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P.J. Hine, M.J. Bonner, I.M. Ward, Y. Swolfs, I. Verpoest

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THE INFLUENCE OF THE HYBRIDISATION CONFIGURATION ON THE MECHANCIAL PROPERTIES OF HYBRID SELF REINFORCED POLYAMIDE 12/CARBON FIBRE COMPOSITES

P.J.Hine[a], M.J.Bonner[a] and I.M.Ward[a], Y.Swolfs[b], and I.Verpoest[b]

[a] School of Physics and Astronomy, University of Leeds, Leeds, LS2 9JT, UK

[b] Department of Materials Engineering, KU Leuven, Kasteelpark Arenberg 44, B-3001 LEUVEN (Belgium),

corresponding author p.j.hine@leeds.ac.uk

Abstract

This paper compares and contrasts the properties of self-reinforced polyamide 12/carbon fibre hybrid composites made by three different hybridisation routes, termed intra-yarn, intra-layer and inter-layer. The starting point for each route was to manufacture layers of woven cloth (containing both components), from which the hybrid composites were manufactured using the Leeds hot compaction technique. In all cases, a carbon fibre volume fraction of around 8% was the target.

On balance, the intra-layer hybrids had the best combination of properties, although all three hybridisation routes yielded interesting results. This intra-layer hybrid configuration showed a significant increase in tensile modulus and strength, bending modulus and strength and penetration impact energy compared to a pure self-reinforced polyamide sheet. The only negative aspect was a reduction in the tensile failure strain from 11 to 2%, whereas the ductility in bending was unaffected by the incorporation of the carbon fibres.

1. Introduction

The interest in hybridisation as a method for altering the balance of mechanical properties of fibre reinforced composites continues to grow. This is due to the requirement in a number of industries, such as the automotive sector, for materials that can save weight, while maintaining a good portfolio of mechanical properties such as modulus, strength and impact resistance. In general, no single fibre can (to date) deliver high modulus, high strength and ductility, so hybridisation is an obvious direction for study. Traditional fibre-reinforced composites (usually carbon) offer high specific modulus and strength, but can often suffer from low failure strains (~2% is common) and consequently brittle behaviour. At the other end of the performance scale to these traditional structural fibre composites, are a new class of materials termed self-reinforced polymer composites (SRPC). In these materials, both the reinforcing elements (fibres or tapes) and the matrix phase are polymeric and usually the same polymer. The only difference is that the reinforcing fibres or tapes have preferred molecular orientation, thereby giving substantially improved modulus and strength over a normal isotropic polymer, but without an increase in density. In these materials the modulus and strength are much lower than in traditional structural fibre composites, but the failure strains can reach ~20% and the toughness can be exceptional.

This trade-off between modulus and toughness, often called the modulus/toughness dilemma [1-4], continues to be a major area of research interest, with the continuing objective of developing so called 'hybrid' materials that could combine these two important properties. One popular route to hybridisation is to combine two different fibre types in a composite, often a high modulus but brittle fibre (usually carbon) and a ductile, and often lower modulus, polymeric fibre. A good summary of the current state of the art in hybrid composites can be found in the reviews of Kretsis [5] and more recently Swolfs et al. [1]. Pegoretti et al. [6] described some of the possible fibre combinations that had been reported in the literature at that time, including carbon/Kevlar, carbon/ultra-high-modulus-polyethylene (UHMPE), aramid/UHMPE and UHMPE/glass.

In the majority of these studies the goal was to increase the failure strain/ductility/toughness of high volume fraction carbon fibre reinforced polymer composites, by sacrificing a proportion of the composite modulus and strength by adding a ductile fibre. In this study we have adopted a different strategy, which is to start with an already ductile material, in this case a self-reinforced polymer composite, and add a small amount of carbon fibres (<10%) in order to increase modulus and strength and hopefully not reduce the ductility significantly.

The first reported, purely, self-reinforced polymer composite (SRPC) study is usually considered to be that of Capiati and Porter [7] in 1975, who embedded highly oriented gel spun polyethylene fibres into an isotropic polyethylene matrix. Subsequently, a range of other processing methods have been reported in the literature including utilising bi-component tapes [8-12], film stacking [13-15], injection moulding [16] and a combination of processing techniques to produce nanofibrillar composites [17, 18]. One of the most widely reported techniques for the production of single polymer composites has come from the work at Leeds University, using a process termed hot compaction [19-21]. The difference in this process, compared to the other reported work, is that it utilises only a single starting component, usually an array of oriented thermoplastic fibres or tapes. The research showed that if this array was taken to a chosen temperature close to the melting point, then a fraction of the surface of each oriented element could be selectively melted. On cooling, this molten fraction then recrystallised to form the matrix of the single polymer composite. While the processing window is narrow, it is achievable commercially [22] and has the advantage that good compatibility is achieved between the reinforcement and matrix as the two components are identical. A good summary of the preparation and properties of self-reinforced polymer composites can be found in the reviews of Alcock et al. [23] and Matabola et al. [24].

Self-reinforced polymer composites (mostly based around highly drawn polypropylene tapes - Curv® [25]) are a new developing material and are finding increased commercial usage in areas where lightweight and outstanding toughness are key drivers (for instance Samsonite luggage and Bauer ice hockey boots). However, semi-structural applications, such as in the automotive sector, remain out of reach due to the moderate modulus and strength of these materials and their elevated temperature performance. It is therefore an obvious extension of research into SRPC, to apply the ideas of hybridisation described earlier by adding a much stiffer fibre to these materials. The interest is to investigate the expected trade-off between increasing the modulus and strength of these materials with an associated decrease in failure strain and toughness, which are the unique properties of SRPC. As an illustration of the disparate properties of these two, very different, material types, Figure 1 compares typical tensile stress-strain curves for a carbon fibre/polyamide prepreg tape (unidirectional arrangement) and a self-reinforced polyamide sheet (SRPA12) in a 0/90 configuration. The former material (prepreg tape) shows a strength above 1GPa but a failure strain of ~1% whereas the latter materials (SRPA12) shows a strength of ~100MPa but a failure strain of ~10%.

In their recent review of hybrid composites, Swolfs et al. [1] described three main hybrid configurations for combining ductile and brittle fibres, which they termed inter-layer (or layer-by-layer), intra-layer (yarn-by-yarn) and intra-yarn (fibre-by-fibre). For inter-layer hybridisation, discrete layers of composite sheets, containing the brittle and ductile fibres, are stacked on one another. In intra-layer hybridisation, each layer is comprised of a co-woven mixture of the two fibre types, while in intra-yarn, the hybridisation is done within each individual fibre tow using co-mingled brittle and ductile fibres.

While research on the hybridisation of SRPC is a new research area, recently reported studies have investigated these different hybridisation configurations for different fibre and SRPC combinations. Taketa [26], Fabich [27] and co-workers investigated inter-layer hybridisation by combining discrete layers of a carbon fibre (prepreg) composite and a self-reinforced polypropylene sheet. Taketa et al. found that the failure strain of the hybrid laminate was improved over the pure carbon fibre laminate and this was linked with a compressive pre-strain associated with the shrinkage of the SRPC fraction during consolidation and cooling, while Fabich et al. found some beneficial effects on the impact performance from the hybridisation.

Following on from this study, Swolfs et al. [2, 3, 28] looked at the same material combination, oriented polypropylene tapes and carbon fibres, but in this case in an intra-layer configuration. Here, an oriented polypropylene tape was co-woven with a carbon fibre/polypropylene prepreg tape to produce a co-woven cloth, which was then processed using the hot compaction process to produce hybrid composite sheets. These intra-layer hybrids showed an interesting portfolio of properties. It was found that, at a critical carbon fibre volume fraction of around 8%, a significant increase in modulus could be achieved without seriously compromising the high failure strain and excellent penetration resistance of the pure SRPC.

One suggested way to evaluate the effectiveness of each chosen hybridisation configuration is to assess the measured properties of the hybrid against a chosen micro-mechanical model (often, but not exclusively, the rule of mixtures). Marom et al. [29] proposed such an idea for a range of different property measures. In their recent review paper, Swolfs et al. [1], while warning of the simplicity of this approach, agreed that this approach gives, at least, an indication of the success of any hybridisation. In particular, a ‘positive hybrid effect’ is achieved when the experimentally measured property is found to be greater than predicted by the chosen model. For the results of the intra-layer carbon fibre/PP hybrids described by Swolfs et al. [2], the tensile test results of these hybrids suggested a positive hybrid effect for the failure strain of the carbon fibres (observed as a stress drop in the stress-strain curve), with an increase in the composite tensile modulus in line with model predictions, but with a minimal decrease in the composite failure strain, up to a carbon fibre loading of around 7 volume percent . Swolfs et al. proposed that this was mainly due to the beneficial effect of having intermediate bonding between the layers when using polypropylene, allowing delamination and additional energy absorption to take place. Observation of samples during tensile failure revealed that delamination between the woven layers occurred at the point where the carbon fibre tapes failed (around 2% strain). Such delamination diffused the strain localisation around the carbon tape fracture and so allowing the SRPC fraction to survive and continue to be load bearing until nearly 20% strain. Interesting, and of significant relevance to the current study, the addition of inter-layer PP films during processing both improved the level of inter-layer bonding but also led to more localised damage in the SRPC fraction on failure of the carbon fibres and a significantly reduced ultimate failure strain.

The third hybridisation configuration, intra-yarn, was studied in a recent paper by Hine et al. [30] utilising co-mingled tows of oriented polyamide 12 (PA12) multifilaments and T700 carbon fibres. The study showed that hybrid samples with interesting properties could be made using a carbon fibre fraction of 13% and a traditional hot compaction technique. In this study, and probably due to the much greater adhesion between polyamide and carbon fibres, the hybrid composite did not survive in tension past the breakage of the carbon fibre fraction and so failed at a tensile strain less than 2%. However in bending, the hybrid material showed an increased bending modulus and ductile behaviour to strains of 5% and beyond.

To date, no study has compared and contrasted the three important hybridisation routes on the same material combination. Building on our previous studies on intra-yarn hybridisation of PA12 fibres and carbon fibres described above, we have manufactured inter-layer and intra-layer composites using the same two materials. In addition to the co-mingled yarns used in the previous study, intra-layer samples were manufactured by combining oriented PA12 tapes (manufactured in-house) and PA12/carbon fibre prepreg tapes, while inter-layer samples were made by laminating layers of self-reinforced polyamide 12 sheet (SRPA12) and layers of a polyamide 12/carbon fibre composite. Following the work of Swolfs et al. on polypropylene/carbon fibres SRPC hybrids [28], a carbon fibre volume fraction of 8% was the target for each of the three hybridisation configurations. All the composite samples were submitted to three mechanical property tests: in-plane tensile, in-plane bending and falling weight penetration impact.

2. Experimental

2.1 Hybrid manufacture

2.1.1 Intra-yarn hybridisation

2.1.1.1 Component materials

In co-mingling, each fibre tow is composed of a mixture of the two chosen fibre types (as shown schematically in Figure 2a). In this study, the two types were carbon fibres and Polyamide 12 (PA12) oriented filaments. The co-mingled yarns for this study were produced by Schappe Techniques, France using their patented stretch-breaking technology [31]. In this process, continuous carbon fibres are stretched until they break at what are termed 'natural break points' and then blended with a thermoplastic fibre. The aim is to increase the drapability of the resultant co-mingled tows without compromising mechanical performance.

For this project, these stretch broken tows were supplied in a carbon fibre volume fraction of 13%, which was the lowest that was could be produced commercially (the normal industrial usage is around a 50/50 fraction). Microscopy of the combined tows showed that the mixing and dispersion of the two fibres was excellent. The carbon fibres had a quoted modulus of 240GPa, a strength of 4GPa and a failure strain of 2%. Their length varied between 30 and 70 mm. Since this is well above their critical length, their discontinuous nature should have a negligible effect on the composite modulus and strength. The PA12 filaments had a measured modulus of 2.9GPa, a strength of 300MPa and a failure strain of 11%. In order to produce composite sheets with balanced properties, the co-mingled yarns were woven into cloth using a twill 4 x 4 weave style, as shown in Figure 2b.

2.1.1.2 Intra-yarn hybrid sample manufacture

Composite samples were produced from the woven hybrid cloth using the Leeds hot compaction process. As described in the introduction, the aim of this procedure is to selectively melt a fraction of the oriented PA12 fibres (using a chosen temperature), which then forms the matrix of the resulting self-reinforced hybrid composite. A typical process used was as follows. The layers of woven cloth were placed between soft aluminium sheets (thickness 0.1mm), two layers of silicone rubber to even out the pressure distribution, and then outer brass sheets of 2mm thick. A thermocouple was placed in the centre of the assembly, and this was used for monitoring the temperature throughout the process. The whole assembly was then placed into a compression press set at the required temperature, termed the hot compaction temperature. A pressure of 5MPa was immediately applied and temperature monitoring was started. Once the material reached the required temperature, it was left for a dwell of 2 minutes and then rapidly cooled to 50°C (using circulated water cooling which takes around 3 minutes). Following the previously reported study on intra-yarn hybridisation [30], a compaction temperature of 175°C was considered to be the best choice for manufacturing a sample from the 13% carbon fibre tows.

Parallel studies on intra-layer self-reinforced *polypropylene*/carbon fibre hybrid composites [28] suggested that a carbon fibre fraction of around 8% was a good choice for achieving a balance between tensile, bending and penetration impact performance. As the carbon fibre fraction in the intra-yarn hybrids was increased above this value, the modulus and strength were found to continue to increase as expected, but was accompanied by a fall in the failure strain and a significant reduction in the penetration impact performance. For this reason, a carbon fibre fraction of 8% was the target for the three hybridisation routes investigated in this study.

As a co-mingled tow with 8% carbon fibres was not available (13% was lowest commercially available), the carbon fibres were further diluted in respect of the overall composite sample, by alternating the layers of the woven hybridised cloth with layers of pure polyamide 12 film, made from the same grade of polymer as used in the PA12 multifilaments. It was found that four layers of the PA12 film, and three layers of the woven hybrid cloth (in the lay-up PA12/Hybrid/PA12/Hybrid/PA12/Hybrid/PA12), gave a final carbon fibre volume fraction of 8 ± 1% (see section 2.5 for details of how this was measured). A similar hot compaction temperature of 175°C was used for the manufacture, consistent with the previous studies, in order to achieve only a partial melting of the PA-12 filaments. A typical manufactured sample is shown in Figure 2c, while Figure 2d shows a typical polished cross section from the final hybrid composite. All the relevant details of the intra-yarn hybrids are given in Table 1.

2.1.2 Intra-layer hybridisation

2.1.2.1 Component materials

For intra-layer hybridisation carbon fibre/PA12 prepreg tapes (~40% carbon fibre fraction manufactured by Jonam Composites) were co-woven with oriented PA12 tapes. The carbon fibres were type T700S and had a stated modulus of 230GPa, a strength of 4.9GPa and a failure strain of 2.1%.

To produce the co-woven cloth, oriented PA12 tapes were required. For consistency, these were manufactured using the same PA12 grade as above for producing the PA12 filaments, used in the intra-yarn studies. After extrusion the tapes were then drawn on the Leeds drawing frame to a draw ratio of 4:1, at a temperature of 120°C. The carbon fibre – PA12 prepreg tapes were 5mm wide and 0.20mm thick, while the extruded and drawn PA12 tapes were 8mm wide and 0.10mm thick.

A truly balanced co-woven cloth, as shown by the ideal schematic diagram in Figure 3a, would have a mixture of both tape types in both the warp and weft directions. However, in the current study the co-weaving was accomplished on a hand loom, using a warp comprised of only the oriented PA12 tapes and a weft which could be alternated between the carbon prepreg tapes and the oriented PA12 tapes as required, to achieve the target carbon fibre volume fraction of 8%. Figure 3b shows a part of the hand woven cloth where the warp direction is horizontal. Here the weft tapes alternate between the carbon fibre prepreg tapes and the oriented PA12 tapes.

2.1.2.2 Intra-layer hybrid sample manufacture

Intra-layer hybrid samples were manufactured using the same procedure as described above in section 2.1.1.2. To achieve a balanced composite, four layers of the woven cloth were laid in a 0/90 symmetric configuration $[0/90]_{2s}$ and processed using a compaction temperature of 175°C (no additional layers of isotropic PA12 were used for the intra-layers samples). This configuration assured that there was an equal number of carbon fibre tapes in the 0 and 90 directions. A piece from a typical manufactured sample is shown in Figure 3c, Figure 3d shows the stacking sequence while Figure 3e shows a typical polished cross-section. All the relevant details of the intra-layer hybrids are given in Table 2.

2.1.3 Inter-layer Hybridisation

2.1.3.1 Component materials

For the third and final hybridisation strategy, discrete layers of woven pure self-reinforced PA12 tapes were alternated with layers of impregnated carbon fibre woven cloth (as shown schematically in Figure 4a). The carbon fibre cloth chosen to manufacture the carbon fibre layers, was the spread tow carbon fibre fabric made by Oxeon (TeXtreme®). As can be seen from the picture of this cloth (Figure 4b), the carbon fibres form a large dimension chequerboard pattern, where each square is 20mm x 20mm. In the next stage, impregnated carbon fibre layers were manufactured using this TeXtreme® fabric and the same PA12 film used for other two hybrid configurations. The prepregs were produced at 220°C in between 5 mm thick aluminium plates. Aluminium foil was also added in between, and this foil was treated with Chemlease PMR-90 for an easy release of the prepreg. The cycle time was 10 min, during which the pressure on the stack was alternated between 16 and 0.5 bar to aid impregnation of the high viscosity PA12 into the carbon fibre cloth. The pressure level changed from one to the other every minute. Afterwards, the entire stack was removed from the hot stage and inserted into the cold stage, where it cooled down to room temperature in about 3 minutes.

2.1.3.2 Inter-layer hybrid sample manufacture

Inter-layer hybrid samples were made by interleaving the impregnated carbon fibre layers with layers of pure woven PA12 tapes (hand woven to a plain weave as shown in Figure 4c) with a compaction temperature of 175°C. As this sample utilises discrete layers, there is a choice over where the carbon fibre layers are located. To maximise bending properties, the impregnated TeXtreme® layers in this current study were placed on the surface of a five layer composite: TeXtreme®/PA12 tapes/PA12 tapes/PA12 tapes/TeXtreme® which gave a final carbon fibre volume fraction of 8 ± 1%. Figure 4c shows a typical sample, while Figure 4e shows a polished cross-section. Table 3 shows the additional details of the inter-layer hybrids. In terms of the carbon fibre properties, the PA12 properties and the volume fractions of the various phases, these are the same as shown in Table 2 for the intra-layer hybrid configuration.

2.2 Mechanical tests

2.2.1 Tensile

The tensile tests were carried out in accordance with ASTM D3039 using an RDP servo-mechanical test machine at a temperature of 20°C and 50% RH. Samples were left to equilibrate at these conditions for 5 days before testing.

The tensile test samples were 10 mm wide and 150 mm long. The gauge length was set to 65 mm and the sample strain was measured in the middle 15 mm of each sample using a Messphysik video-extensometer. The testing speed was 5 mm/min, giving a nominal strain rate of 10^{-3} s^{-1}. Five samples were tested for each condition using a 5kN load cell. With respect to the interlayer hybrid, the specimen width is less than the unit cell size. However, in many ways, spread tow fabrics behave more like a UD laminate than a woven fabric. UD laminates do not have any issues with unit cell sizes. This does not imply that spread tow fabrics do not have this issue, but they are less sensitive to it.

2.2.2 Bending

The three point bending tests were carried out in accordance with ASTM D6272, at a temperature of 20°C and 50% RH. The bending test samples were 10mm wide and 70mm long. Modulus measurements were made using a span/thickness ratio of 25:1 (span = 30mm) while stress-strain curves to break were made using a span/thickness ratio of 16:1 (span = 19mm) as recommended by the testing standard. The testing speed was chosen to be 5mm/min, to give a nominal maximum strain rate (on the sample surfaces) of 10^{-3} s^{-1}.

2.2.3 Penetration impact

Falling weight penetration tests were carried out according to ASTM5628. All samples were clamped between metal holding plates using an air pressure of 0.7 MPa. The plates had an opening diameter of 76mm and were covered in rough emery paper to improve clamping. Impact was via a hemispherical striker with a diameter of 12.7mm, connected to an impact mass of 25kg. The drop height of the mass/striker was chosen so that the impact speed was 3.33 m/s (200m/min) and a piezoelectric load cell in the striker allowed the force-time curve during impact to be measured.

It is well known that the penetration impact energy can be significantly dependent on the sample thickness, so most of the samples were made to be of a comparable thickness of 1.1 ± 0.1 mm. The only exception was the intra-yarn sample at 8% carbon fibre volume fraction. Diluting the carbon fibre fraction of this sample by adding the PA12 films resulted in a sample of 2.5mm thick. The measured impact energies were therefore quoted in terms of the measured energy divided by the sample thickness. As with previous studies [32-34], we have integrated the force/time curve until the point at which the peak load drops to 50% of its maximum value.

2.3 Volume fraction determination

The volume fraction of carbon fibres in the various hybrid composites was measured using the burn off test (ASTM D2184). Each sample (with a weight of around 2g) was placed into a crucible and then into a furnace. The furnace was set to 450°C, dwelled for 4 hours and then cooled back to room temperature. While the standard recommends a temperature of 565°C, previous studies showed this could volatilise carbon fibre as well, hence the use of the lower temperature. A measurement of the weight before and after the burn off allowed the weight fraction to be calculated, and hence a volume fraction assuming a carbon fibre density of 1800 kg/m³ and a PA12 density of 1010 kg/m³. Repeated measurements suggested an accuracy of ± 1 for all the measured values (in effect an actual error of ~ ± 10%).

2.4 Classical laminate theory prediction

The classical laminate theory was used to predict the modulus of the hybrid composites, taking into account their crimp. For the PA12 fibres/tapes, the measured longitudinal modulus was used. The transverse modulus, longitudinal shear modulus and longitudinal Poisson's ratio were estimated to be 1 GPa, 1 GPa and 0.4 respectively [35]. For the carbon fibres, the longitudinal modulus was taken from the manufacturers' data sheet, whereas the other engineering constants were taken from [36]. For the PA matrix, a modulus of 1 GPa and Poisson's ratio of 0.4 was assumed. The engineering constants of the unidirectional carbon fibre prepreg and SRPA12 plies were obtained by using the relevant fibre volume fraction and applying the Chamis' formulae [37].

All these data were used as unidirectional ply properties for the classical laminate theory. $(0_{CFPA}/0_{SRPA12}/90_{CFPA}/90_{SRPA12})_s$ laminates were created, where the subscript CFPA indicates the unidirectional carbon fibre prepreg ply. The thickness of the plies was chosen to correctly reflect the relative volume fractions. The overall carbon fibre volume fractions were varied from 7% to 9% to obtain the spread on the modulus. The modulus was calculated at an off-axis angle that corresponded to the average out-of-plane angle measured from the optical microscopy images (see Figs. 2d, 3d and 4d). This was 7.0°, 3.5° and 1.4° for the intra-yarn, intra-layer and inter-layer hybrids, respectively. The averaging of the angle is an approximation as the modulus-angle dependency is strongly non-linear. For the small angles used here, this approximation would lead to a slight overestimation of the modulus.

3. Results

3.1 Intra-yarn hybrid

3.1.1 Tensile tests

Figure 5a shows a typical tensile stress-strain curve for a sample with a carbon fibre volume fraction of 8 ± 1%. The stress-strain curve is linear to failure, with a failure strain of 1.4 ± 0.1%. A typical result is also shown from the previous study on intra-yarn hybrids, which had a carbon fibre fraction of 13% so made without the additional interlayer film used for the 8% sample. The results show that although the initial modulus is similar for the two materials, the 8% sample, surprisingly, has a higher strength, although a slightly lower strain to failure. Average results for the two different samples are shown in Table 4. The reason for this difference could be that the addition of the inter-layer film for the 8% sample (required to dilute down the carbon fibre fraction) has the effect of adding additional matrix and improving the consolidation of the composite, as will be further discussed in the section on the bending tests.

The CLT prediction of the tensile modulus was 10.2 ± 0.9 GPa, which matches well with the measured value of 10.0 ± 0.3. Without incorporating the crimp in the CLT predictions, the tensile modulus was predicted to have been 11.6 ± 1.1 GPa. This proves that incorporating the crimp is essential for accurately predicting the tensile modulus.

3.1.2 Bending tests

Bending tests were carried out on the 8% carbon fibre intra-yarn plates, and a typical stress-strain curve is shown in Figure 5b. As can be seen from the results in Table 4, the measured flexural modulus was very similar to the tensile modulus, and hence again in good agreement with the predictions from modelling.

More interestingly, the failure behaviour was different for the lower carbon fibre fraction. This sample failed in a brittle manner once the maximum stress of around 200MPa was reached, whereas, in the previous study, the higher carbon fibre fraction sample (13%) failed in a more progressive manner once the peak force had been passed. It is perhaps a further indication that the 8% carbon fibre hybrid sample is well consolidated and homogeneous due to the increased matrix material delivered by the incorporation of the inter-layer film. As was shown in a previous study on PP based hybrids [28], the addition of more matrix by adding PP films as interleaves, increased the inter-layer bonding significantly. This reduced the failure strain and led to more brittle behaviour by localising the failure.

For the higher carbon fibre volume fraction (13%), we can therefore speculate that there is insufficient isotropic matrix produced during the hot compaction process to fully consolidate the composite, and hence some carbon fibres are less well bonded to the PA matrix. When reaching higher strains, the weaker fibres gradually start fracturing, but the strain localisation (normally leading to early catastrophic failure) is less severe than in the case in well-bonded fibres. During flexural testing, this leads to a gradual decrease of the modulus, first in the outer layers of the composite at the tensile side, and moving inwards at higher bending loads, in this way leading to progressive failure behaviour.

3.2 Intra-layer hybrid

3.2.1 Tensile tests

For the intra-layer hybrid samples the carbon fibre prepreg tapes were only in the weft direction (Figure 3b), so a $[0/90]_{2S}$ configuration was used to produce a balanced sample. Figure 6a shows a typical tensile stress-strain curve for an 8% carbon fibre intra-layer sample compared to the same carbon fibre fraction intra-yarn sample. Each sample was tested with the carbon fibre tapes in the outer layer parallel to the testing direction. The modulus of the two samples was very similar (as might be expected) although the failure strain, and hence the strength, is significantly higher for the intra-layer sample.

The lower out-of-plane crimp in the intra-layer hybrids has been taken into account in the modelling predictions, which were again made using the philosophy described above for the intra-yarn hybrid. Table 2 details the various input parameters for the modelling, including the constituent properties and the weave architecture. This gave a prediction of the in-plane modulus of 11.0 ± 1.0 GPa compared to the measured average value of 9.2 ± 1.0 GPa. The measured modulus is lower than that predicted by the model. This is likely due to some additional observed in-plane misalignment (as seen in Figure 3c), as the CLT predictions only captured the out-of-plane misalignments which were measured from microscopy of a through thickness section.

For the PA12 based intra-layer hybrid, it is clear that in tension the hybrid is so well bonded that when the carbon tapes break, the amount of stored energy at that point is enough to completely fracture the remaining SRPA12 fraction. To further investigate this aspect, a sample was made by attaching a piece of carbon fibre-PA prepreg tape to the external surface of a pure self-reinforced PA12 sheet, as shown schematically in Figure 7. The width of the prepreg tape was chosen so as to give a volume fraction of

~4% carbon fibres in the testing direction (similar to the amount of carbon fibre in one direction in the intra-layer 0/90 hybrids). Figure 7 shows a typical tensile stress-strain curve for this combination. In this case, when the carbon fibre prepreg tape broke, at around 2% strain, the remaining SRPA12 fraction survived and then continued to be load bearing until 11% failure strain (which is typical for these pure SRPA12 materials). So if the carbon fibre tapes are located on the outside of the sample, and so the components can act independently, then the tensile behaviour is very similar to the SRPP/carbon fibre hybrids from the previous study [2]. In those SRPP/carbon fibre hybrids, delamination between the layers had the same effect of 'separating' the two components.

3.2.1 Flexural tests

Figure 6b compares typical flexural stress-strain curves of the intra-layer hybrid and the intra-yarn hybrid, both with a carbon fibre volume fraction of 8%. As the intra-layer sample only contains carbon fibres in one direction in the outer layers, a choice has to made in which in-plane axis to test. For this study the samples were tested with the prepreg tapes in the outer layers in the direction of bending. While the flexural modulus of the intra-layer sample is significantly lower than the intra-yarn hybrid, the failure strain is at least four times larger and is therefore significantly more ductile. The lower flexural modulus could be due to the different distribution of carbon fibres through the thickness in the two hybrids. In the intra-yarn sample the out-of-plane shear is equal over the whole thickness (as the carbon fibres are evenly distributed), whereas in the intra-layer samples, there are regions of high carbon fibre fraction and regions where there are no carbon fibre tapes, leading to less reinforcement in these regions and leading to a lower overall bending modulus. As the equations assume pure bending, this could cause unwanted artefacts in the modulus calculation. Table 5 shows average tensile and bending results for all three hybrid configurations.

3.3 Inter-layer hybridisation

3.3.1 Tensile tests

The final configuration to be tested was the inter-layer hybrid. In these samples, impregnated carbon fibre layers were located on the surface of the sample, while the interior was self-reinforced PA12 (see Figure 4). Figure 8a compares a typical tensile stress-strain curve from this hybrid with the other two configurations described above, which failed in a catastrophic brittle manner as for the other two hybrid types. Although the carbon fibre fraction is similar, the inter-layer hybrid showed a lower tensile modulus than the other two hybrids, where the carbon fibres are more dispersed throughout the sample. It proved quite difficult to successfully impregnate the tightly packed carbon fibre layer with the high viscosity PA12 polymer. Combining this with some observed in-plane fibre misalignment could cause some fraction of the carbon fibres not contributing to the overall modulus.

The modulus predicted from modelling (11.3 ± 1.0 GPa) was significantly higher than the measured in-plane modulus (8.3 ± 0.4GPa) for this hybrid configuration.

3.3.2 Flexural tests

Figure 8b compares the flexural properties of the inter-layer hybrid with the other two configurations. As the carbon fibres are located on the surface of the samples, it would be expected that the bending modulus would be the highest of the three configurations, and this proved to be the case, although there was quite a large variability, again suggesting difficulty of obtaining even impregnation of the carbon fibre layer. However, in terms of bending, the inter-layer hybrid had the highest strength and modulus, as would be expected from the location of the carbon fibres. Failure occurred first on the tension side of the sample in the outer carbon layer (at around 2% strain) but the PA12 layer continued to be load bearing to higher flexural strains.

3.4 Penetration impact measurements

The final mechanical tests carried out on the hybrid samples was a penetration impact test. Figure 9 compares the measured impact energies for the various measurements.

In general the impact energies are of a similar magnitude, although there are some interesting differences. The intra-yarn hybrid, where the carbon fibres are very evenly distributed throughout the hybrid, gave the lowest impact energy. On the other hand the intra-layer hybrid showed the highest impact energy (30% higher than the pure SRPA12 sheet), so for impact there is potentially an advantage of having a combination of high carbon fibre fraction regions, and pure SRPA12 regions where the ductility of these regions is less constrained. For the inter-layer hybrid, the results were essentially the same as the pure SRPA12 sheet.

4 Discussion

This study has reported the tensile, flexural and penetration impact properties of the three hybrid configurations, intra-yarn, intra-layer and inter-layer, all at a similar carbon fibre fraction of 8 ± 1%. The major difference in these three configurations is the location and dispersion of the carbon fibres (as can be seen from Figures 2-4). These are an even dispersion in the intra-yarn hybrid; high volume fraction regions, but evenly dispersed, in the intra-layer hybrid and finally thin surface layers of a high carbon fibre fraction for the inter-layer hybrid.

As well as the dispersion of the carbon fibres, the three different configurations have specific production issues. While the intra-yarn hybrid configuration is an attractive proposition, it is probable that it is not ideal for the particular processing method used in this study, namely that of hot compaction which requires the selective melting of only a fraction of the oriented PA12 fibres. On processing, this molten fraction has to penetrate the carbon fibre bundles and produce a homogeneous composite, which looks to have been achieved only at the carbon fibre fraction of 8%, where additional PA-films had been introduced to achieve the lower carbon fibre volume fraction (and hence the higher matrix fraction). Other issues involved with this configuration are the increased fibre crimp and the discontinuous fibres.

For the inter-layer hybrid, the major challenge is to impregnate the tightly packed carbon fibre cloth, with a high viscosity thermoplastic resin. These cloths are mainly used for thermosetting resins, where the matrix is of a very low viscosity until crosslinking takes place. Moreover, in this configuration, the carbon fibres are located in high fraction layers on the surface of the sample. This maximises the bending properties, but may not be optimum for the penetration impact resistance.

For the intra-yarn hybrid, the prepreg tapes are already impregnated, making the final laminate less sensitive to the hot compaction processing temperature. The prepreg tapes also introduce additional isotropic PA12 to aid the processing, similar to adding an interleaved film which has shown to have significant benefits in optimising the hot compaction processing technique [38, 39]). Additionally, the very straight prepreg tapes give lower crimp in the final hybrid laminate and hence a better translation of the carbon fibre properties. This configuration also gave the best penetration impact energy, 30% higher than the pure SRPA12 sheet. As compared to a pure self-reinforced PA12 sheet, the intra-layer hybrid shows over double the tensile modulus and strength although a significantly lower strain to failure, again over double the bending modulus and strength but without compromising ductility, plus an increase in the penetration impact resistance.

It is proposed, therefore, that the intra-layer hybrid is the most promising hybridisation route, both for ease of manufacture and balance of mechanical properties. Figure 10 shows a comparison of typical tensile and flexural stress-strain curves for the intra-layer hybrid and a pure SRPA12 sheet, and clearly shows visually the difference in the behaviour. That is, a significantly improved tensile modulus and strength, accompanied by a large drop in the failure strain, together with significantly improved bending properties with no reduction in ductility.

To a large extent, this work, and the choice of the carbon fibre volume fraction (8%) was motivated by the parallel study on hybrid self-reinforced *polypropylene* carbon fibre composites [28]. In that study, a carbon fibre fraction of 8% was found to be optimum as this level the stiffness and strength were significantly increased without significantly reducing the failure strain of the self-reinforced polypropylene fraction. This second aspect was attributed to the level of intermediate adhesion seen in this material, allowing debondings and delaminations to occur when the carbon fibres fail, leading to reduced strain localisation and increased energy absorption up to failure. Increasing the carbon fibre fraction above 8% resulted in a steady reduction of the final failure strain. However, for the materials in this current study, where the bonding between the polyamide fraction and the carbon fibres is much better during tensile loading brittle behaviour occurs even at a carbon fibre fraction of 8%.

Figure 11 shows a comparison of the tensile and bending properties of these two different intra-layer based hybrids (both with an 8% carbon fibre fraction) highlighting the different balance of properties that can be achieved with the two different base polymers (the PP hybrid results are taken from our previous work described in [2]). In tension, the PP based hybrid retains the high failure strain of the pure SRPP sheet (>20%) due to local debonding and delaminations at the point where the carbon fibre fraction fails, contrasting with the PA12 based hybrid which fails when the carbon fibres fail at less than 2% tensile strain. Conversely, in bending, the improved bonding of the PA12 hybrid leads to significantly better flexural modulus and strength. For this reason, a higher carbon fibre fraction may be even better for the PA12 hybrid, as there is no worry in causing brittle failure in tension as it is already present, as opposed to PP where increasing the carbon fibre fraction above 8% led to a significant reduction in the ultimate tensile failure strain. A higher fraction for the PA12 hybrid could further improve tensile and bending modulus and strength. The upper limit would be the point at which the bending failure strain, and potentially the penetration impact, was significantly affected. Such a study, will be the subject of future work.

4. Conclusions

This paper has compared the properties of self-reinforced polyamide 12/carbon fibre hybrid composites made by three different hybridisation routes, termed intra-yarn, intra-layer and inter-layer, using a carbon fibre fraction of 8% for all cases. Tensile, bending and penetration impact tests were carried out on all the samples to assess the different hybridisation routes.

On balance, the intra-layer hybrids were considered to have both the easiest processing route, together with the best balance of mechanical properties. As the carbon fibre prepreg tapes were already impregnated for this hybridisation strategy, then this was definitely an advantage for producing homogeneous hybrid composites. The tensile modulus and strength were significantly improved over a pure self-reinforced polyamide sheet, although the tensile failure strain was significantly reduced from 10% to <2%. In bending, however, the modulus and strength were again significantly improved without compromising the ductility. Finally, the penetration impact energy of the intra-layer hybrid was greater than all the materials tested, including the pure self-reinforced polyamide 12 sheet.

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Table 1: Details of the intra-yarn hybrid

Intra-yarn hybrid	
Carbon fibre tensile modulus (GPa)	240
Carbon fibre strength (GPa)	4
Carbon fibre failure strain (%)	2
PA12 fibre longitudinal modulus (GPa)	2.9
Isotropic PA12 modulus (GPa)	1
PA12 fibre transverse modulus (GPa)	1
PA12 fibre longitudinal strength (GPa)	0.30
PA fibre strain to failure (%)	11
Carbon fibre fraction	8 ± 1%
PA12 fibre fraction	70 ± 5%
Isotropic PA12 fraction	22 ± 5%

Table 2: Details of the intra-layer hybrid

Intra-layer hybrid	
Carbon fibre tensile modulus (GPa)	230
Carbon fibre strength (GPa)	4.9
Carbon fibre failure strain (%)	2.1
PA12 tape longitudinal modulus (GPa)	3.5
PA12 tape longitudinal strength (GPa)	0.28
PA12 tape strain to failure (%)	10
Isotropic PA12 modulus (GPa)	1
Carbon fibre tape spacing – weft (mm)	14
Carbon fibre tape width (mm)	5
Weave Architecture	Twill 4/4
PA12 tape width (mm)	8
PA12 tape spacing (warp) (mm)	17
Carbon fibre fraction overall	8 ± 1%
PA12 tape fraction	70 ± 5%
Isotropic PA12 fraction	22 ± 5%

Table 3: Details of the inter-layer hybrid

Inter-layer hybrid	
Carbon fibre tape spacing (mm)	20
Carbon fibre layer thickness (mm)	0.1
Inner layers - PA12 tape width (mm)	8
PA12 tape spacing (inner layers) (mm)	~17

Table 4: A summary of the tensile and bending results for the intra-yarn hybrid samples at carbon fibre fractions of 13 and 8%.

	Intra-yarn [30]	Intra-yarn
Carbon fibre fraction	13 ± 1%	8 ± 1%
Tensile modulus (GPa)	9.6 ± 0.8	10.0 ± 0.3
Tensile strength (MPa)	111 ± 7	136 ± 4
Tensile failure strain (%)	1.9 ± 0.2	1.4 ± 0.1
Bending modulus (GPa)	9.5 ± 0.3	10.3 ± 0.5
Bending strength (MPa)	185 ± 4	202 ± 7

Bending strain to peak stress (%)	3.6 ± 0.1	2.5 ± 0.1

Table 5: A summary of the tensile and bending results for all three hybridisation routes at a carbon fibre fraction of 8%

	Intra-yarn	Intra-layer	Inter-layer
Carbon fibre fraction	8 ± 1%	8 ± 1%	8 ± 1%
Tensile modulus (GPa)	10.0 ± 0.3	9.2 ± 1.0	8.3 ± 0.4
Tensile strength (MPa)	136 ± 4	175 ± 8	126 ± 3
Tensile failure strain (%)	1.4 ± 0.1	2.0 ± 0.1	1.6 ± 0.1
Bending modulus (GPa)	10.3 ± 0.5	5.7± 0.4	10.6 ± 1.5
Bending strength (MPa)	202 ± 7	186 ± 4	186 ± 10
Bending strain to peak stress (%)	2.5 ± 0.1	9 ± 1	6 ± 1

Table 6: A comparison of the tensile modulus as predicted from modelling (incorporating the out-of-plane orientation angle) with the measured tensile modulus for the three hybrid configurations.

Hybrid type	Average out-of-plane angle (°)	Predicted tensile modulus (GPa)	Measured tensile modulus (GPa)
Intra-yarn	7	10.2 ± 0.9	10.0 ± 0.3
Intra-layer	3.5	11.0 ± 1.0	9.2 ± 1.0
Inter-layer	1.4	11.3 ± 1.0	8.3 ± 0.4

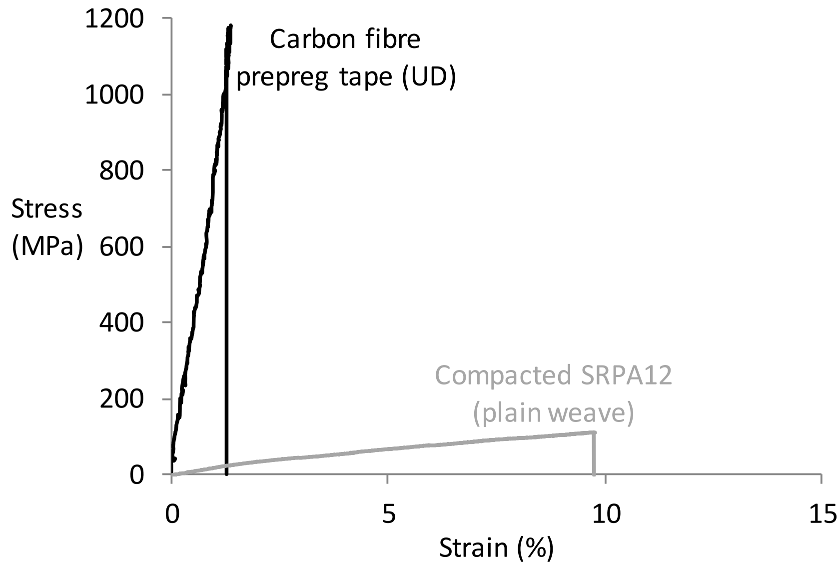

Figure 1: A comparison of the tensile stress-strain curves of a typical carbon fibre reinforced polyamide prepreg tape and a self-reinforced polyamide sheet.

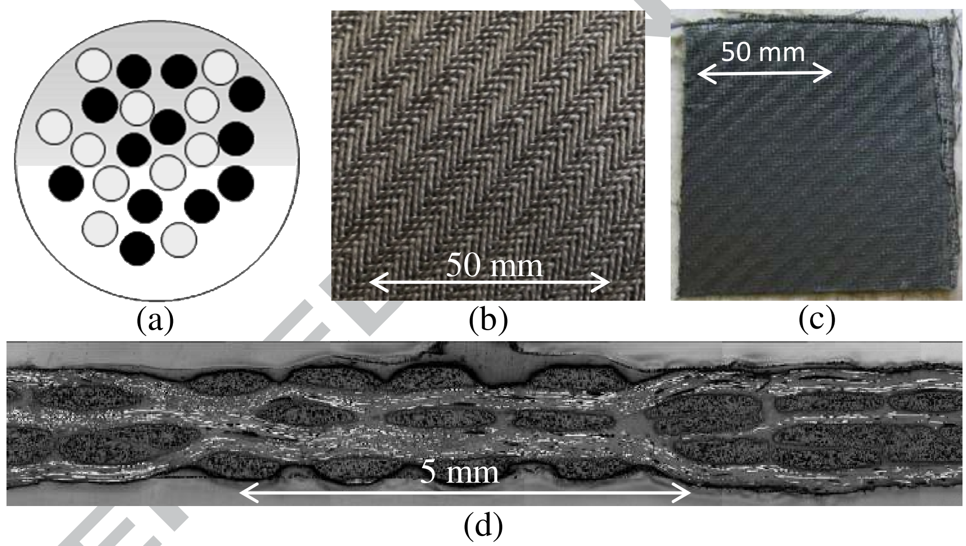

Figure 2: Details of the intra-yarn hybridisation route: a) a schematic diagram of the carbon fibre arrangement in each co-mingled tow, b) a picture of the woven cloth, c) a picture of the manufactured sample, d) a polished section from the sample.

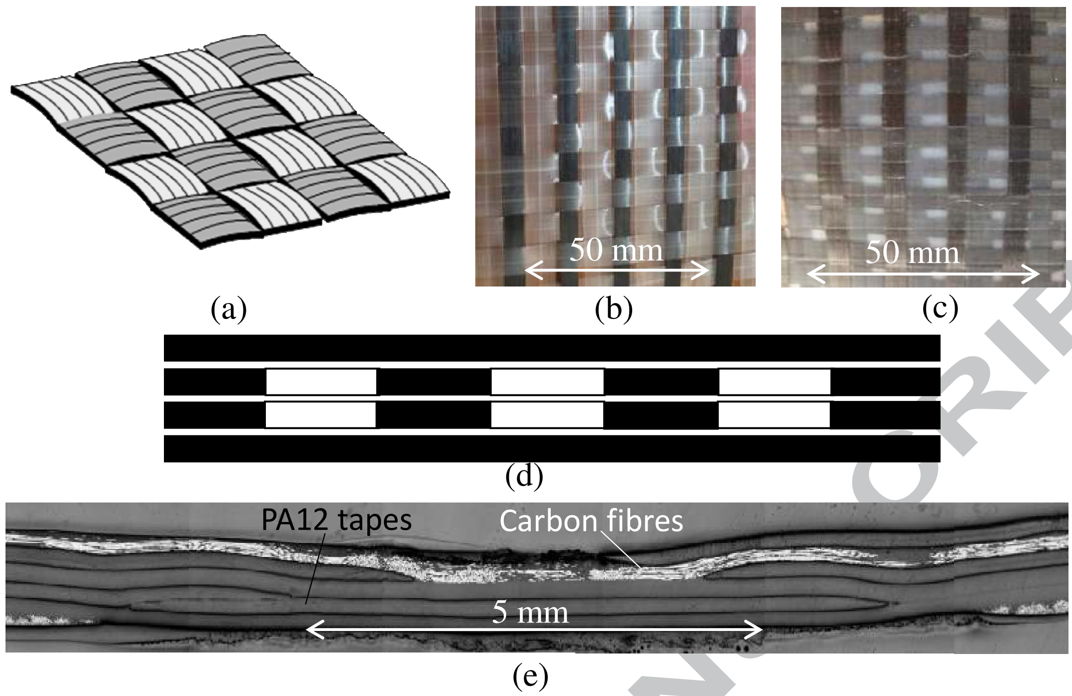

Figure 3: Intra-layer hybridisation: (a) schematic diagram of a co-woven layer [28], b) picture of the co-woven cloth before compaction and (c) after compaction, (d) stacking sequence of the hybrid laminate (0/90/90/0) and (e) optical microscopy cross-section of a manufactured sample $[0/90]_{2S}$.

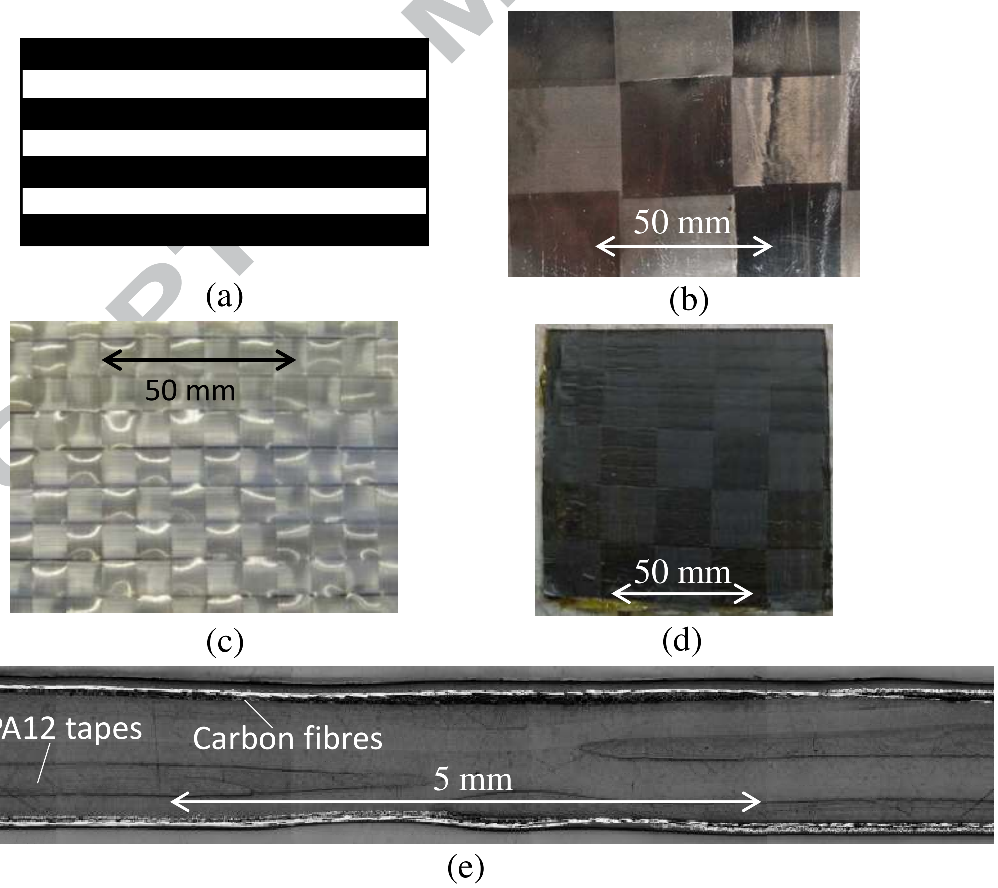

Figure 4: Inter-layer hybridisation: a) schematic diagram of an inter-layer sample, b) the TeXtreme impregnated cloth, c), a picture of the compacted hybrid (d) picture of woven PA12 tapes, and (e) a section of a manufactured sample

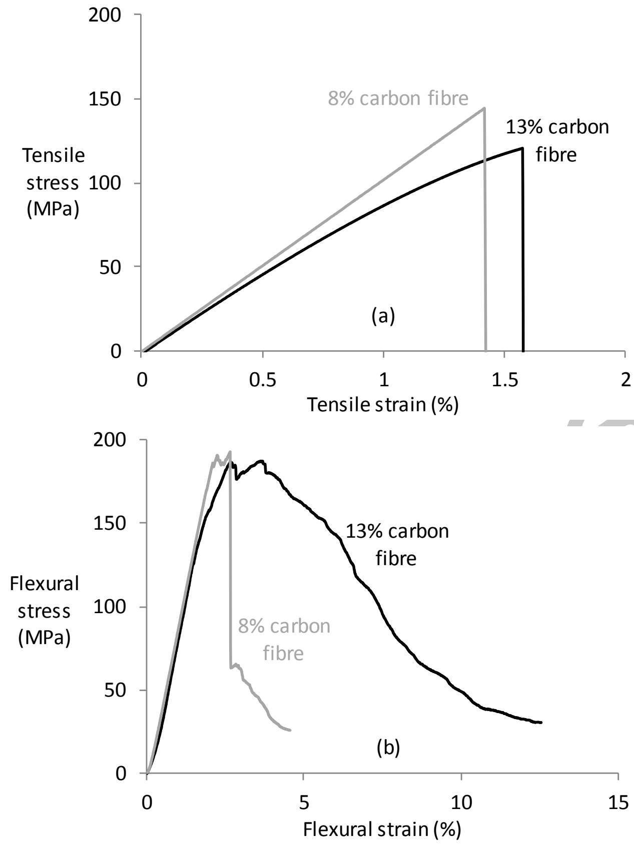

Figure 5: Typical tensile stress strain curves for intra-yarn hybrids with 8% and 13% carbon fibres: (a) tension, and (b) bending. 13% carbon fibre data from reference [30]

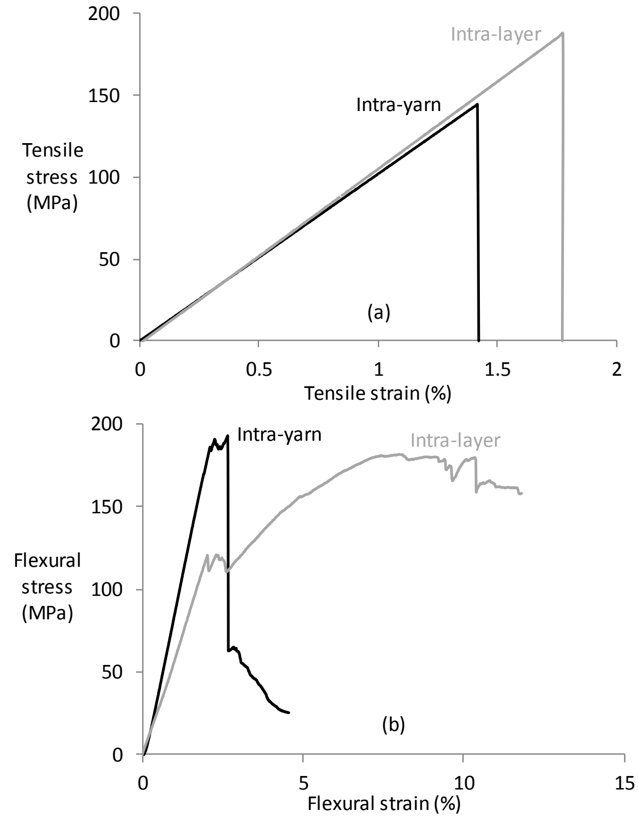

Figure 6: Typical tensile stress strain curves for an 8% intra-layer hybrid and an 8% intra-yarn hybrid: (a) tension, and (b) bending.

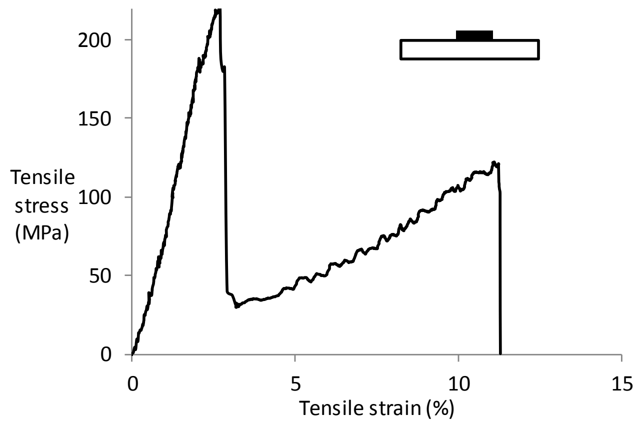

Figure 7: Typical tensile stress-strain curve for a combination of an SRPA12 sheet with a prepreg tape glued to the outside of the sample

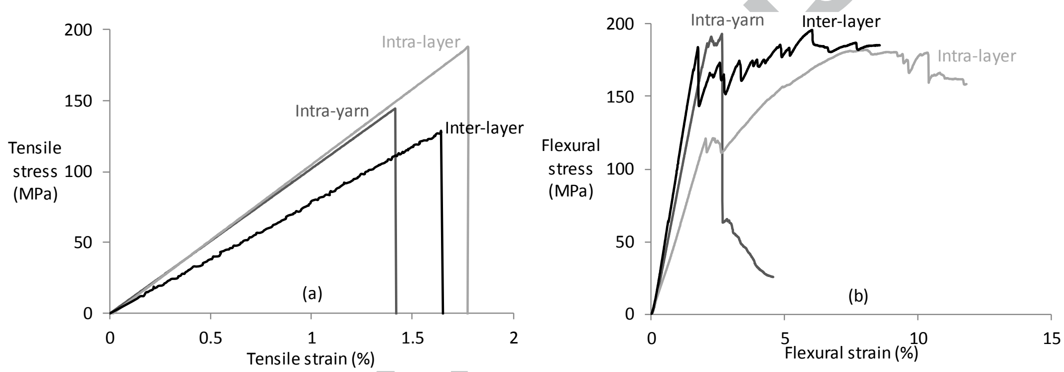

Figure 8: Typical tensile stress strain curves for an 8% inter-layer hybrid in comparison with an 8% intra-yarn and intra-layer hybrid: (a) tension, and (b) bending.

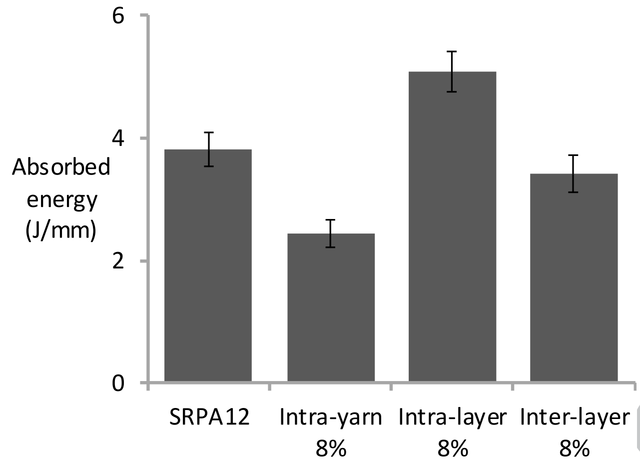

Figure 9: Penetration impact for the various hybrid samples. The error bars represent the standard error for five measurements.

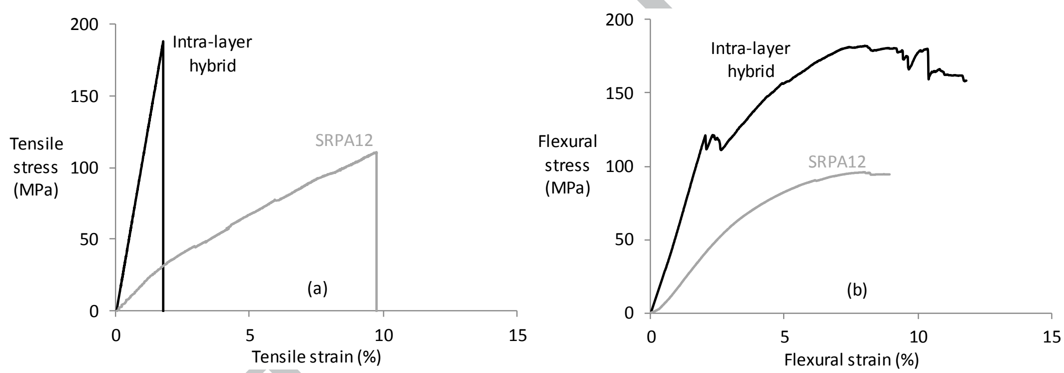

Figure 10: A comparison of the tensile (a) and flexural behaviour (b) of a pure self-reinforced polyamide 12 sheet (gray line) and the intra-layer hybrid (8% carbon fibres, black line).

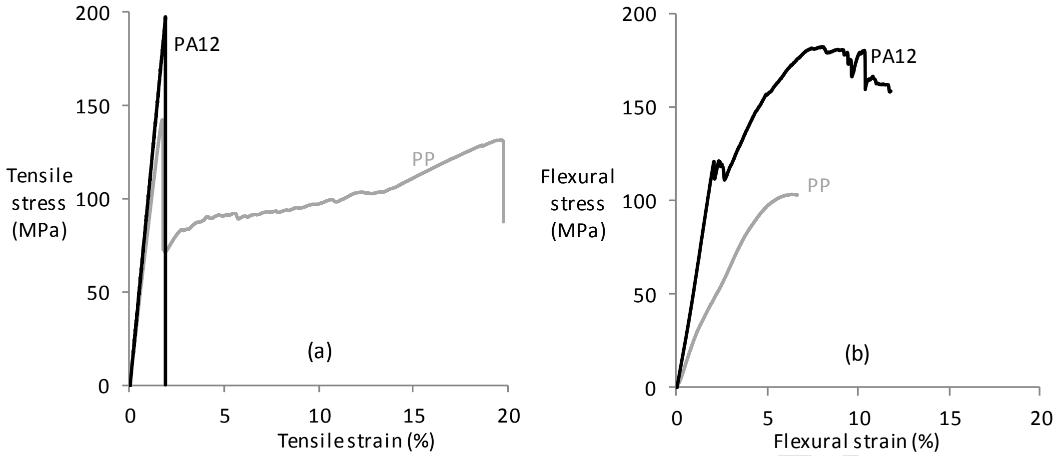

Figure 11: A comparison of the (a) tensile and (b) flexural behaviour of a PA12 intra-layer hybrid and a PP intra-layer hybrid (both 8% carbon fibre volume fraction). PP hybrid data here from [2]

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