

FLAT GLASS FIBRES: A STUDY ON FLAT GLASS FIBRES AND THEIR INJECTION MOULDED POLYAMIDE 6,6 COMPOSITES

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ABSTRACT

The glass fibre reinforcement industry continues to enjoy continuous growth due to the challenges faced by the global climate crisis and the need for lightweighting across multiple sectors. However, as a well-established and mature industry, stepwise changes to glass fibres are required to maintain the level of innovation that has preceded present-day products. One such change is flat glass fibres. While already used on the industrial scale, flat glass fibres have had less impact on the academic literature. With patents and press releases stating flat glass fibres lead to improved mechanical properties, processability and bonding, more academic evidence is needed to explain why this might be. At the most fundamental level, an understanding of the properties of individual flat glass fibres is required. The properties of individual fibres dictate how they will reinforce and interact with various polymer systems. This paper discusses the initial results of such characterisation, with improvements in fibre tensile strength and modulus found for sized flat fibres versus circular fibres. Accurate characterisation of fibre cross-sectional area is found to have a significant effect on the values obtained for fibre tensile properties. The variation in fibre geometry and tensile strength leads to changes in the critical fibre length. Residual fibre length of glass-reinforced polyamide 6,6 was measured, with flat fibre composites exhibiting a longer fibre length. Coupled with a lower critical fibre length, this should translate to improved mechanical properties. Yet, only marginal differences are noted at high fibre weight fractions. When conditioned for 24 hours in boiling water, however, the properties of flat glass fibre composites are enhanced compared to their circular counterparts at high weight fractions. From the results presented, flat glass fibres improve various properties of short-fibre reinforced thermoplastics; however, further investigation is required to understand why and realise these benefits further.

1 INTRODUCTION

Composite materials are at the heart of the shift towards a net zero economy. They enable the transition to new fuel sources in transportation sectors with their high strength-to-weight ratio creating lightweighting opportunities across various sectors. Glass fibres (GFs) account for over 90% of the reinforcement fibres used in fibre-reinforced composites. In particular E-glass fibre is frequently used in high-rate production processes, such as injection moulding which service transportation sectors [1, 2]. Innovation in reinforcement fibres in these applications is required to maintain the level of progression which has proceeded in the previous century. Flat glass fibres are a recent development that creates a stepwise change in the glass fibre industry.

Flat glass fibres are fibres with a ‘flattened elliptical’ cross-section. While they have been available on the market for some time and are currently used in industrial applications, their impact on academic literature is less pronounced. Only a handful of papers have been published exploring their influence on composite properties [3-7]. The bulk of these focus on injection moulded composites, where the reduced second moment of area of the fibre is thought to lead to an increased residual fibre length and, as a result, improved mechanical properties; particularly at higher fibre weight fractions [3-5]. However, there

remains a lack of detail on the properties of individual flat glass fibres and a body of knowledge which explains how the fibre cross-section influences the micromechanics of these composites [2].

For nearly a century, it has been known that the cross-sectional area of individual glass fibres influences their tensile strength [8, 9]. More recent studies have highlighted significant variations in the cross-sectional area of commercial glass fibres [10, 11]. Therefore, it is vital to understand how best to characterise the cross-sectional area of novel-shaped fibres to generate reliable data on single fibre tensile properties and subsequently improve our understanding of the micromechanics of composites fabricated from such fibres. Thomason has recently explored the analytical relationships between fibre cross-section and composite micromechanics [2, 12]. Thomason modified the commonly used Kelly-Tyson equation to explore the relationships between fibre cross-section shape, the critical fibre length, and composite strength.

Studies have already shown the residual fibre length of flat glass fibres to be greater than circular fibres [4, 5], however, no studies have investigated length degradation throughout the injection moulding process. Additionally, the research community is yet to have thoroughly examined properties at all characterisation scales, from the fibre to the interphase and finally to the composite. This paper presents some of the initial findings of such a study.

2 EXPERIMENTAL

2.1 Materials

Rovings of circular and flat E-glass fibres (2400 and 300 tex, respectively) were provided by Nippon Electric Glass (NEG). Rovings were supplied as sized (T262, polyamide compatible), and unsized. The composition of the glass fibres is the same. Circular glass fibres supplied had a nominal 13-micron diameter, and flat glass fibres were of an equivalent cross-sectional area, with an aspect ratio of 4:1. NEG also supplied injection moulded circular and flat glass fibre-reinforced polyamide 6,6 (Ultramid A27E) (PA6,6) in different weight fractions (30, 35, 40, 45, 50, 55 wt%).

2.2 Single fibre cross-sectional area assessment

The cross-sectional area of single fibres was characterised using 3 different techniques:

- Transverse optical microscopy – using a Leitz Ergolux microscope with fibres glued onto card windows typical of those used in single fibre tensile testing, as described in section 2.3. The fibre diameter was measured directly using ImageJ, and the cross-sectional area estimated from the diameter measurement.

- Cross-sectional reflected optical microscopy – using an Olympus GX51, the individual fibres on the end tabs of single fibre tensile test templates were cut and mounted vertically, and then embedded in a mounting resin. The samples were prepared for microscopy by grinding and polishing progressively with different grade papers and diamond paste. The fibre cross-section was measured directly using the circle/polygon tool in ImageJ.

- Cross-sectional scanning electron microscopy (SEM) – using a Hitachi TM3000 benchtop SEM, mounted specimens (prepared as described above), were placed in the machine, uncoated, and captured images were assessed in ImageJ using the circle/polygon tool.

The image outputs of the three different methods are shown in Figure 1.

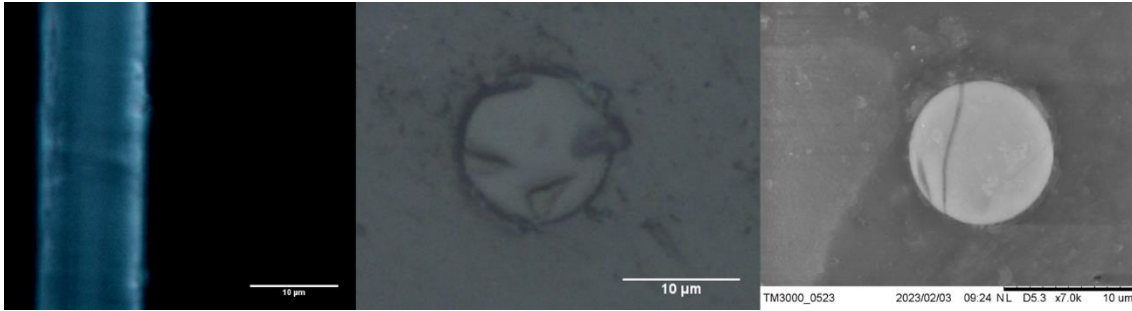


Figure 1 Comparison of different measurement techniques for the same fibre, from left to right: transverse optical, cross-sectional optical and cross-sectional SEM.

2.3 Single fibre tensile testing

Single fibre tensile testing was conducted following the method described in ASTM C1557-03. Single fibres were separated from the received bobbins and glued (Locite™ Gel Superglue) onto card (250 g/m²) tabs, with a section of card removed to match the desired gauge length for the test. Testing was conducted at three different gauge lengths; 20 mm, 40 mm and 80 mm. Subsequently, the compliance was estimated, and modulus results were corrected. Testing was undertaken on a Testometric M250-2.5CT with a 500 gf load cell. A minimum of 75 fibres were tested for each averaged datapoint. During testing, the type of failure was recorded, as either: shattered, clean (with remains), or clean (with remains) near the end tab. For the circular fibres, the cross-sectional area was evaluated using the transverse optical method described above. For the flat fibres, the cross-sectional area was evaluated using the cross-sectional SEM method described in the section above.

Weibull analysis was applied, consistent with ASTM C1239-07. A uni-modal model was applied to the data, using Equation 1:

$$P = 1 - \exp\left(-\left(\frac{\sigma}{\sigma_0}\right)^m\right) \quad (1)$$

Where P is the probability of failure, σ the stress at a given probability, σ_0 the characteristic strength, and m the Weibull modulus.

2.4 Fibre length analysis

Fibre length analysis was conducted using an IDM FASEP fibre length measurement system. For analysing the residual fibre length in composite samples, a 65mm section from an ISO type 1A dogbone specimen was cut at the centre and placed inside a ceramic crucible. The ceramic crucible was placed inside a Carbolite No8 furnace for 30 minutes at 650°C. Subsequently, the remaining fibres were dispersed in a plastic jug with 1L of deionized water. The concentration of fibres was reduced using a multi-stage dilution series, with half the water-fibre mixture decanted repeatedly, until an appropriate quantity of fibres were present in the jug; not too many that there would be too much fibre-fibre overlap; not too little so an adequate amount of data could be generated. The mixture was then poured into a plastic petri dish and placed inside the FASEP machine. Around 1000 fibres were scanned in each petri dish, and a minimum of 4000 fibres were scanned for each sample. Data was subsequently post-processed using an in-house Python script.

2.5 Composite tensile properties

Composite tensile testing was conducted in accordance with ISO 527-2, using a Testometric X500-50 (50kN load cell, 50mm extensometer), with self-tightening wedge grips. ISO type 1A bars were produced in a Battenfeld 750CD twin-extruder. Samples were stored in air tight aluminum bags with self-indicating silica gel to maintain a dry as moulded state until testing.

Conditioned samples were prepared in a waterbath at 100 °C for 24 h, before being cooled to room temperature in a water bath initially at 90 °C, for 1 h, and allowed to air dry for 30 min before being tested.

3 RESULTS & DISCUSSION

3.1 Fibre geometry

A comparison between the tensile strengths at 20 mm gauge length with the different cross-sectional area measurement methods outlined in section 2.1 is shown in Table 1. There are significant differences in the values reported and subsequent conclusions depending on the measurement method used.

	Optical, transverse diameter	Optical, cross-sectioned	SEM, cross-sectioned
Mean Cross Sectional Area (μm^2)	145 ± 6	134 ± 9	143 ± 7
Mean Fibre Tensile Strength (MPa)	2080 ± 100	2315 ± 158	2345 ± 174

Table 1 Average tensile strength and CSA of unsized, circular fibres, tested at 20mm gauge length, and the error at 95% confidence, measured with different cross-sectional area methods.

For each 20mm circular filament, a comparison between the cross-sectional area calculated by the transverse diameter and the two different cross-sectioning (optical and SEM) methods is presented in Figure 2. The data obtained from optical cross-sectioning is significantly different from the traditional transverse method. This is likely due to poor contrast differences between the fibre and mounting epoxy, edge effects at the fibre/epoxy interface, and limited magnification compared to SEM. The significantly lower gradient for the SEM cross-sectioned line indicates that the cross-sectional area measured using the SEM method is closest to the of cross-sectional area as traditionally determined by transverse optical methods.

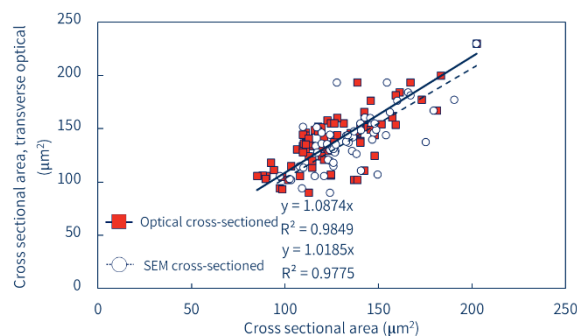


Figure 2 Comparison between the cross-sectional area determined using the optical transverse diameter measurement, and the cross-sectional optical and SEM methods, comparing individual measurements for single fibres.

Flat fibres are particularly laborious to work with due to their orientational preference when laying onto card. Of the 487 single flat glass fibres mounted for transverse observation, it was found that only 31% of these fibres lay on their major axis, with the majority of fibres being at an intermediate orientation between the major and minor axis. Therefore, even if a relationship could be established between the major/minor axis of the fibre and its cross-sectional area, the low frequency of observation of the major/minor axis makes this impractical. As a result, transverse optical observations of flat glass fibre dimensions were not used for determining the fibre tensile properties. Additionally, as can be seen

in the images in Figure 3 the flat glass fibres can be skewed in their cross-section, so relationships between the major/minor axis based upon standard geometrical equations may not always be valid.

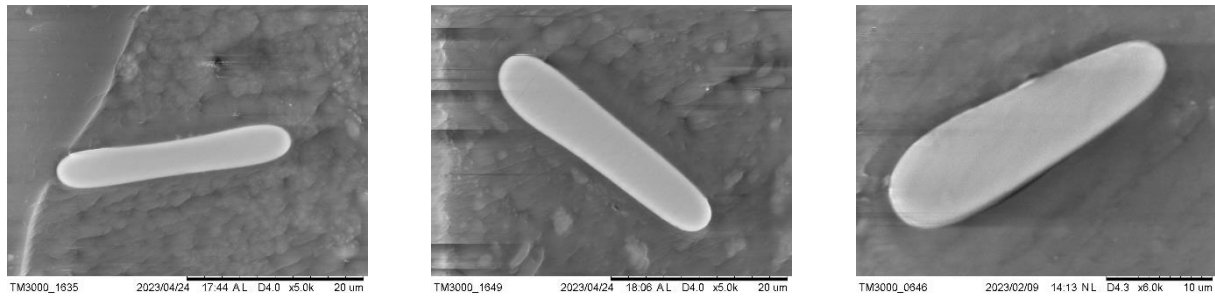


Figure 3 Images of flat glass fibres, highlighting the sometimes-skewed nature of the cross-section, using the cross-sectioned SEM method.

3.2 Fibre tensile properties

The tensile strengths at 20 mm, 40 mm and 80 mm gauge lengths are presented in Figures 4. The sized flat fibre has a consistently higher tensile strength when compared to the sized circular fibre, while significant differences between the flat and circular unsized fibres are not always observed across the different gauge lengths. Irrespective of fibre shape, however, the importance of sizing application remains critical to maximizing the extrinsic fibre strength. Given no differences in batch composition, further investigation is required to understand where extrinsic strength differences arise. Nonetheless, the increase in strength due to the cross-sectional shape is consistent with the limited data in the literature [5, 7].

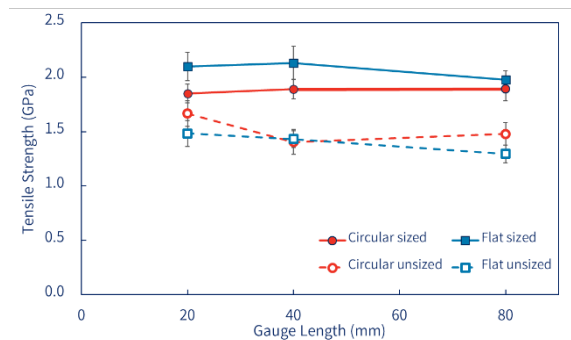


Figure 4 Tensile strength of flat and circular, unsized and sized fibres, at 20 mm, 40 mm and 80 mm gauge length, with 95% confidence interval error bars.

The failure behaviour of each fibre was recorded during testing. Fibres which shattered were marked as 'S', while fibres which broke 'cleanly', with remains after the test, were marked C. Fibres which broke near the end tabs were marked B/T. The data recorded at 20mm is shown in Figure 5 — the majority of circular fibres, sized or unsized, shattered at failure. While for flat fibres, sized or unsized, fibres generally tended not to shatter more frequently. Similar to the data for strength, this would imply something different, intrinsically or extrinsically, altering the failure behavior of the glass fibres based on their cross-sectional shape.

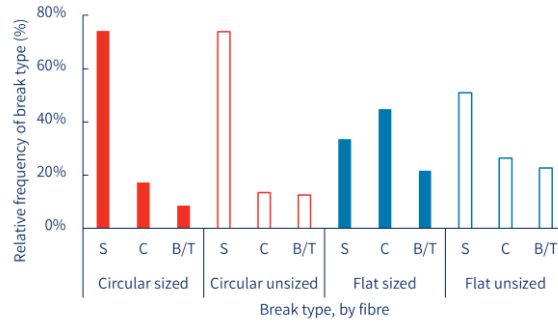


Figure 5 Failure behaviour of circular and flat, sized and unsized fibres, at 20 mm gauge length, where: S, denotes shattered fibres; C, fibres which broke cleanly with remains; B/T, fibres which broke cleanly with remains near the end tabs.

Many authors assess the tensile strength of glass using Weibull analysis. Uni-modal Weibull plots for the sized and unsized fibres are shown in Figure 6. The characteristic strength of the sized flat glass fibre is greater than the sized circular glass fibre, while the characteristic strength of the unsized flat glass fibre is lower than the unsized circular glass fibre.

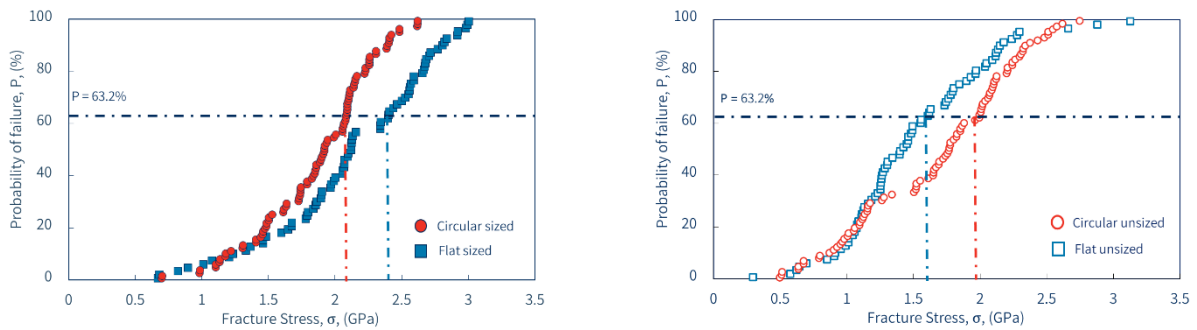


Figure 6 Uni-modal Weibull plots for circular and flat, sized (left) and unsized (right) fibres, with the characteristic strength at 63.2% P shown by the dotted lines.

The strain and modulus results were corrected for compliance with testing conducted at multiple gauge lengths, shown in Figure 7. The compliance corrected modulus for the flat fibres was 76 GPa and 79 GPa, while for circular fibres, 69 GPa and 68 GPa, for sized and unsized, respectively. Similar to the tensile strength, the sized flat fibres have a greater tensile modulus, consistent with the limited literature. As found previously, the modulus for unsized fibres is greater in the case of flat and circular fibres. A search of the patents shows that in some cases, the forming temperatures may be different to alter the viscosity, preserving the flat cross-section as it is tension wound. Forming temperature has been shown to influence the tensile properties of the fibre [8]. Additionally, the difference in failure behavior highlighted in Figure 5 may suggest the fracture behavior of flat glass fibres differs from circular fibres. A summary of the: characteristic strength; Weibull modulus, estimated using linear regression; and compliance corrected tensile modulus is presented in Table 2.

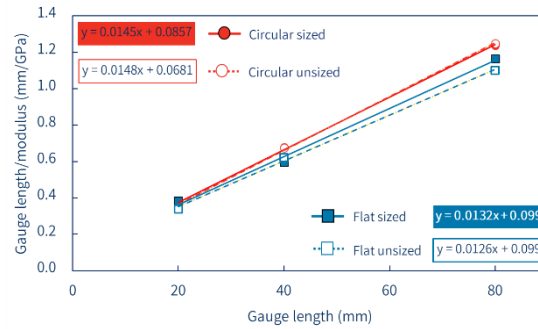


Figure 7 Fibre modulus and compliance analysis at 20 mm, 40 mm & 80 mm gauge length, for circular and flat, sized and unsized fibres.

	Circular unsized	Circular sized	Flat unsized	Flat sized
Mean Tensile Strength (MPa), @20mm gauge length	1666	1850	1483	2100
Characteristic strength (MPa), @20mm gauge length	1995	2084	1608	2402
Weibull modulus, @20mm gauge length	3.0	4.7	3.2	3.7
Compliance corrected tensile modulus (GPa)	68	69	79	76

Table 2 Summary of the mean tensile strength, characteristic strength, Weibull modulus at 20 mm, and the compliance corrected modulus.

3.3 Fibre length analysis

Fibre perimeter, cross-sectional area, and tensile strength are required for commonly applied micromechanical models [13-16]. With these parameters characterised, material scientists can predict how a change in fibre shape may influence composite properties, including the critical fibre length (L_c). An increase in the fibre surface area, will tend to decrease the critical fibre length. Meanwhile, increases in fibre cross-sectional area and fibre strength tend to increase the critical fibre length. Since flat fibres have a greater perimeter than circular fibres for an equivalent cross-sectional area, they should theoretically be more efficient at transferring stress i.e. have a lower critical fibre length. The equation for critical fibre length is shown below:

$$L_c = \frac{A_o}{P_o} \left(\frac{2\sigma_f}{\tau} \right) \quad (2)$$

Where A_o and P_o are the fibre cross-sectional area and perimeter respectively, τ is the interfacial shear strength (IFSS), and σ_f the tensile strength of the fibre.

Using the data from section 3.1 and 3.2, and an assumption that the IFSS of glass/polyamide 6,6 is 30 MPa, based upon the literature [17], the critical fibre length can be calculated using Equation 2. In Figure 8, the cumulative frequency of fibre length and the calculated critical fibre length are shown, for flat and circular fibre compounds, at 30 wt% and 55 wt%. At 30 wt%, c.44 % of flat glass fibres are greater than the critical fibre length, meanwhile for circular, only c.37% are greater. Similarly, at 55 wt%, c.35% of flat glass fibres are greater than the critical fibre length, and only c.18% of circular fibres are greater.

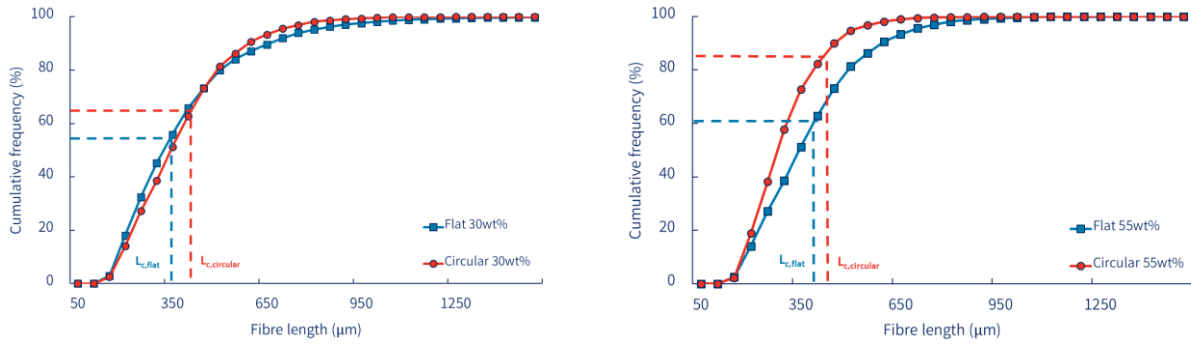


Figure 8 Cumulative frequency of fibre length plots at 30wt% (left) and 55wt% (right) of fibre, with critical fibre length denoted by the dashed lines.

From the 55wt% curves, it is apparent that the flat glass fibre compound has a higher fraction of longer fibres. More broadly, flat fibre compounds tend to have a greater residual fibre length across the measured weight fractions, as shown in Figure 9. This data supports the theory that the reduction in the second moment of area of the fibre does tend to increase fibre flexibility and reduce instances of fibre breakage during processing. This is particularly notable when assessing the volumetric averages.

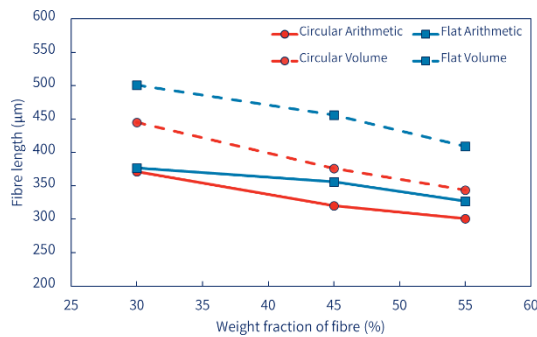


Figure 9 Arithmetic and volume mean residual fibre length of circular and flat fibre reinforced polyamide 6,6, composites, with fibre weight fractions of 30 wt%, 45 wt% and 55 wt%, with standard error, error bars.

When assessing the residual fibre length distributions in the composite bars, and the input chop fibre, the flat fibre shape tends to decrease fibre breakage throughout the production process. As can be seen in Figure 10, the flat glass fibre shape leads to a broader distribution of fibre lengths in the virgin chop product.

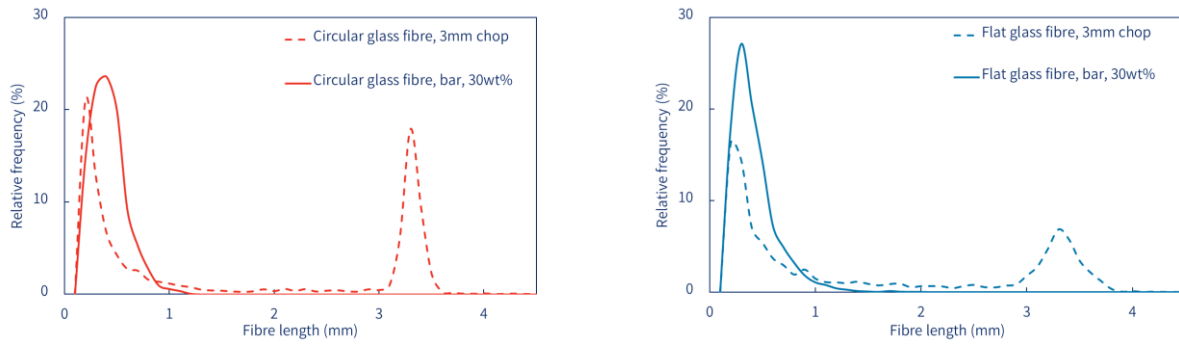


Figure 10 Fibre length distributions for virgin input chop and residual fibre length in composite bars with 30wt% fibre, circular fibres left, flat fibres right.

The combination of a reduced critical fibre length, and increased residual fibre length, should lend itself to increased composite tensile strength.

3.4 Composite tensile strength

As discussed, the increased fibre tensile strength, decreased critical fibre length, and increased mean fibre length, should result in improved composite tensile properties. In Figure 11, the ultimate tensile strength and failure strain are shown for glass (flat and circular) reinforced polyamide 6,6 at various weight fractions.

Unconditioned, flat glass fibres don't have a significant improvement on tensile strength until above 40 wt%. At the same time, circular fibre reinforced PA6,6 has a greater strain to failure, consistently across all fibre weight fractions. Additionally, there is no statistical difference in the modulus results, other than at 50 wt%, in which the flat glass fibre compound is statistically different (at 95% confidence limits).

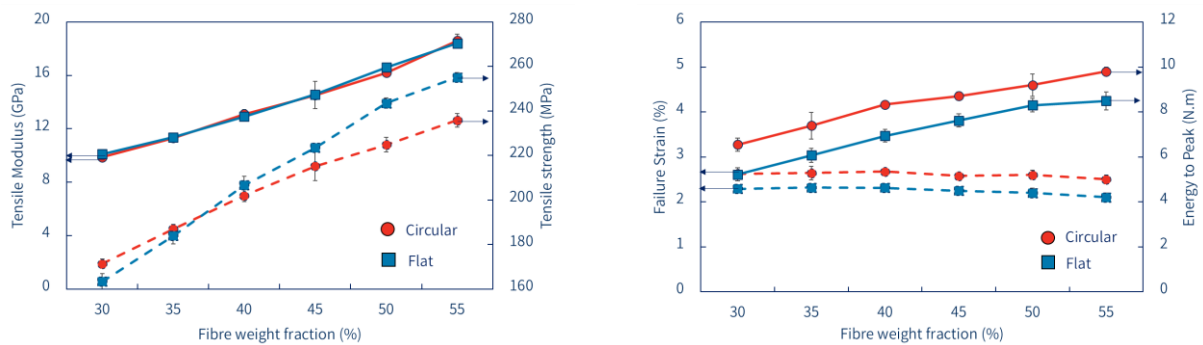


Figure 11 Failure strain and Tensile strength (left), and Energy to yield and tensile modulus (right), of unconditioned flat and circular reinforced polyamide 6,6, at 30 wt%, 45 wt% and 55 wt% fibre content, with 95% confidence interval error bars.

The improvement in tensile properties is less pronounced than the results presented in the literature [3-5].

In the conditioned composite samples, flat glass fibres have a significant impact on tensile properties. In Figure 12, tensile modulus and strength, strain to failure and energy to failure are shown. The differences after conditioning in boiling water for 24 h are significant at higher weight fractions. This is consistent with Thomason's prediction that after hydrolysis, the advantage of flat glass fibres over circular fibre will be more evident [2]. Interesting to note is the significant differences in strain to failure

and energy to yield.

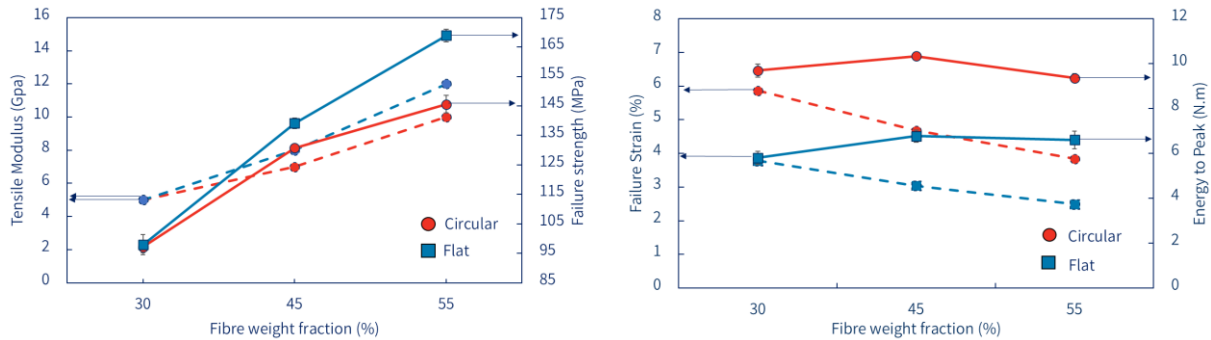


Figure 12 Failure strain and Tensile strength (left), and Energy to yield and tensile modulus (right), of conditioned (24 h boiling water) flat and circular reinforced polyamide 6,6, at 30 wt%, 45 wt% and 55 wt% fibre content, with 95% confidence interval error bars.

Given the significant differences in tensile strength, residual fibre length and critical fibre length, the question arises, *why aren't the properties significantly improved at all weight fractions?* Particularly in the dry as moulded state.

Two parameters which may be vital in understanding this are the IFSS and the fibre orientation.

The former is difficult to characterise due to the complexities associated with interfacial testing [18]. Any investigations into the IFSS of flat glass fibres vs circular glass fibres must consider the suitability of existing tests for fibres with non-circular cross-sections. The existing literature which assesses the IFSS of non-circular glass and carbon fibres lacks detail on the experimental method used, particularly how the non-circularity of the fibre is handled [6, 19].

For the latter, press releases [20-22] around flat glass fibres cite the fibres as being more 'dispersed'. In some, a more isotropic fibre dispersion is rumored to lead to more isotropic thermal expansion and, as a result, less warpage. While such claims may imply that flat fibres are orientated differently in the composite, experimental data needs to be generated to prove such claims and its impact on tensile properties.

4 CONCLUSIONS

The importance of accurate characterisation of the cross-sectional geometry has been demonstrated. Miscalculations of the cross-sectional area and perimeter can influence conclusions on fibre tensile properties and micromechanical properties. At the fibre level, it has been shown that sized flat glass fibres demonstrate improved tensile properties in terms of failure strength and modulus. There is no empirical evidence as to why there is a difference in tensile properties; however, it is postulated that changes in thermodynamic conditions during fibre forming may be at least partially responsible. The reduced second moment of area of the flat fibre is shown to increase the residual fibre length in composite bars and in the chop length of the virgin input fibre. The increased fibre perimeter and tensile strength should theoretically lead to an improved composite tensile strength through a decrease in the critical fibre length. This is true at higher fibre-weight fractions, with no evidence yet to explain the lack of improvement at lower-weight fractions. After conditioning for 24 h in boiling water, flat fibre composites possess a significantly higher modulus and tensile strength at higher weight fractions. The results of this study show how interdependent the properties of the reinforcement fibre and composite part are and the importance of thorough characterisation at all levels. Further investigation is required to understand the differences highlighted at the fibre, interphase, and composite level.

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