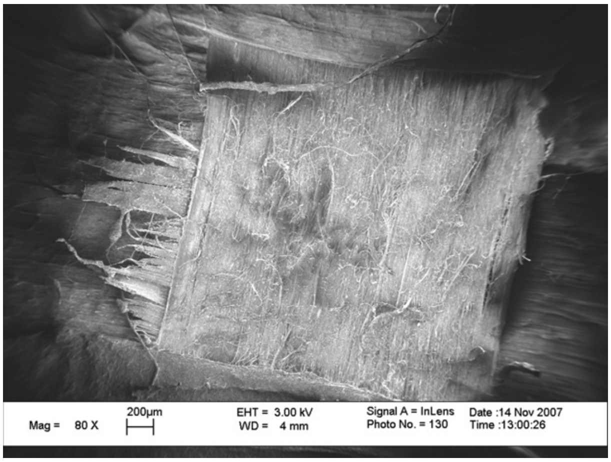

Fig. 11. SEM micrograph of peel surface for hot compacted woven PP/CNF tapes with interleaved PP/CNF film, compacted at 187.6 °C.

Table 1 shows the comparison between the measured Young's modulus and the laminate model calculated modulus. Although the laminate model overestimates the composite Young's modulus, it does follow the same trend in change in modulus, in that the reduction in modulus when the interleaved films are incorporated can be attributed to an increase in the volume fraction of isotropic material. The overestimate can be explained, since although the fabric is a flat-weave, there is some crimp in the tapes. The model is based on the properties of a unidirectional aligned hot compacted sheet with no crimp. The crimp slightly reduces the reinforcement efficiency of the tape, since it no longer lies completely in the plane of the applied load, and also means that the same level of constraint cannot be applied on the woven layers.

3.3. T-peel testing (interlayer adhesion) of woven hot compacted sheets

The peel loads of the woven hot compacted sheets are shown in Fig. 5. The incorporation of CNF into the hot compacted sheets (without interleaved films) results in an order of magnitude increase in the interlayer adhesion, with the measured peel strength increasing from 0.5 N/10 mm[1] for the PP hot compacted sheets to 5 N/10 mm for the PP/CNF hot compacted sheets, similar to those seen in a previous study [11]. The nanoscale dimensions of the CNF mean that they have an extremely large surface area, and it is suggested that the extra surface area means that a great deal more energy needs to be done in order to de-bond the layers during the peeling process, since failure can be generated along the surface between the polymer and the CNF particle.

The introduction of a thin film to the compacted sample also results in an order of magnitude increase in the measured peel strength, and is double that of the woven PP/CNF sheets. As determined by previous studies [12], the morphology of the samples with interleaved films is such that the film melts completely during the compaction process, and completely covers the surface of the woven layers. On cooling, this recrystallises on the woven surface and binds the layers together. The measured peel load is then greater due to the extra work done to peel apart the increased bonded surface area.

The incorporation of CNF into either the tape or film phase of the hot compacted sheet resulted in another increase in the measured

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[1] Peel loads are expressed as $N/10$ mm, since the sample width used is 10 mm.

peel strength of the hot compacted sheet. All three samples showed an average peel load of ~18 N/10 mm, again with the increase most probably due to the extra surface energy along the peel interface provided by the CNF, i.e. de-bonding of the CNF from the PP. This is particularly highlighted by the fact that the sample with PP/CNF tapes and an interleaved PP film showed a similar measured peel load as those samples with CNF in the interleaved films, and suggests that it is the actual interface region, between the tapes and films that is important in the peel process, since in this particular sample the CNF can only have come from the melted skin of the oriented tapes.

Since all three samples show the same peel load it is also believed that failure is no longer only due to de-bonding between the layers, but also between the CNF and polymer, and there is also widespread yielding of the polymer. This supposition is supported by observations of the peel surfaces, discussed below. Internal fibrillation also is promoted by the incorporation of the CNF, since local voiding can occur due to de-bonding at the weak interface between the polymer and the fibre. This effect was seen in CNF-filled drawn tapes [11].

The measured peel load of these composites compares favourably with other hot compacted single-polymer composites, with only hot compacted poly(ethylene terephthalate) multifilaments [18] and nylon 6,6 multifilaments [15] showing similarly high peel loads. The increased peel load in hot compacted PP composites offers significant advantages. In addition, the CNF do not need to be incorporated into the woven tapes and the interleaved film layers require only a fraction of the material needed to melt-spin and draw the tapes. Because CNF do not need to be incorporated into the tapes, this means that higher draw ratio tapes can be used to offset the loss of mechanical properties (tensile Young's modulus, strength) due to the introduction of the isotropic material layer, although this has not been done in this study.

4. Scanning electron microscopy (SEM) of peel surfaces

To help to understand the changes in peel load, the morphology of the peel surfaces was imaged using SEM in order to study mechanisms of peeling. All samples were sputter-coated with platinum (thickness of 3 nm) in argon plasma (before observation) and observed in a LEO1530FEGSEM. The samples were coated along the edges with silver paint to improve sample conductivity.

Fig. 6 shows the peel surface of the woven PP tapes without CNF. Little damage can be seen across the surface of the peel area, corresponding to a low measured peel load. The joins between the tapes can clearly be seen, and there is little or no sign of fibrillation. Significantly more damage can be seen in the peel surface of the hot compacted woven PP/CNF tapes, shown in Fig. 7. There are now clear signs of fibrillation across the surface and several points where areas of the surface have been pulled away during the peel test. When a PP film was introduced in between the woven PP layers, the entire peel surface of the sample shows intense fibrillation, confirming that the increase in peel load due to the incorporation of the thin film is due to improved bonding across the surface and coating of the entire surface.

The peel surfaces of the samples of woven PP/CNF tapes with PP film, woven PP tapes with PP/CNF film and woven PP/CNF tapes with PP/CNF film, shown in Figs. 9–11 respectively, all show a similar amount and type of damage, consistent with the fact that that all three samples had a similar peel load. Intense fibrillation is still visible across the surface, as observed in Fig. 8; however the delamination of the surfaces is no longer smooth and the main forms of damage are tape fracture, tapes pulled away from the surface and pullout of the tapes from the weave. This suggests that the mechanism of failure here is no longer simply the work done in peeling apart the hot compacted layers (delamination). Instead, it is proposed that failure is dependent on the fibrillation behaviour of the polymer, and so cohesive failure of the polymer dominates, limiting the energy absorbed due to failure along the interface between the polymer and the CNF particles.

5. Conclusions

It has been shown that the introduction of a small amount of carbon nanofibres (CNF) can lead to major improvements in the interlayer adhesion of polypropylene single-polymer composites. The advantage of using interleaved films, shown in previous studies [12], has been developed further, with the inclusion of interleaved films into the PP/CNF composites. Single-polymer composites produced with the CNF in either the drawn tapes, in the film layer or in both all had peel loads double that of composites with an interleaved film. Electron microscopy has provided insight into the mechanisms of failure. Observation of the peel surfaces has shown an increasing amount of damage (fibrillation) as the measured peel load increased. The samples produced with CNF in either the tape phase, film phase or both phases showed similar damage, consistent with the fact that all three showed the same measured peel strength. The SEM images also indicated that failure in these samples was dependent on the fibrillation and fracture behaviour of the PP, tape fracture and pullout being observed. Most importantly, it is shown that the CNF do not need to be incorporated into the drawn PP tapes, only into the interlayer films. There are a number of key advantages to this. First, since the tapes can be composed of pure PP, they can be drawn to a higher degree of orientation, thus meaning improved properties of the overall compacted sheets. Secondly, it does not require additional complication of attempting to produce a CNF-filled PP tape on a continuous process.

From the measured peel loads and the evidence in the SEM micrographs, the mechanism for peeling has been examined. Two key factors determine the peel strength: (1) The fraction of melted matrix at the interface between the woven layers. (2) The degree of fibrillation in the tapes or film, leading to failure, an effect which is promoted by the incorporation of CNF, since there is localised de-bonding on the weak PP/CNF interface. At the maximum peel load measured of 18 N/10 mm the SEM micrographs show widespread fibrillation and fracture of the polymer.

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