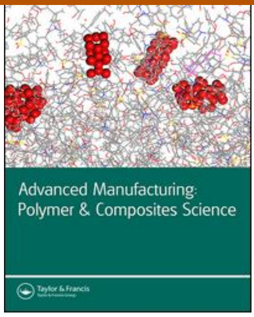

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The use of discontinuous PEEK/carbon fiber thermoplastic moulding compounds for thick-section componentry

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ABSTRACT

The hot-pressing of discontinuous fiber moulding compounds (DFMCs) is an established way of forming geometrically complex components, however, it is not a simple process. Rapid and irreversible cure cycles hinder the use of thermoset resins, and thermoplastic resins offer inferior mechanical performance. The recent availability of DFMCs utilising a Polyether Ether Ketone (PEEK) matrix offer an alternative, combining the usability of thermoplastics with significantly enhanced mechanical properties. A novel manufacturing approach is proposed and investigated, in which virgin material is consolidated into multiple 'pre-charges' prior to pressing the final component, combating the limitations of DFMCs; loft, voidage and fiber orientation. Short beam shear tests were employed to assess the mechanical implications of laminating DFMCs, demonstrating minimal differences to a standard sample. Three-point bend tests assessed rudimentary orientation of fiber bundles, showing significantly improved mechanical performance at the cost of toughness. A novel method to determine the interlaminar shear modulus is also presented and successfully validated.

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Thermoset prepreg; manufacturing; hot press forming; material characterisation

1. Introduction

The rapid and repeatable production of geometrically intricate, structural composite components has long been a challenging goal. Even basic processes such as the hand lay-up of woven prepregs are limited by the drapeability of the material, whilst being extremely time consuming if defects are to be avoided. Discontinuous fiber moulding compounds (DFMCs) have long offered an attractive alternative, with the ability to rapidly hot press complex parts in a repeatable manner—albeit often with a fraction of the mechanical performance offered by standard continuous fiber 2 D flat panel skins due to the randomly orientated fibers and the limitations on fiber length. The latter factor is largely due to the requirement for a DFMC to be able to demonstrate a degree of flowability. The maximum viable fiber length is therefore limited to around 50 mm, although shorter lengths of around 25 mm are more commonly used. Another common issue with the use of randomly orientated fibers is the potential for significant variability in mechanical performance—particularly in small samples. This behavior also makes the characterization of DFMC coupons challenging, as the small sample size coupled with variability can lead to problems with repeatability. Reducing the knockdown on mechanical performance and improving the consistency of the material are therefore two vital challenges which must be overcome if the material is to see more widespread use in industry. A simple answer to both problems is to somehow orientate the fibers. Orientated short fiber composites can have mechanical properties verging on those of long fiber composites [1–3] and the control of fiber orientation would naturally lead to a reduction in variability. The problem with this is the increased complexity of orientating thousands of individual fibers, a process which itself is limited and further complicated by the thermoset resins typically used in industry.

1.1. Hot press forming thermoset DFMCs

The desire for competitive mechanical properties has historically necessitated the use of a thermoset resin matrix in hot pressed DFMC components, however, this material has a number of significant drawbacks when used in this form. DFMCs typically suffer from excessive 'loft' in their virgin state, that is to say the volume occupied by the raw material is much larger than the volume of the final part. Hot-pressing components of any appreciable thickness, therefore, requires that a large throat or storage reservoir be incorporated into the tool in which the raw material might be held and heated prior to

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pressing. The press must then remove all of this bulk in a single cycle, as the thermosetting nature of the material prevents any cyclic melting and pressing. This also hinders any attempts at orientating fibers, particularly in thick section components. If fibers are to be aligned to some orientation, they must be done so in a single pass, and the orientation must be somehow maintained during a significant loft consolidation stage. Particularly, thick section components are also increasingly vulnerable to porosity defects due to the difficulty of maintain a constant heat gradient across the component leading to regions curing before being fully pressurized—the so-called skin-core effect [4].

A potential solution to all these problems is the use of a thermoplastic resin, which can be remoulded multiple times, allowing the part to be constructed in several steps. The limiting factor with thermoplastic resins has long been their mechanical performance, with composites made from nylons and polypropylenes having notably reduced properties when compared with epoxies. The low heat resistance of these materials also restricts them from being used in a number of emerging applications. Recent advances in high temperature, high performance thermoplastics are leading to a resurgence in their viability however.

1.2. PEEK

Of all the modern, high performance thermoplastics, PEEK is perhaps the most promising when looking to compete with thermoset resins. Already widely used in the medical industry, PEEK offers exceptionally high toughness and heat resistance and retains its tensile properties over several melt cycles, with a negligible effect on crystallinity [5, 6]. The modulus of PEEK at operating temperatures is typically ~3.5GPa, compared to ~4.7GPa for a typical aerospace epoxy such as the Hexcel 8552 [5, 7], however, it possesses a significantly improved fracture toughness of $4.8\mathrm{MN}\sqrt{\mathrm{m}}$ compared to $1.62\mathrm{MN}\sqrt{\mathrm{m}}$ for 8552. This improvement in toughness is of particular interest to industry, as impact damage and delamination remain the two most common issues with longevity of composite parts. Another key advantage offered by the improved toughness is the ability to machine CF/PEEK composites in a manner similar to metals. Attempting complex machining such as a thread (Figure 1) with epoxy composites typically results in excessive delamination, however, PEEK can be handled in a much more robust manner, improving its suitability to mass production [5].

From a hot-pressing perspective, the ability to cyclically remelt PEEK several times is of great

Figure 1. A demonstration of the machinability of MC1200/AS4 DFMC, showing a series of lathe-made cuts on a ring and two 5/16th threaded holes tapped into plates.

interest, as it allows for parts to be constructed in several stages. This ability removes the impact of 'loft', by allowing the virgin material to be compacted into charge blocks prior to the pressing of the actual part. The use of these charge blocks also presents an exciting opportunity regarding the random nature of the fiber orientation. Given the high viscosity of the CF/PEEK mix (even at high temperatures) the opportunity exists to potentially place 'packets' of highly aligned precharges within a mould, allowing a tailoring of localized structural performance in a manner similar to determining the ply-book for a conventional prepreg laminate. It is worth noting that although PEEK itself is not a recent thermoplastic development, or even its use as a matrix for unidirectional prepregs, its use as a matrix for DFMCs has only recently been explored, predominantly by Tencate Advanced Composites in conjunction with Victrex themselves [8]. Mass adoption of the material remains limited by its high cost to weight; however, it is the author's opinion that factors such as an increasing focus on recyclability and the manufacture of complex, thick section moulding scenarios will greatly improve its appeal.

1.3. Plan of work

The aims of the study were threefold: first, to investigate any impact of using pre-charges on the mechanical performance of laminates formed by hot-pressing MC1200/AS4 DFMC (the properties of which are detailed in Section 2.4) [8], second, to assess the effect of using highly aligned pre-charges and finally to introduce a new method by which interlaminar shear modulus might be determined for short fiber composites. Several pre-charge approaches were considered, in which charges of varying shapes are pressed in several manners, some aiming to minimize flow and some aiming to maximize it. This allowed the investigation of scenarios in which there is a high chance of a lamination effect occurring, as well as scenarios in which significant repositioning of the fibers will occur between the pre and post pressed state. The mechanical performance of the samples was assessed via short beam shear tests as detailed in ASTM-D-2344, as the potential for thick samples gives ample opportunity for both flow and lamina formation. Although 3-point bend testing would provide more accessible data, the difficulty of creating samples of an appropriate length relative to the necessary thickness, renders the test impractical, as discussed in the following section.

In Section 2, the effects of forming composites from precharges are discussed, focusing on the likelihood of the formation of distinct lamina in various scenarios. To highlight the complications with modeling randomly distributed fibers, the data collected was compared to simple bending equations using data provided by the material manufacturer. The complication of performing three-point bend tests on thick samples is discussed, and a metric laid down by which the mechanical performance of the experimental samples might be judged. Section 2 also introduces the new method for measuring interlaminar shear modulus, along with a discussion of the shortcomings of the current ASTMs when applied to short fiber composites. The methodologies employed to both create and test the samples is laid out in Section 3 with the results presented in Section 4, and the discussion of said results in Section 5. Finally, conclusions are drawn and plans for future work laid out in Section 6.

2. Precharged components

A number of significant problems exist with the manufacture of thick composite components from DFMCs including achieving a constant temperature throughout the mould and material loft. Initial tests have shown a consolidation of ~650% from the raw to the moulded state, with raw material filling a volume of 400 cm^3 for resulting in a compacted plate with a volume of 60 cm^3. If a part were to be formed in a single shot the mould would therefore necessarily have a raw material feed-in region (or throat) up to 7 times larger than final part. This is clearly impractical for thick-section parts, both in terms of cost of tooling and the scale of the pressing equipment that would be required for such a large tool.

The thermoplastic nature of PEEK allows an interim stage, where quantities of material are 'precharged', i.e. consolidated into smaller, three dimensional charges which are then combined and pressed into the final shape. The consideration of dimension is important, as a three-dimensional precharge which does not experience major flow during final forming will allow for significantly better control of fiber orientation in the final part. Due to the comparative simplicity of these precharge shapes, the tooling required for this step is comparatively inexpensive. This is due to a significant reduction in the required surface finish of the tools (i.e. the charges can be relatively 'rough') and the fact that they are notably smaller than that required for the final press. This also helps improve the consistency of the mould temperature during forming, as the consolidated charges transfer heat far more efficiently than the raw material. This approach therefore combats both of the aforementioned problems, however, precharging comes with the risk of building in defined lamina (or knit lines), as little intermingling of fibers is expected to occur between individual charges during the final press.

The potential implications of defined lamina within a composite component formed from precharges are unlikely to be beneficial, with the drawbacks being well documented for continuous fiber reinforcements such as unidirectional prepregs, which are highly susceptible to delamination failure. This form of failure will be mitigated to a degree by the toughness of PEEK, however, the lack or reduction of tows 'traversing' the lamination plane can be expected to lower the toughness significantly, whilst potentially lowering the stiffness of the structure due to the weak PEEK interface allowing the precharges to slip or shear relative to one another.

2.1. Material structure

The occurrence of lamination within a precharged component, and thus the mechanical impact of the feature, can be tailored by the manner in which the precharges are arranged. In order to assess the mechanical properties of the DFMC, short square beams $\sim 100 \times 20 \times 20$mm were cut from a $100 \times 100 \times 20$ mm plate. Due to the low viscosity of the material it was necessary to ensure that sufficient thickness was allowed to enable flow. Increasing the thickness of the coupon also allows for the formation of thicker and better-defined lamina in the appropriate forming situations. Finally, the cost of the material makes the formation of thick, 3-point bend standard coupons unattractive, as the standard length of the test specimen must be at least 20 times the width to eliminate the occurrence of significant shear deformation, which in this case would require a beam 400 mm in length. The Short Beam Shear test (ASTM D 2344) was considered preferable, with a sample aspect ratio allowing for a thickness sufficient for multiple thick precharge layers to be used during the manufacture of

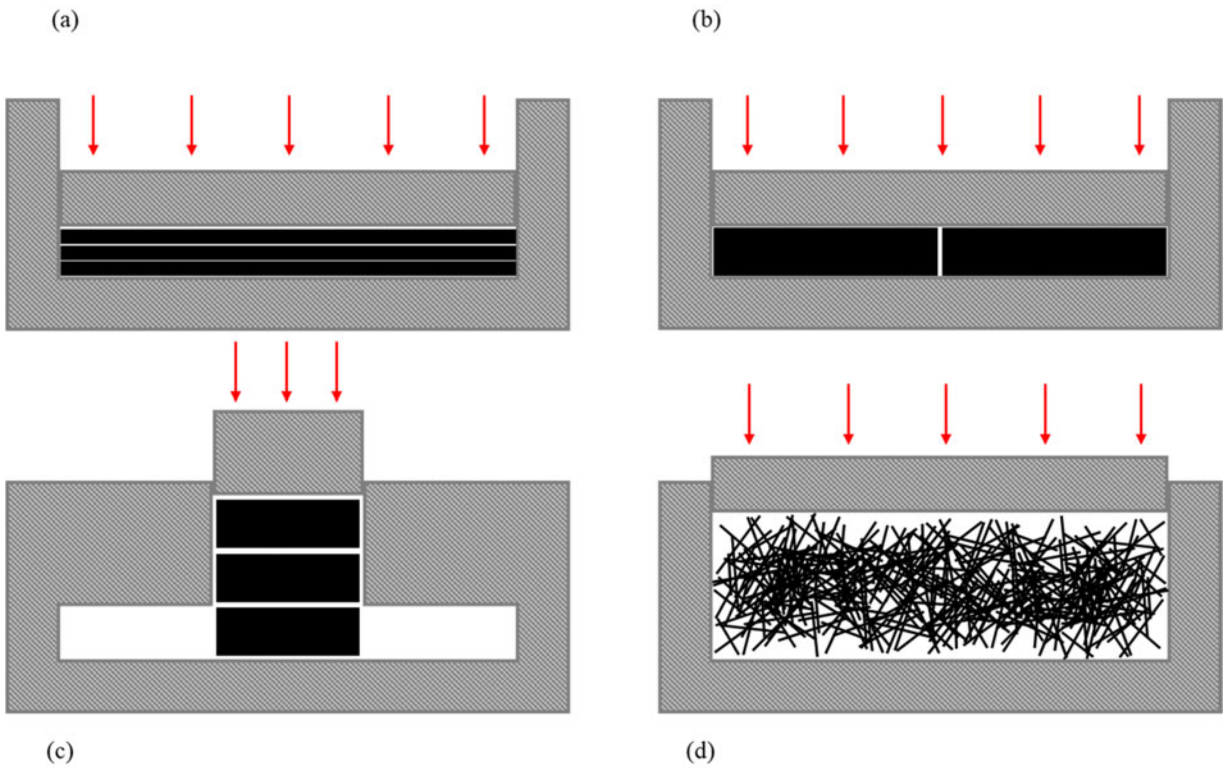

Figure 2. Methods for the manufacture of the short beam shear samples showing (a) lamination, (b) the formation of a knit line, (c) a method in which flow is forced, and (d) a simple single press. These images depict a cross-section of a cuboid mould with a reservoir footprint of 100x100mm.

relatively short samples. Three approaches to manufacture were considered and are detailed in Figure 2.

Approach 1: stacked precharge laminate

Three precharge plates $\sim 100 \times 100 \times 6.6$ mm were stacked on top of one another then formed into one thick plate with a second round of heat and pressure. A reduction in bending stiffness was expected in this plate, due to limited 'interleaving' of tows at the interfaces between the precharge plates. This lack of interleaving effectively resulted in the formation of continuous weak planes within the material. Work on the shearing behavior of unidirectional prepregs has already identified that shear stresses will localize around a continuous weak plane [9], and the bending of a laminated material will naturally result in a significant level of interlaminar shear, reducing the stiffness of the structure. Although the solid nature of the laminates investigated in this work will reduce the degree to which localization occurs, some discontinuous shear profile must exist as a result of the changing shear moduli from fiber to resin, and in particular the planes that exist between precharged plates across which very little intermingling of the tows will occur, reducing resistance to shear.

Approach 2: knit line

Two $100 \times 50 \times 20$ mm pre-charges were laid next to one another and pressed into a single plate. Samples are cut so that the interface or 'knit line' lies in the middle of the sample, propagating from one edge to the next. In this sample very few, if any, fibers will cross the interface. This sample should be the best indicator of the mechanical implications of a poorly located interface when using precharges. The fiber alignment in this sample is expected to be more uniform due to the narrow tool used to create the precharges, heightening the edge effect.

Approach 3: forced flow

Three $100 \times 50 \times 12$ mm precharges were stacked on top of one another in a chute, then pressed into a $100 \times 100 \times 20$mm reservoir to form a single thick plate. This approach eliminates the formation of any lamina by forcing the precharges to flow a significant distance from their starting position. Given the high levels of shear and flow it is expected that a high degree of alignment will exist at the edge of the part. Another potential side effect could be an increase in bending stiffness due to the disruption and limitation of the lamination effect expected from the stacked precharge approach resulting in significantly increased levels of interleaved tows.

Single press

The final specimen that ought to be studied is shown in Figure 2(d), in which the plate is pressed from virgin material in a single pass. This has proved impossible to achieve, however, due to the issues discussed previously. In order to create this 20 mm thick plate the mould cavity must be at least 160 mm deep to accommodate the material, and the ramming component >140 mm long. For heat to be transferred into the mould this entire mass of metal must be heated to 400 °C, which has proven impossible to achieve simply due to the requirement for the ramming part to move. For proper heating to be achieved a far more complex mould involving heating elements embedded into the ramming part and the mould itself would be required, which could not be financially justified for this project. Thus, attempts at pressing components in this manner have resulted in exceptionally poor consolidation and high levels of porosity, with the samples not warranting testing.

2.2. Hot press forming methodology

The material used to investigate this precharging approach was Cetex MC1200, a material commercially available from Tencate Advanced Composites consisting of preimpregnated 25 × 6 × 0.2 mm strips of AS4 fiber [8]. Full details for this material can be found in the referenced data sheet, with those relevant to the forming process and mechanical testing being referenced as required.

The hot press employed was a Labtech Scientific LP-S-50 capable of heating to 440 °C through the use of heating elements embedded in the platens and supplied with an inbuilt water-cooling system to provide an uncontrolled cooling of the platens. From observation of the cooling rate and cross referencing with evidence in the literature [10] the cooling rates achieved will have a negligible influence on the crystallinity of the samples, as a rate of 15 deg/s must be achieved if this is to be the case. During the heating phase the press closes to a preset distance, or until a defined pressure is reached. During the pressing stage the press seeks to apply and maintain a constant, pre-set pressure, with the position of the platens automatically changing as the sample consolidates to maintain the desired pressure throughout the cycle.

During the manufacture of the standard precharges, the approach dictated in [8] was largely adhered to. The charges were formed with the following steps:

1. The desired quantity of material was weighed and poured into the mould, with no attempt to control orientation.
2. No mould release was applied, to prevent the inter-laminar inclusion of this agent during further forming steps. Rather, the mould was polished to a 240 grit finish to aid release.
3. The temperature of the platens was set to 400 °C to ensure the material exceeded the recommended value of 385 °C. Discussion with Victrex suggested that heat degradation would not occur until the material exceeded 450 °C, which was impossible to achieve with the press employed.
4. The plates rapidly heated to 400 °C, and the mould was given 1 h to reach an external temperature of 385 °C measured with an independent thermometer, after which full pressure was applied.
5. Full pressure was held for 20 min with an applied pressure of 80 MPa, significantly in excess of the minimum stated in [8] to ensure full compaction.
6. After the 20 min pressure, the cooling cycle automatically initiated cooling the plates to room temperature after approximately 1 h.

Immersion density tests checks performed on the material relayed an average density of 1.56 g/cm^3, in agreement with the value provided by the manufacturer and, alongside the inspection of several flawless cross-sections give confidence in the absence of significant voidage.

This approach was repeated for the manufacture of the final samples, with the precharges being weighed before being placed in the mould as per the sample approach being investigated. All heating ramps and dwells were kept the same, as heat transfer to the material was expected if anything to improve as a result of the increased compaction of the charge material.

2.3. Orientation and methodology

In pre-charging a component, the manufacturer gains the possibility to better control the final position of short fibers as the distance they must travel or flow from the filling of the mould to the final part can be significantly reduced. The possibility of creating DFMC components with dictated fiber orientation as a way of optimizing mechanical performance therefore exists. In order to assess the impact of orientation some simply orientated samples were manufactured using a rudimentary alignment approach. This approach involved placing a grid made from 100 × 45 × 1mm steel inserts fixed to one another at the ends and held at 15 mm intervals (see Figure 3.) running parallel to the desired fiber orientation, pouring the material into the mould and tamping the fibers into place with a rod. Due to the 25 mm length of the fibers these inserts limited the maximum deviation from the desired orientation to ± 37° within the grid, and ±30° at the edges where the gap is reduced to 12.5 mm.

This is a simple geometrical constraint based on the length of the tows, the gaps between the spacers and the high stiffness of the tows due to the solid thermoplastic matrix. The inserts were carefully removed before pressing, leaving the fibers free to rearrange their position during forming, although this was not expected to happen to a noticeable degree due to the high packing density of tows prior to the hot pressing and the simple plate geometry of the coupons. The impact of the alignment was gauged through simple three-point bend testing. This process was combined with the Stacked Pre-charge Laminate approach from the previous section (Figure 2(a)), with the fibers aligned in the same orientation (i.e. 0°) to create an aligned short beam shear test from three thin, aligned plates.

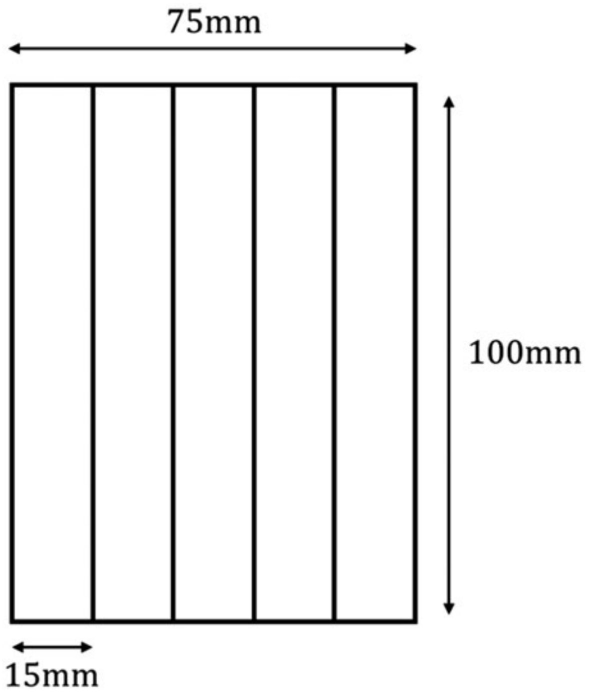

Figure 3. Top view and dimensions of the rudimentary grid used to achieve fiber alignment.

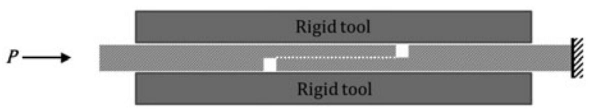

Figure 4. Schematic of in-plane shear force test set-up as prescribed by ASTM D 3846.

2.4. Determining the interlaminar shear modulus of DFMC coupons

There is a noticeable lack of experimental techniques designed to determine the various shear moduli of laminated materials. When considering carbon fiber composites, the primary method is detailed in ASTM D 3518, in which a cross-plied coupon of unidirectional prepreg is loaded at ±45° to the fiber orientated, resulting in a zone of pure shear in a manner similar to the bias extension test employed for woven fabrics. The test method itself is complex in its analysis due to the varying forms shear across the sample, however, when considering short fiber composites, it is entirely unsuitable, as it is heavily dependent upon the exaggerated Poisson effect which exists as a result of the ±45° lay-up and details the in-plane shear modulus, often termed G12, rather than the interlaminar modulus (G13 or G23) that is of interest. A new method was therefore required to assess the shear behavior of our discontinuous samples.

A test method to determine the interlaminar shear modulus of discontinuous fiber composite laminates—and indeed unidirectional composites at some angle other than ±45°, can be developed based on ASTM D3846 [11] which is used to determine the interlaminar shear strength of a fiber-reinforced polymer. The test requires the production of the 'notched' samples as shown in Figure 4.

The notches in these samples combined with application of a compressive load, serve to dictate a

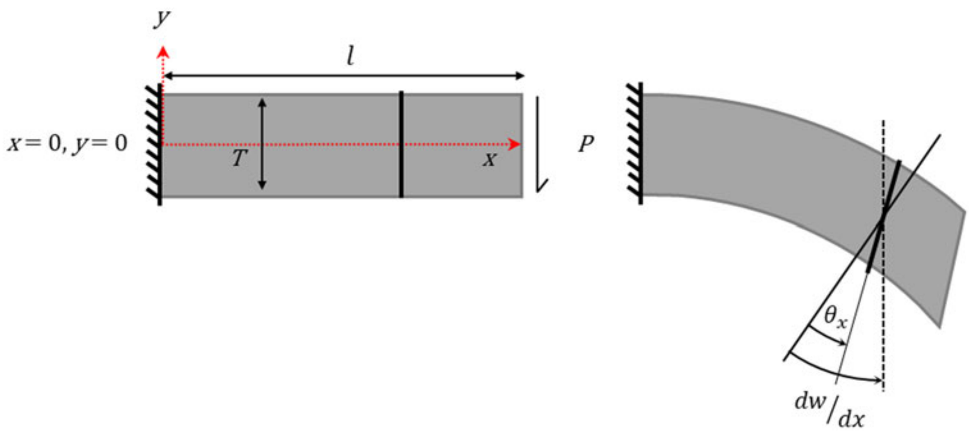

Figure 5. In a Timoshenko beam the rotation of the 'normal' is equal to θ_x, which is not equal to the curvature dw/dx (Figure from [12]).

plane where sample will fail in shear. By measuring the area of the failure plane and noting the maximum load, the shear strength can be determined. To determine the shear modulus G the shear strain γ must be determined. The difficulty of measuring this value is why the short beam shear test is unsuited to determining shear modulus. In a standard beam following engineers bending theory (EBT) the assumption of infinite stiffness in shear is made. Thus, when bending such a beam the normal is equal to the curvature dw/dx (see Figure 5). In a Timoshenko beam the 'normal' rotates toward the vertical, with the magnitude of the rotation θ_x being dependent upon the shear modulus G.

Theoretically, this rotation is displayed at the edge of a short beam shear sample when bent and can be used as a measure of shear strain γ. In practice, this attribute is extremely difficult to measure accurately in this way, therefore a different approach is proposed using an altered 'notched' sample in place of the test procedure described in ASTM D3846, shown in Figure 6.

By offsetting the notches from the mid-plane, the shear force is no longer applied to a rectangular plane but rather to a cuboid volume, thereby allowing the determination of the shear strain, γ, as shown in Figure 6—assuming small shear strain. Determining the shear strain is greatly simplified with the use of a visual extensometer, with dashes marking the top and bottom of the shear region as shown in by the solid red lines in Figure 7.

It can be seen in this image that the cut notches are rounded, rather than square as prescribed by ASTM D 3846. This is in response to an article written by Dr. Donald Adams [13] in which concern was raised regarding the introduction of stress

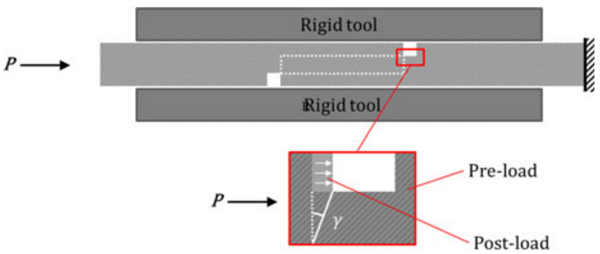

Figure 6. Modified test set-up from ASTM D 3846 in which the notches are offset from the center-line to provide the shear area shown in the dashed white box. This allows for the calculation of the shear strain γ.

concentration points with the notches. By rounding the notches this stress concentration is reduced. Furthermore, and most importantly, this test is not looking to yield the sample, merely measure the elastic portion of the response. These two factors are therefore considered to adequately address Adams' reservations. Whilst the rounded edges do complicate the 'pure shear' boundaries, the volume this affects is minimal ($\sim$1mm^3 from a shear volume of $\sim$810 mm^3) and the effect is therefore considered to be negligible.

The shear strain itself must first be decoupled from the compressive strain imposed by the test set-up. Pure shear is achieved as a result of the differential movement of the material in line with the notches, which disrupt the transmission of load (Figure 8 (Left)), however, the test set-up proposed also consists of a pure compressive load running through the middle of the sample (Figure 8 (Middle)).

With the use of a visual extensometer, a compressive test is performed on the sample prior to notching to determine the compressive modulus in the region to be sheared. The use of the visual extensometer is important, as the compressive modulus may vary across the sample due to the randomly distributed nature of the fibers. The sample is then notched, with new visual extensometer markings applied and tested again. The displacement Δ_s from this test is then compared to the predicted compressive displacement Δ_c using the value of compressive modulus and a portion of the applied load (discussed below). Due to the location of these markings, the difference in displacement gives the distance s (see Figure 9) which represents the deformation as a result of shear. This can then be used to determine the shear strain γ and, in conjunction with the portion of load which transmutes to traction forces, used to determine the shear modulus of the material.

The load acting to compress the sample must then be decoupled from the load acting to impart shear. At present this is achieved by simply considering the proportion of the load that acts through the cross-sectional area of the un-notched center of the sample as generating compressive strain, and the load that is disrupted by the notches completely transferring into an effective traction force on this central region, generating shear deformation (see

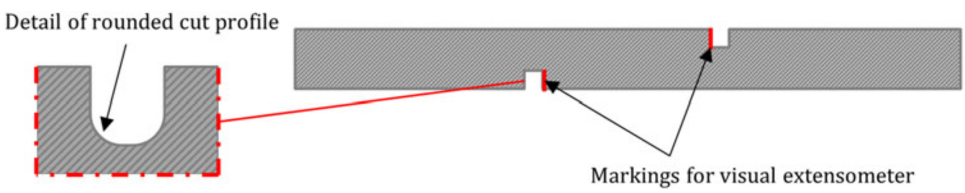

Figure 7. Image detain the location of the marking used for the visual extensometer set-up.

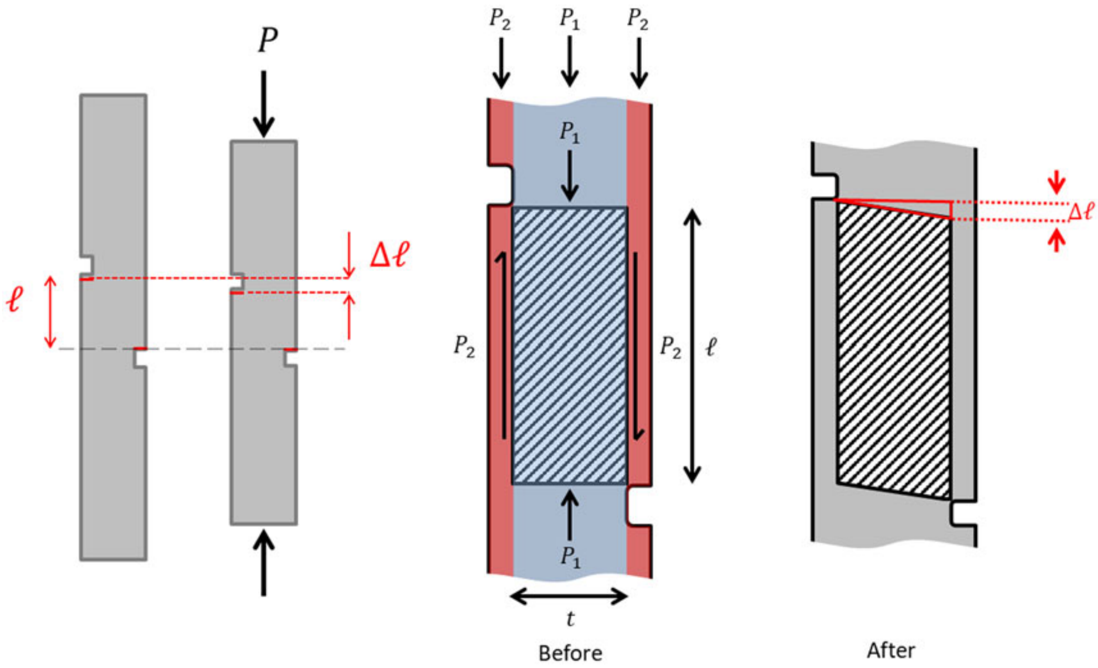

Figure 8. Showing (Left) the simplified approach to attributing load to shear and compressive components respectively, and (Right) the resulting shear deformation in the sample, *s*.

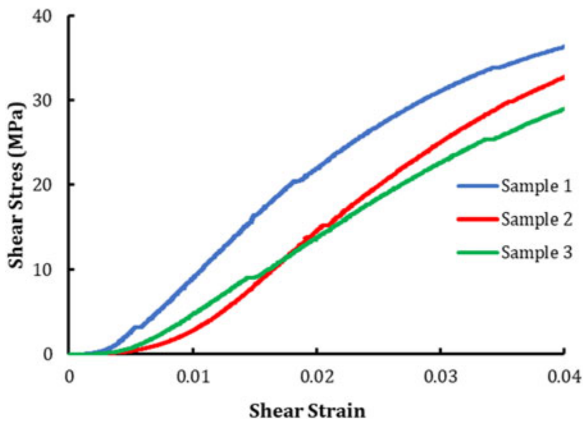

Figure 9. Plot of shear stress τ versus shear strain γ for three samples of Nylon 66.

Figure 9). It is acknowledged that this is a significant over-simplification of the actual loading scenario which will require addressing in future work. There are however a number of factors which inform the assumption that some level of pure shear will occur. First, rotation of the 'limbs' formed by the notches which apply the traction force to the shear area can be considered to be negligible due to the compression rig employed as per ASTM D 3846. Second, the expected shear modulus of the material will be significantly less than the compressive moduli in all aspects, therefore, a shear deformation will be preferential as a path of least resistance. It is acknowledged however that pure and uniform shear will not be achieved across the area of investigation, and the shear distribution will require further investigation in the future.

For the time being, a rough approximation can be obtained by utilizing the recorded load and the known dimensions of the shear region the shear modulus can then be determined and applied in Equation (2) to allow for the calculation of the flexural modulus. The steps to determine the shear modulus using this approach are therefore as follows:

1. Determine the compressive modulus in the region of the sample that is to be subjected to shear using a visual extensometer (see Figure 8 (Left)).
2. Notch the sample and apply visual extensometer markings for shear test (see Figure 8 (Middle)).
3. Determine displacement $\Delta\ell$ from experimental results.
4. Calculate approximate expected compressive displacement $\Delta\ell_c$ using compressive modulus and compressive load P_1.
5. Subtract $\Delta\ell_c$ from $\Delta\ell$ to find the displacement $\Delta\ell_s$ which can be attributed to shear (see Figure 9)
6. Using small shear approximation shear strain $\gamma = \tan^{-1}\left(\frac{\Delta\ell_s}{t}\right)$.
7. Using the portion of load which converts to the traction forces P_2, determine shear stress and then shear modulus.

To build a degree of confidence in this method, it has been applied to a series of tests conducted on Nylon 66, which has a shear modulus $\boldsymbol{G} \approx 1.1$GPa. The results of the testing are shown in Figure 9 and

Table 1. Table of shear modulus, *G*, values obtained for Nylon 66.

Sample number	Shear modulus (MPa)
1	1112.32
2	1089.44
3	918.16
S.D.	81.2

Table 1. The results provide a high degree of confidence in the values obtained. Verification shall be available by comparing the final values of shear modulus obtained from this test against the shear modulus provided from the data sheet values tested as per ASTM D3518-M. Although the standard deviation could be considered high for this material, it is considered to be a result of the infancy of the test method and is largely the result of the third sample tested.

3. Experimental methodology

A number of different test methodologies were employed to gather the desired data. Where possible, these tests stuck to the specifications of the relevant ASTM, and in cases where deviations were required, for example the novel shear modulus method, every effort was made to remain as close to the relevant ASTM as possible in terms of sample dimensions.

3.1. Short beam shear testing

The test methodology employed for the short beam shear testing is as described by ASTM D 2344 M. For each manufacturing approach investigated four $100 \times 20 \times 20$ mm samples are tested, cut from a single $100 \times 100 \times 20$ mm plate itself produced in the relevant manner described in the previous section. The press is set to impart a pressure of 80 MPa upon the mould for 20 min once the mould has reached 400 °C. The span size of the bending rig employed is set to 80 mm to be within the dimensional range prescribed by [14] whilst allowing a 10 mm overhang for the sample on each end. The deformation rate is set to 1 mm/min and a 300 kN load cell was used in order to cope with the expected high loads.

3.2. Three-point bend testing

The test methodology for the three-point bend testing was followed as prescribed by ASTM D 790-03. For each configuration investigated a minimum of five $100 \times 14 \times 4$mm samples were cut from a $100 \times 100 \times 4$mm panel pressed in a single pass using 60 g of charge material. In order to ascertain whether or not surface finish has an impact on flexural performance, two sets of standard panels were tested with 60 and 1200 grit finishes, respectively. To ensure that processing conditions did not impact the results 360 grit and three 1200 grit samples were cut and tested from a single plate, with two such sets of samples being investigated for a total of six samples at each finish. A further five 'orientated" samples were tested from a plate which inserts were employed in order to control the fiber orientation during charging, which were subsequently removed prior to hot pressing. Samples were moulded using a single 20-min press at 400 °C and a pressure of 80 MPa on the mould. The span length on the three-point bend rig was set to 64 mm with the contact points having a 3.2 mm radius, as prescribed in [15].

3.3. Shear modulus testing

The technique by which the shear modulus of the material might be determined first consists of an elastic compressive test (i.e. not triggering failure) to determine the compressive modulus on a pristine sample. The use of the visual strain gauge allows the precise measurement of the modulus for the region that is then subjected to shear in the following test. This shearing is achieved by notching the sample in such a way that the applied load results in a combined shear and compressive strain (see Section 2.4). The marking system for the visual extensometer detailed in Section 2.4 effectively captures the deformation in a straight path as a result of these combined strains. Application of the applied load and previously calculated compressive modulus then allows the isolation of the shear deformation, which thus allows the calculation of the shear strain arising as a result of the applied load, and thus the determination of the shear modulus.

The test methodology for the compressive modulus and shear modulus values was identical, the difference being in sample itself, as mentioned in the previous section. The test methodology is detailed in both ASTM D 695 and ASTM D 3846 and shall be summarised here. In both instances the sample was mounted between constraining plates designed to prevent lateral deflection in the sample, especially in the shear case in which a moment is invoked as a second order effect, leading to the sample attempting to rotate. A fixed compressive deformation rate of 1 mm/min is then applied to the sample and the resulting load measured. A preload of 20 N was applied to eliminate slack from the deformation readings. Sample dimensions for both tests were $80 \times 13 \times 7$mm, with a single 1.6/1.6 mm notch cut on each side for the shear tests, the center of which was located 4 mm from the middle of the sample.

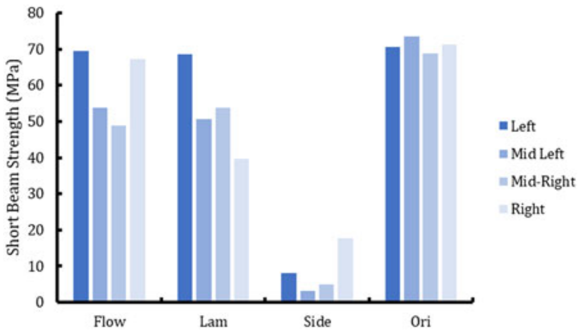

Figure 10. Plot showing the short beam strength for samples taken from three thick plates manufactured using different techniques.

This resulted in a $6.4 \times 13 \times 3.8$ mm zone in which shear would occur. Due to the high variability expected of the mechanical properties of the DFMC samples each sample was used for compressive testing first before being ‘notched’ for the shear testing. This required that the level of compressive loading applied during the first test was small to avoid reaching the elastic limit of the material. Strain measurements were taken using a visual extensometer for both samples to avoid the effects of any slack in the test set-up. Two approaches to forming the notches were considered, filing to the desired profile and cutting using a diamond edged tile cutter. Comparisons of initial tests on samples manufactured via both approaches showed negligible differences in results, and the tile cutter approach was chosen to speed-up sample preparation.

4. Results

The results shown in Figure 10 detail the short beam shear strength data gathered from samples cut from plates manufactured using the techniques described in the previous section. The plates made from the flow (Flow) and lamination (Lam) approaches show similar results, with the side samples typically showing improved properties. The plate with the knit line (Side) shows markedly reduced mechanical properties—as expected. Finally, the plate with orientation fibers (Ori) showed significantly increased consistency in results, but now significant increase in results when compared to the edge samples from the Flow and Lam results. Stress/strain traces for the shear tests are shown in Figure 11, generally showing good agreement especially when considering the random nature of the fibres.

The data displayed in Tables 2 and 3 the flexural moduli and the maximum bending stresses obtained for the polished, rough, and orientated 3 pt bend samples. Very little difference is observed in the average values for the polished and rough samples,

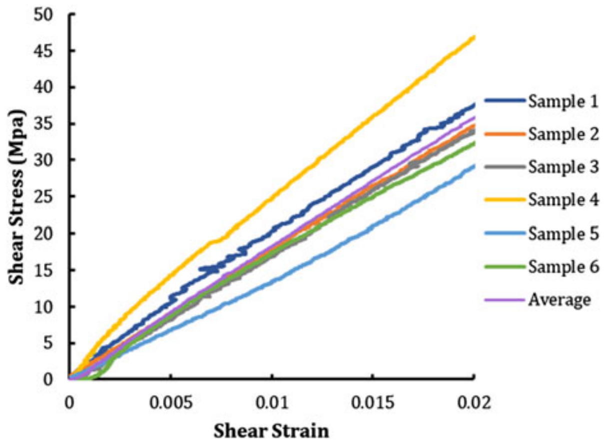

Figure 11. Shear Stress vs. Shear Strain for MC1200 DFMC samples.

however, the orientated samples show significantly improved properties, as expected.

The data displayed in Figure 12 and Table 4 show the results from the new test method as described in Section 2.3. Repeatability appears good with the exception of Sample 5, resulting in the lower shear modulus value in Table 4. The data range analyzed to provide the shear moduli was between shear strains of 0.0025 and 0.005. Sample 6 shows some evidence of slack at the start of the trace. On closer inspection of the sample this appears to be a result of the crushing of a region of burring that was not properly removed after the sample was cut. To counter this modulus readings were taken from regions unaffected by the slack.

5. Discussion

5.1. Short beam shear

One of the immediate observations from the flow and laminated short beam shear tests is the clear existence of two failure mechanisms; flexural and shear (see Figure 13). Shear failure was typically observed to occur in samples cut from the center of the plate, whilst flexural failure occurred in samples cut from the edge of a plate. This is considered to be a result of the edge effect of the mould, whereby the fibers buckle when pushed against the side of the mould, dramatically increasing the stiffness of the material in the otherwise normally weak z-axis. This effectively results in a significant increase in shear stiffness, impeding delamination and forcing flexural failure. Interestingly, the shear strength of both the flow and laminated samples is similar, suggesting that the potentially detrimental side effects of stacking precharges are minimal, if at all present. It also shows how significant the edge effect can be, as the reorientation of fibers was still clearly evident when the charge material was placed near to its final position.

In both samples, the central samples clearly failed in shear with obvious delamination. The flow samples appeared slightly tougher, judging by the more eventful post failure load traces and capacity for carrying significantly more load post yield. That said, the near identical mechanical properties of the laminated and flow samples suggest that the potentially negative implications of laminating a sample by stacking pre-charges is far less significant than previously expected, with the only obvious difference being the post failure behavior.

The knitted samples showed a massive reduction in mechanical properties however, with a maximum shear strength being between 10% and 25% of that of the shear strength recorded for the other samples. This is by no means unexpected, as the lack of any bridging fibers at the point of maximum deflection means that the test was effectively loading the PEEK matrix without any reinforcement to act as crack 'frustraters'. Interestingly, the edge samples again showed a significant increase in mechanical performance, suggesting that the act of forcing the fibers up against the mould caused some to displace over the knit line.

The oriented samples showed a significant reduction in variability when compared to the other samples and, importantly, showed no difference between samples cut from differing regions of the plate. This confirms not only the edge effect imposed by the tool helps orientate the fibers, but also that any orientation imparted to the precharged fibers can be reliably carried over into the final part.

The key observations from these tests are therefore as follows:

- Edge samples have significantly improved mechanical properties due to the fact that edges help align fibers against the tool during pressing.
- Laminating precharges does not negatively impact the strength of the coupon.
- Knit lines along highly stressed regions clearly have a severely detrimental effect on mechanical performance.
- The sample constructed from orientated precharges showed a significant reduction in variability, alongside improvements in both stiffness and shear strength.

Table 2. Table of flexural moduli obtained through 3 pt bending tests for polished, rough, and orientated samples.

Flexural modulus (MPa)		
Polished	Rough	Orientated
57565.03	56839.40	53082.60
38936.65	33102.60	66371.25
33742.22	32220.45	67942.73
24924.55	51155.41	63483.78
46894.12	29449.05	46480.10
26578.92	28161.59	
Average		
38106.91	38488.08	59472.09
S.D.		
9691.69	10339.55	7752.59

Table 3. Table of maximum bending stresses obtained through 3 pt bending tests for polished, rough, and orientated samples.

Maximum bending stress (MPa)		
Polished	Rough	Orientated
915.83	895.52	867.36
634.37	696.23	982.53
660.17	647.33	960.6
759.24	403.39	936.86
563.52	793.87	747.54
539.34	441.51	
Average		
678.75	646.31	898.98
S.D.		
105.86	149.24	73.22

5.2. Three-point bend

The first key finding from the three-point bend testing was that surface finish has no impact on mechanical performance, as displayed in the similarities between the red and blue lines in Figure 10. This was not unexpected, as the thermoplastic PEEK matrix is significantly tougher than the thermoset resins used in the vast majority of other carbon fiber composites, where surface flaws can act as

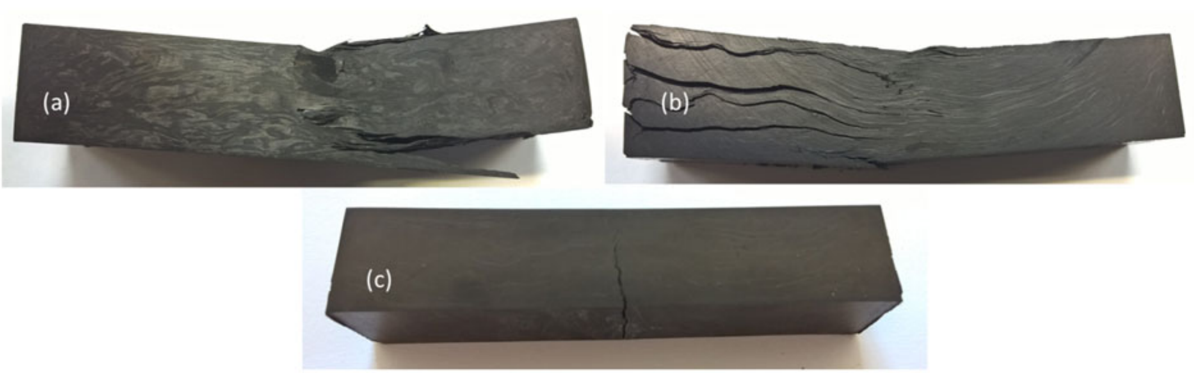

Figure 12. The various failure mechanisms observed in the Short Beam Shear tests, showing (a) tensile failure in an edge sample, (b) shear failure in a central sample, and (c) matrix failure in a knit line sample.

stress concentrators and crack initiators. This finding also informed the finish used on all other samples, saving considerable time which would otherwise have been spent polishing samples. A critical observation of these two sets of data is the extreme level of variability in the mechanical properties, ranging in some cases from 14 to 40 GPa across a single plate. As discussed previously this is a known limitation of DFMC composites, however, the results from the 'orientated' plate showed significantly reduced levels of variability, and, more importantly, significant increases in overall mechanical performance. In fact, only a single sample displayed a modulus value lower than the highest modulus attained for the randomly orientated samples.

Knowing that simple attempts at orientation of the fibers can result in such drastic improvements in mechanical properties, and such a significant improvement in the level of variability, future work must focus on understanding how best to arrange orientated pre-charges with a view to minimizing flow so as to preserve intended orientation. The key observations from these tests are as follows:

- Surface finish has a negligible impact on mechanical performance.
- A huge degree of variability exists in the 'quasi-isotropic' samples.
- The apparent mechanical improvement of the mould edge effect observed in the short beam tests is also present here.
- Orientating the fibers results in a significant increase in flexural modulus and a major reduction in variability.

5.3. Shear modulus

The value of average interlaminar shear modulus of 1.814GPa displayed in Table 4 is significantly less than the value displayed on the data sheet for TC1200 of 5.2GPa [16] (the precursor for MC1200, so the same resin and fiber, but in a different form), however somewhat greater than the average shear modulus for PEEK of 1.3GPa. There are a number of potential reasons for the discrepancy with the value quoted in [16], first and foremost being the form of shear invoked. The value of 5.2GPa is gathered from ASTM D 3846 and is therefore an in-plane shear modulus. The rotational deformation of the ±45° fibers therefore not only require the laminate to shear at the interface between the plies, but also between the fibers within the plies themselves. This intra-ply shear stiffness is expected to be somewhat higher than the inter-ply shear stiffness, as a result of the absence of a clear plane of deformation

Table 4. Shear Modulus values from the test method proposed in Section 2.3.

Sample number	Shear modulus (MPa)
1	1932.87
2	1741.97
3	1741.86
4	2150.52
5	1543.68
6	1774.28
Average	1814.2
S.D.	151.67

due to the complex structure of fibers and resin within a ply. The test methodology developed in this technique should invoke a more focused interlaminar shearing, therefore promoting deformation in the weak resin interface between fibrous layers. The fact that the observed shear modulus is greater than the shear modulus of neat PEEK suggests that the test is somewhat successful in this regard, however the complexity of the microstructure in the coupon combined with the relatively simple approach to decoupling the loads that contribute to shear and compression, respectively, does not provide adequate confidence in the results, promising though they may be.

There is a significant degree of variability in the results obtained, but again this is considered unsurprising due to the small sample size, the randomly orientated samples and the relative immaturity of the methodology. The small sample area within the coupon is a potential weakness of the test method, however this is mitigated to a degree by the test measuring interlaminar behavior, and the tendency for fibers to align fairly horizontally under pressure, reducing the likelihood of a scenario in which a single tow connects the two acute corners of the sheared shape, where in such a scenario, the measured shear stiffness would drastically increase. In retrospect, a recommended amendment to the methodology would be to ensure that the length of the shear region exceeds the maximum tow length. The key observations from these tests are as follows:

- Results are less than the shear modulus for in-plane shear on a similar sample, however this might be due to the different shear mechanism invoked.
- The shear modulus obtained is approximately 0.5MPa greater than that expected in neat PEEK resin.
- Large variability in value of shear modulus (±20%) in DFMCs.

6. Conclusion

A number of experimental methodologies have been employed to investigate the mechanical impact of creating DFMC components with preconsolidated charges. The first set of tests looked at three separate forming approaches in which precharges were arranged in different ways, finding little difference between a 'laminated' approach or an approach in which the fibers were required to flow a significant amount during moulding, thereby more closely resembling a single press component. The final approach in which a 'knit line' was created along the point of applied load showed a significant reduction in load capacity as expected. This confirms that precharging can be used without detrimental effects if the expected loading regime of the component is properly accounted for. This does however require a change in thinking from typical discontinuous composite manufacture. Rather than creating a quasi-isotropic 'black aluminum' substitute this manufacturing approach promotes the manufacture of highly designed components, taking advantage of the possibility of increasing fiber orientation in tailored precharge packets arranged to minimize the detrimental effects of lamination. Whilst this manufacturing approach is obviously more complex than simply pouring virgin material into a mould, the potential performance improvements and reductions in porosity in thick section components are extremely exciting.

The novel approach to determining shear modulus developed has provided interesting results, with a lower shear modulus to that determined from in-plane tests on a unidirectional material with the same fiber and resin, but higher values than the shear modulus reported for neat PEEK resin. To build trust in these results a proper FEA analysis of the load distribution in the sample is required to determine exactly how much of the applied load generates shear and compression respectively. It is worth highlighting at this stage that this remains a highly experimental procedure that is still in the early stages of development. A number of alterations to the methodology and sample preparation are planned to improve the accuracy and viability of the method. A key problem with the current methodology is that it is reliant upon the simple disentanglement of the shear and compressive deformation, which is unlikely to be wholly representative of the sample given that the applied compressive load will doubtlessly alter the shear behavior of the material. Future work shall look to investigate the suitability of this technique on different material forms, such as unidirectional laminates of varying stacking sequences. Another area requiring future work is the risk of undesirable stress concentrations in the notched samples. If this process is to be developed with a view to creating a new ASTM standard, these concentrations will require some numerical analysis.

Future work shall look to further investigate the potential benefits of precharging, following two avenues of interest; improved mechanical performance and recyclability. Research into mechanical performance will look at the concept of orientated precharges and how reliably orientation can be maintained from the charges to the final coupon. This work will likely include a detailed study of the flow behavior of the material and will doubtless look to begin a detailed characterization regime in order to enable flow modeling during the manufacturing process. The research area of most interest is recyclability however, as this system offers the prospect of 100% material recovery, as there is no need to burn off the resin to recover the fibers. Rather, the material can simply be reheated and repressed in exactly the same manner as the precharges. Critical research areas will be cyclic fatigue of manufacturing properties and polymer degradation effects, as well as the prospect of maintaining any fiber orientation from the original part.

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Disclosure statement

No potential conflict of interest was reported by the authors.

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