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CHARACTERISATION OF CARBON FIBRE REINFORCED POWDER EPOXY COMPOSITES FOR WIND ENERGY BLADES

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Abstract

Powder-epoxy resins have been shown to possess desirable properties making them suitable as a matrix for the production of composite wind blades. CFRP laminates manufactured using a powder-epoxy matrix were tested to determine mechanical properties. The laminates were manufactured using a non-crimp stitched unidirectional fabric and were compared to laminates manufactured from fibre tows with tension applied during the curing cycle. The tensioned case was investigated as a benchmark for superior fibre straightness mimicking the pultrusion process whereas the fabric was representative of materials used in industry. The materials were mechanically tested in the longitudinal and transverse directions in both tension and compression to determine strength and modulus. Results showed that the stitched case had a normalised tensile strength notably less than the tensioned tows in the longitudinal and transverse directions. While there was less of a drop in compressive properties in the longitudinal direction, the strength of the stitched case in the transverse direction was approximately 7% greater. No significant drop in modulus was observed due to stitching. It can be concluded that non-crimp carbon fabrics with powder epoxy matrices offer exceptional stiffness properties making them a suitable candidate for the production of spar caps in wind blades.

1. Introduction

The wind energy market has seen significant investment in recent years and is continuously growing [1]. Due to developments in offshore technologies and European Union (EU) targets, the wind energy sector in the EU has seen growth from 2.4% to 11.4% in electrical demand between 2000 and 2015 [2], [3]. As larger rotor diameters are more economical, and hence more popular, increasingly thicker root sections are required to bear the larger moments and loads generated. The vast majority of these blades are currently made from fibre reinforced polymers (FRPs), mainly glass fibre reinforced polymer (GFRP) in the shell and carbon fibre reinforced polymer (CRRP) in the spar caps. The primary purpose of spar caps in wind turbine blades is to increase the blade's bending stiffness and carry the large loads endured over time. Distributed loads across the blade's length are experienced due to wind pressure, causing bending. This is one of the many load cases endured by a functioning blade which varies depending on angle of rotation, wind speed and many other factors. The bending caused by wind loading is resisted mainly by the spar caps, which bear massive compressive and tensile forces.

Unidirectional CFRPs are often required in the spar caps due to their high specific stiffness and strength. Carbon fibres reinforcements can take many forms, including fabrics where the individual carbon tows are either woven or stitched, and fibre sheets which have been pre-impregnated with resin (prepregs). While the former is often used in vacuum infusion processes and resin transfer moulding, high pressures and temperatures are often required to process the latter, usually requiring an autoclave, an expense which is justified mainly by the aerospace industry for premium quality. In cases where most of the loading occurs in one direction such as spar caps, unidirectional CFRPs can effectively handle principal stresses. Woven fabrics are known to possess poorer mechanical properties than prepregs due to fibre disorientation and crimp, whereas non-crimp fabrics (NCFs) offer superior fibre straightness, something which is critical in handling high loads [4]. Warping causes reduced strength, which can lead to premature failure of structures due to micro buckling in compression, something which is less likely to occur for non-crimp fabrics [5][6]. Non-crimp fabrics are an effective solution with good drapability but they are often difficult to handle due to movement between tows and fragility of the stitching.

Though a decrease in properties have been observed in many studies due to stitching, others reported an increase in properties with the exception of compressive strength where in almost all cases, an 11-16% reduction in the longitudinal direction was observed [7]. In the transverse direction, stitching can often improve properties, depending on the strength and orientation of the stitch. Pultrusion processes are known for exceptional fibre straightness which is gained during the process by applying tension. Mamalis et al. developed an apparatus which can effectively replicate the straightness achieved from pultrusion processes by tensioning fibre tows during the cure [8]. This method is not a viable one for large scale production of parts but can be used for material characterisation as a benchmark for comparing fibre straightness with other processes.

Out-of-autoclave techniques such as vacuum infusion are popular for manufacturing shells in wind blades, however the spar caps are often bonded separately as a post-process using adhesives, which is time consuming. As a result, there is a strong demand for cheaper manufacturing processes within the industry. Recent developments in vacuum bag only (VBO) processes for the manufacture of aerospace components have shown that autoclave-quality results can be achieved by partially impregnating fibre reinforcements with resin [9]. The partially impregnated *semi-preg* materials allow for the evacuation of moisture and air via dry fibre pathways, which can result in reduced void formation. Research is underway using such VBO processes, innovative tooling and powder epoxy technologies which have demonstrated significant advantages for manufacturing wind turbine blades [8], [10], [11]. The use of such VBO systems allows for a *one-shot* production process whereby the shell and spar caps can be made in a single heating cycle, thus cutting out expensive post processing steps using adhesives.
For the production of thick root sections in blades, resin systems with a low exotherm, long infusion window and rapid cure response are desirable [12]. Powder epoxy systems consisting of resin, hardener and other additives possess reaction enthalpies during cure that are approximately 10% of standard resins used for infusion [11].

In this study, the mechanical properties of CFRP laminates manufactured using unidirectional stitched NCF and powder-epoxy were determined. As an effective control, laminates were produced from carbon fibre tows with powder-epoxy using a tensioning apparatus designed by Mamalis et al. [8] and were tested such that tensile and compressive results using the stitched case could be compared.

2. Methodology

2.1 Materials

Two different forms of carbon fibre reinforcement were used in this study: (i) a unidirectional non-crimp stitched carbon fabric (U-C-529g/m^2-500mm) from SAERTEX® containing ZOLTEK PX35 50K fibres with polyester (PE) stitching in a tricot pattern; and (ii) an array of continuous carbon fibre tows (T700S-24K-50C, 800 g/1000m) from TORAYCA®. For both cases, the matrix material used was a heat activated powder-epoxy resin (EC-CEP-0016) from ÉireComposites Teo. which contained all reactants in the powder mixture. The material properties are summarised in Table 1. below. The CFRP laminates produced are referred to as the stitched and tensioned cases respectively.

Table 1. Summary of basic properties of the constituent materials [13][14][8].

Name	Tensile Modulus (GPa)	Tensile Strength (MPa)	Density (g/cm^3)
SAERTEX® U-C-529g/m^2 CF	242	4137	1.81
TORAYCA® T700S CF	230	4900	1.80
EC-CEP-0016 Powder-Epoxy	2.99	73.09	1.22

2.2 Manufacture of laminates

For both the stitched and tensioned cases, materials were laid up on a CFRP caul plate with a coefficient of thermal expansion similar to that of the carbon fabric to minimise relative displacement between the plies and lay-up surface. A relatively thick polyimide release film (75μm) was used between the caul plates and dry lay-up to prevent wrinkling. Alternating layers of resin and fabric were used such that a fibre volume fraction (V_f) of approximately 50% would be achieved, accounting for the displacement of resin on the edges and bleeding. The powder was spread using a paper strainer with a fine sieve filter to distribute the resin as evenly as possible for consistent laminate properties. Laminates for the stitched case were produced using 2/4 plies with 3/5 layers of resin and those for the tensioned case with 5/10 plies and 6/11 layers of resin. This was carried out to meet test standard specifications such that the resulting thicknesses would be as close to 1 mm / 2 mm respectively while also maintaining the desired V_f. For the stitched case, a layer of breather cloth was applied followed by bagging with a vacuum breach. The bag was sealed along the periphery to a stainless steel base plate using sealant tape as shown in Fig. 1 (a). For the tensioned case, a novel hand lay-up method using the discussed tensioning apparatus was used to achieve high levels of fibre straightness. The tow ends were placed in between steel clamps at either end of the tensioning device with rubber pads on the surface to improve gripping as shown in Fig. 1 (b). Bolts were used to tighten the clamps to prevent slippage. The fibres were then tensioned by rotation of bolts on threaded bars, thus increasing the distance between the clamps.

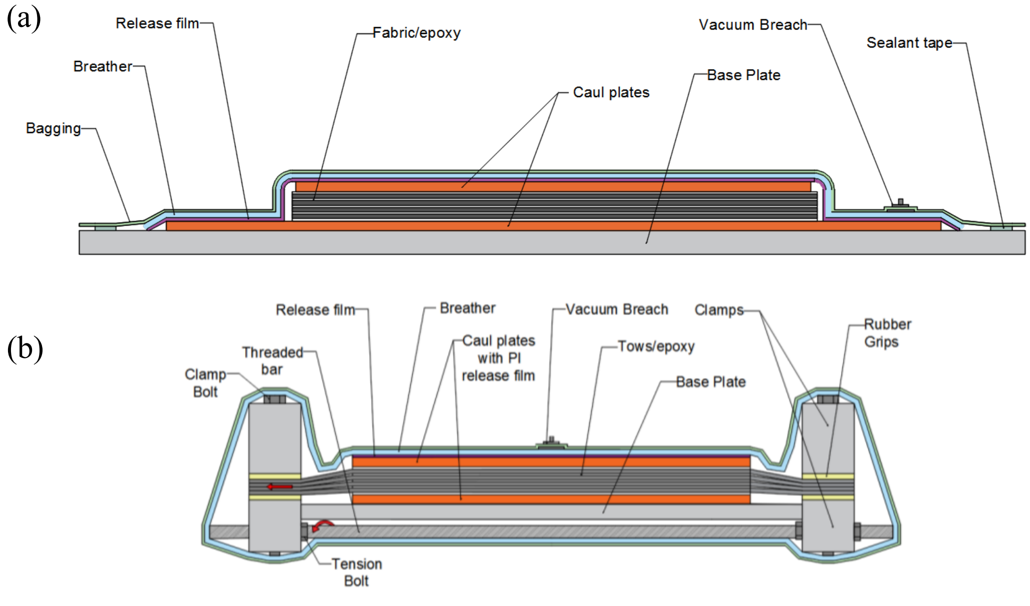

Figure 1. Assembled lay-up for (a) the stitched case using a standard VBO process and (b) the tensioned case which applies tension to fibres to increase straightness.

2.3 Drying and curing cycles

The same drying and cure cycles were carried out for both the stitched fabric and tensioned tows. It was demonstrated by Maguire et al. [11] that such powder epoxies are slightly hygroscopic, capable of absorbing up to 0.6% moisture in ambient storage conditions and so, an oven drying cycle for 1000 minutes under vacuum at 55°C was used to remove moisture from the lay-up. The temperature was then ramped up to 120°C at a rate of 90°C/hour, followed by a dwell for 90 minutes. The decreased viscosity resulting from this step allows fibre infiltration to occur. For the cure cycle, oven temperature was ramped up to 180°C at a rate of 90°C/hour followed by a dwell for 150 minutes at this temperature.

2.4 Inspection and testing methods

Inspection of laminates was carried out at ÉireComposites Teo. and specimen preparation and testing was carried out at Composites Testing Laboratory, an ISO17025 and NADCAP approved testing laboratory. Initial inspections were carried out on each laminate using a MIDAS NDT ultrasonic C-scan unit to determine any defects or inconsistencies that may have been present. The thickness of each laminate was measured in over 50 locations to determine areas that may be resin rich/poor due to process anomalies.

In order to determine density, the mass in air and apparent mass in distilled water of three samples from each laminate were measured using an analytical balance and immersion apparatus. The density was then determined using Archimedes' principle. Samples underwent acid digestion in accordance with BS EN 2564 and the fibre volume fraction and void content were determined from mass lost to the dissolved resin.

For both manufacturing cases, an identical test matrix was used to determine tensile and compressive properties in the 0° and 90° directions. All mechanical tests were carried out in a controlled environment at 23 ± 2 °C with a relative humidity of 50 ± 10% and at least 7 specimens were used for each test type. For initial measurements of strain, an extensometer was used to accurately determine the modulus and was removed prior to failure. Tensile testing was carried out in accordance with BS EN 2561 (for longitudinal properties) and ASTM D3039 (for transverse properties) using a calibrated Zwick/Roell Z250 universal test machine with a 250kN load cell. The specimens were end-tabbed by bonding ±45° glass fibre-epoxy material to the gripping surfaces using a high strength adhesive. Compression tests were carried out in accordance with BS EN 2850 for determination of both longitudinal and transverse properties and were end-tabbed with CFRP material.

Dynamic mechanical thermal analysis (DMTA) was carried out to determine the glass transition temperatures and observe the storage modulus as this has been shown to relate to interfacial adhesion between the fibre and matrix [15]. A single cantilever bending configuration was used in accordance with prEN 6032 on a Tritec 2000 DMA. Specimens were dried for 48 hours at 70°C prior to testing. A heating ramp rate of 5°C/min was used from 23°C to 200°C with a test frequency and displacement of 1Hz and 0.03mm respectively.

3. Results

3.1 Inspection

The laminates manufactured from the stitched fabric (Fig. 3 (a)) and tensioned tows (Fig. 3(b)) were both produced successfully with a high quality surface finish in both cases. Resin gathered at the edges of all panels due to bleeding as expected. Results from the ultrasonic C-scans (Fig. 4 (c)) were reasonably consistent for both cases with no obvious defects or anomalies present in laminates however larger variations were observed for the stitched case. Optical microscopy was carried out on samples to detect possible macro voids (Fig. 4 (e)) and wet-out of tows (Fig. 4 (f)). Results were consistent in all cases, with little/no detection of voids either between or within fibre tows. The thickness of laminates for the stitched case were 2.32 ± 0.08 mm and 1.15 ± 0.04 mm and those for the tensioned case were 1.02 ± 0.08 mm and 2.03 ± 0.15 mm.

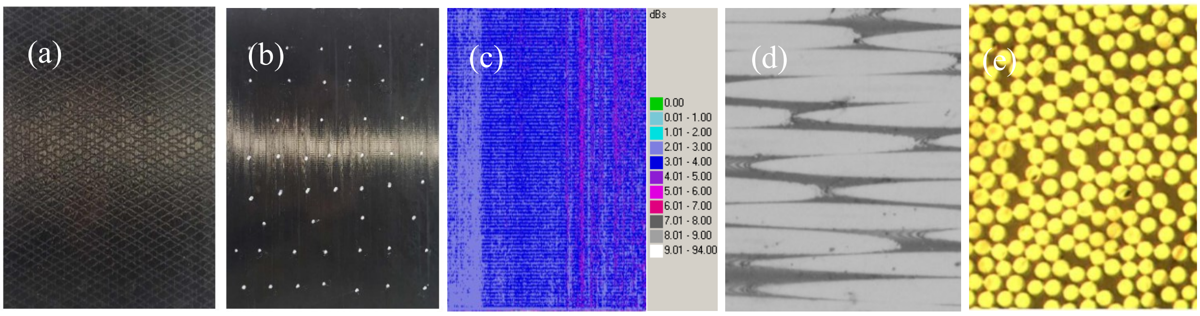

Figure 3. Laminates produced from (a) UD stitched fabric and (b) tensioned tows. (c) is an example of an ultrasonic C-scan, (d) typical microscopic image showing several fibre tows and (e) typical microscopic image showing individual fibres within a tow.

3.2 Density and fibre volume fraction

The density of the composite, fibre volume fraction and void content were determined using Archimedes' principle and mass loss due to acid digestion for three samples from each laminate. The results from these tests are summarised in Table 2. below. The values for V_f given here are average values and are coincidentally the same for each case. Normalisation of test results was still required using the values from each individual laminate.

Table 2. Summary of density, fibre volume fraction and void content

Material Case	Composite Density	Fibre fraction by volume	Void fraction by volume
	ρ_c (g/cm^3)	V_f (%)	V_v (%)
Stitched	1.493 ± 0.027	50.3 ± 2.7	1.5 ± 1.6
Tensioned	1.540 ± 0.040	50.3 ± 3.0	1.0 ± 0.2

3.3 Mechanical properties

The results for all mechanical tests carried out are summarised in Table 3 below. Results indicate that the laminates produced using the stitched fabric have inferior tensile properties to those produced using the tensioned tows. For the longitudinal tensioned specimens, full delamination occurred in an explosive fashion for the entire set while for the stitched specimens, failure occurred in the direction of the stitch. Typical failure was observed in the net section for all transverse specimens. The longitudinal compressive properties for the tensioned case are superior but not by a significant amount when the difference in fibre strength is considered. Nearly all compression samples failed in the net section, with the exception of some transverse samples failing in the clamps. Due to the large number of specimens for this test (12 in this case), this was not an issue.

Table 4. Tensile and compressive modulus and strength results in both the longitudinal and transverse directions.

Material type	Tensile properties				Compressive properties			
	Modulus (GPa)		Strength (MPa)		Modulus (GPa)		Strength (MPa)	
	E_{1t}	E_{2t}	S_{1t}	S_{2t}	E_{1c}	E_{2c}	S_{1c}	S_{2c}
Stitched	106 ± 2	6.4 ± 0.2	1534 ± 73	37.9 ± 1.8	99 ± 5	8.5 ± 0.2	757 ± 83	175 ± 8
Tensioned	117 ± 3	7.1± 0.2	2259 ± 84	54.8 ± 5.6	106 ± 3	8.8 ± 0.3	902 ± 69	164 ± 10

3.4 Thermomechanical properties

Dynamic mechanical thermal analysis was carried out on 9 samples for each case (3 samples from 3 laminates) to determine thermomechanical properties. Results for glass transition values are presented in Table 5. Tg_{onset} indicates the first drop in storage modulus (E') where increased chain mobility occurs in the polymer. The temperature at which glass transition occurred for the NCF was slightly lower than that for the tensioned case. The value for the storage modulus for this case was higher and the area under the tan δ curve was noticeably less. All three of these observations indicate superior interfacial adhesion for the tensioned case [15].

Table 5. Values for glass transition temperature found using dynamic mechanical thermal analysis. The onset of glass transition was determined by the first drop in $E'$and by the peak from the crest of the tan δ curve.

Material Type	Tg_{onset} (°C)	Tg_{peak} (°C)
Stitched	101.6 ± 2.3	116.8 ± 2.3
Tensioned	104.0 ± 3.3	117.0 ± 1.7

4. Discussion

4.1 Inspection

Variation in the C-scan images for the fabric case is not unexpected. Firstly, the fibre tows in the fabric are 50 K compared to 24 K in the tensioned case, though the fibre diameter is similar (7.2 µm v. 7 µm) [14][13]. Due to the larger tows in the fabric and the fact that less layers were used means that the distinction between the tows and resin were more noticeable. Because fibres in stitched fabrics are tightly compacted and due to the large size of the tows in this case, it was originally suspected that dry fibres may have been present within the tows of the NCF [16]. However, optical microscopy confirmed that tows were fully wetted out in all cases.

4.2 Density and fibre volume fraction

Acid digestion methods are often used to determine the resin fraction by volume in composites by subtracting the mass loss from that of the composite prior to digestion. It should be noted that the polyester stitching was also dissolved along with the epoxy matrix and fibre sizing during acid digestion and so, the exact resin content cannot be determined from this test. The void content for the stitched case is inconclusive as the standard deviation is too high. This shows that manual powder spreading is subject to much variation. Automated spreading however could result in more repeatable results.

4.3 Mechanical properties

The stitch clearly played a role in premature tensile failure in the longitudinal direction as cracks in the net section propogated in the direction of the stitch in several locations during failure. As the stitching is at angles of ± 60° to the fibre direction, additional forces are generated towards the centre of the specimen. With the addition of the stress concentration caused by the stitching, the observed reduction in strength was expected. It is important to note that the strength of the fibres used in each case is different but even when tensile results are normalised based on fibre strength, the tensile strength for the stitched composite case is 20% less than that for the tensioned case. With fibre strength considered, there is no sign that stitching caused a reduction in compressive properties. Transverse properties for the stitched laminates were notably better than those in tension, matching observations by Yudhanto et al. [7].

4.4 Thermomechanical properties

It is not surprising that interfacial adhesion between the fibre and matrix would be poorer for the stitched case. It has been shown in literature that ZOLTEK PX35 fibre tows in powder epoxy have poorer interfacial properties compared to TORAYCA® T700S fibre tows [17]. In this study, the observed effects were attributed to the distribution and volume of sizing on the fibre surfaces, confirmed by FTIR analysis. The study showed that flexural strength of the PX35 in the transverse direction was found to be ~38% of that for the T700S with the 50C sizing, indicating significant differences.

5. Conclusions

Laminates were manufactured successfully using a non-crimp UD stitched fabric and were compared to laminates produced using UD tows with a tensioning device, resulting in superior fibre straightness. There was a noticeable drop in longitudinal tensile strength in the NCF with clear indications that failure occurred along the stitching. Though the compressive properties in the longitudinal direction were less for the stitched case, it cannot be confirmed that this was due to the stitching. On the contrary, transverse compressive properties performed better as a result of the stitching. Overall, the study shows that there is no significant difference in modulus/stiffness between the two cases. It is clear that unidirectional stitched fabrics are a suitable candidate for use in spar caps of wind turbine blades.

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