DEVELOPMENT OF DESIGN DATA FOR DISCONTINUOUS CARBON FIBRE COMPOSITES

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ABSTRACT

The introduction of new composite material systems and cost-efficient manufacturing routes requires the development, or adaptation, of existing material testing standards that can objectively quantify key material properties for design. The properties of discontinuous, randomly oriented, non-woven composites are inherently more variable than their established continuous fibre-reinforced counterparts, demanding some different approaches to testing. We adapted standard tests to better represent the material with significance given to test geometry, load introduction, failure modes and practical testing protocols. The material response to static and cyclic mechanical, physical thermal tests and exposure to various environmental conditions, was studied. We found that standard test procedures require some modification, and data interpretation must be undertaken very carefully.

1. INTRODUCTION

The rapid rise in carbon fibre composite usage in recent years has a large impact on sustainability issues and this has led to the development of carbon fibre recycling technologies [1]. The critical technological challenge which comes with recycled fibres is the inability to replicate the reinforcement structure of the virgin composite system [2]. The recycled carbon fibres need further processing because they are usually tangled and difficult to handle. The recycled fibres are converted into non-woven reinforcement mats and composite laminates are produced by compression moulding techniques [3]. The resulting non-woven composites have discontinuous fibres of 80 – 90 mm in length which are randomly oriented within the epoxy matrix.

The specification and use of recycled composites requires the development of data that can give a comprehensive description of the material system that can be used for design allowables. Test protocols are needed to characterise these materials that adequately represent them. Traditional materials used in structural applications and products are defined by well-established standards and handbooks. The existing standards which characterise composite materials are limited to particular composite reinforcement systems. The major barriers to the increased adoption of composites have been the lack of awareness and standards for composites [4, 5]. Several test methods exist for measuring the same property but while some tests are easy to use they can produce unreliable results, whereas more complex test methods produce more reliable results with appropriate operator skills. The continuous emergence of new materials that do not respond to loads in the same manner as the earlier developed ones makes it difficult to generate a database and standardized test scheme for the composite family [6]. The response of composites on exposure to various environmental conditions in their respective applications is also of significant interest.

2. EXPERIMENTATION

The material of interest in this research was recycled, discontinuous, non-woven mat of carbon fibre impregnated in epoxy resin. The non-woven mats with the tradename of CARBISO™ M provided by ELG Carbon Fibre Ltd. are made of recycled fibres, with the code name IM56R [7]. The reinforcement, i.e., non-woven mats have an areal weight in the range of 200 – 230 gsm. The composites are moulded

by liquid compression moulding (LCM) and sheet moulding compound (SMC) methods to a thickness of about 3.5mm, essentially consisting of 5-8 plies of non-woven mats.
The first step towards understanding the material load response of a composite system is to study the fibre architecture and distribution of fibres in the cured material. Micrographs from an optical microscope are obtained from mounting cross sections of the laminate in an acrylic resin medium and sequential polishing of the mounts with SiC grit papers, diamond suspended suspensions and alumina suspensions. They confirm the random distribution and orientation of the fibres within the epoxy matrix with fibre volume fractions calculated to be around 0.25 [8].

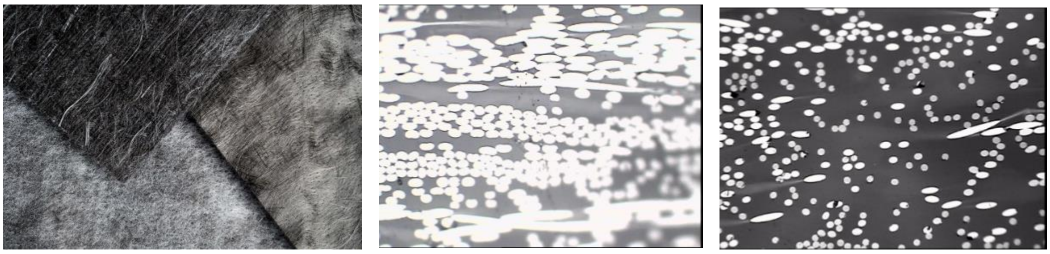


Figure 1 - Non-woven mat after carding (left) [3]; Micrographs at 50x objective magnification displaying the microstructure of the composite system (middle and right)

Composites are sensitive to specimen preparation techniques and the quality of the specimens prepared for mechanical testing is of utmost importance as they influence the reproducibility and reliability of test results [9]. The specimens were maintained at 23°C and 50% RH for at least 24 hours prior to testing. The variability of results, failure mode of the specimens and the data obtained from the above testing methods were analysed and the validity of these test methods for discontinuous, non-woven composites was assessed. The goal of this study was to develop data that is suitable for design with accuracy and repeatability adequate for comparisons and to define an appropriate specific geometry.

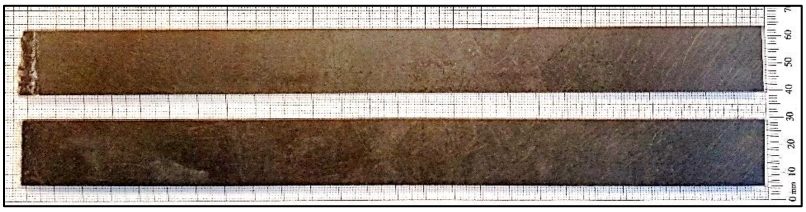
2.1 Short term mechanical testing

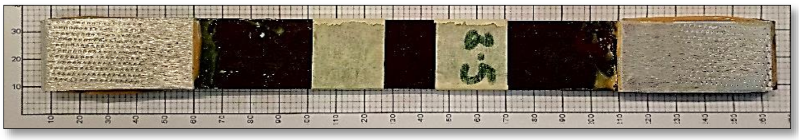
2.1.1 Tensile Tests and specimens:

	Test Method	Figure	Description
1.	ISO 527-5 [10]; untabbed	Fig. 2	(250 x 25) mm^2 Type 2 straight-sided specimen, test strain rate of 5 mm/min
2.	ASTM D3039 [11]; untabbed	Fig. 2	(250 x 25) mm^2 Straight sided specimen, test strain rate of 2 mm/min
3.	ASTM D3039; Aluminium end tab	Fig. 2	Aluminium end tabs (50 x 25 x 1.5) mm were bonded to the test specimens with epoxy adhesives
4.	ASTM D3039; end tabbed with emery cloth	Fig. 4	Specimens were gripped in the test machine with a layer of emery cloth between the surface of the specimen and the wedge grips
5.	ASTM D3039; GFRP end tab	Fig. 7	4 layers of hand-laid 0/90 glass fibre-epoxy reinforced composite end tabs were bonded to the test specimens; the GFRP tab was bevel-edged
6.	ASTM D3039; CFRP end tab	Fig. 7	rCFRP end tabs were cut from the material being tested and bonded to the ends of the specimen with epoxy adhesive
7.	ASTM D638 [12]	Fig. 2	Dogbone-shaped specimens were manufactured to the type-I geometry in ASTM D638

ISO 527-5 and ASTM D3039 are recognised Standards for the study of the tensile behaviour of unidirectional, continuous fibre reinforced composites with straight-sided specimens and ASTM

D3039 further states that it is applicable for discontinuous fibre reinforced composites. MIL-HDBK-17-1F Vol.1 states that tensile specimens with dogbone geometry are generally accepted for characterising low-modulus, unreinforced materials and low-reinforcement volume materials incorporating randomly oriented fibres [13]. ASTM D638, initially developed for testing unreinforced plastics, does not adequately cover testing parameters for advanced composites due to its intended scope, but it is generally used to characterise composites by researchers and are sometimes used in industry [14-16]. ISO 527-4 [17] mentions dogbone specimens may be used for non-unidirectional reinforced thermoset composites if they break within the gauge length. Therefore there is a need to establish the requisite tensile testing practice for discontinuous, non-woven carbon fibre composite presented here.





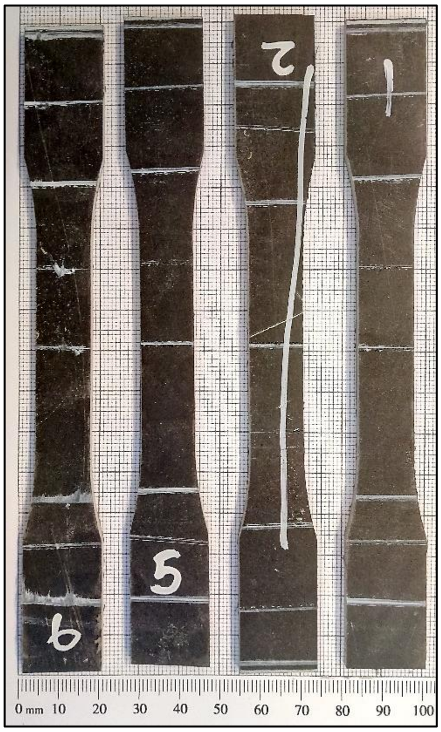


Figure 2 - Tensile Test specimen: untabbed (top left); aluminium end tabs (bottom left) as per ASTM D3039 specifications; dogbone geometry as per ASTM D638 (right)

The straight-sided specimens were prepared on a milling machine with a diamond powder-coated disc; the dogbone geometry was created with a three fluted end mill bit tool made out of tungsten carbide in conjunction with a mild steel dogbone template manufactured in house. These specimen fabrication techniques ensured a good quality of prepared specimens without any delaminations and good surface finish. The tensile tests were carried out on a calibrated Instron 5582 with 100kN load cell at ambient room conditions with the strain measurements taken by a knife-edge extensometer of 25 mm gauge length. Wedge grips with serrations, which are recommended for carbon fibre-epoxy composites, were used. Stringent material qualification requires analysis techniques that consider the statistics of the data that can relate to the design. Data analysis was carried out with IBM SPSS Statistics 25 software [18].

2.1.2 Interlaminar Shear Tests and specimens:

	Test Method	Figure	Description
1.	Short beam Shear (SBS) as per ISO 14130 [19]	Fig. 3	Radius of the loading nose: 5 mm Radii of the supporting sections: 2 mm Span to thickness ratio: 5
2.	SBS as per ASTM D2344 [20]	Fig. 3	Radius of the loading nose: 3 mm Radii of the supporting sections: 1.5 mm Span to thickness ratio: 4
3.	SBS with notches [21]	Fig. 8	ASTM D2344 short beam specimen modified as an I beam by introducing notches on the edges of the specimen

4.	SBS with Sandwich beam [22]	Fig. 8	ASTM D2344 specimen with epoxy-bonded aluminium end-tabs
5.	SBS with thick specimen geometry [23]	Fig. 4	Three x 6 mm thick laminate bonded onto each other with epoxy adhesive resulting in a laminate of over 50 plies of mats
6.	Iosipescu Shear Test as per ASTM D5379 [24]	-	Iosipescu specimen manufactured by bonding laminates and cut into required geometry by waterjet cutting

Interlaminar shear strength is a mechanical characteristic that gives an indication of the fibre to matrix adhesion. The objective of short beam shear test methods is to force the specimen to fail by shear in a three point flexure test by constraining the span to thickness ratio to a small value. The literature maintains that three point-loaded specimens are not acceptable if the specimens do not exhibit shear failure modes and this method is only useful for a quick quality test of the material [9]. The Iosipescu test by ASTM D5379 remains the acceptable out-of-plane shear test. The SBS tests were carried out on a Testometric M500-50CT Universal Tester at room temperature conditions and the strain rate was set at 1 mm/min.

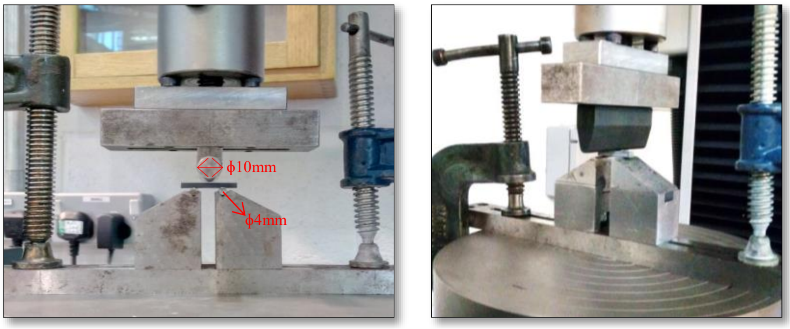


Figure 3 - Short Beam Strength set-up as per ISO 14130 (left) and ASTM D2344 (right)

Figure 4 - Thick beam for short beam strength testing

2.2 Long term Mechanical Testing

2.1.1 Fatigue Testing

The general absence of fatigue test guidelines regarding the geometries of the specimens, test recommendations and specifications in literature, results in a need to develop a specific fatigue test protocol for the material system in use. Fatigue design properties can be generated depending on the structural application of the laminates. Since most fatigue-driven structures are tension-critical [25], the fatigue experimentation in this research was carried out in tension-tension fatigue with dogbone geometry specimens at 10 Hz and a sinusoidal stress cycle ratio of 0.1. Fatigue tests at the coupon level are generally carried out to establish criteria such as:

i. Confirmation that the laminate can withstand high fatigue cycles at lower loads
ii. Determination of an “endurance limit” for the laminate, if any
iii. Establishment of a characteristic S-N curve for the material system.

Criterion (a) was carried out by testing the specimen at a maximum alternating stress corresponding to 10% of its mean ultimate tensile strength for 6 to 8 million cycles on Mayes Fatigue Machine with a 10kN load cell. Criteria (b) and (c) were established with low cycle fatigue by progressively running tests at higher loads until specimen failure, or until 10^7 cycles was reached.

2.1.2 Exposure to fluids

ASTM D5229 [26] provides the procedures for moisture absorption and for calculation of a moisture diffusivity constant. However, standard test procedures to determine the influence of exposure to various environmental conditions on the mechanical properties of composites is limited to testing the specimens using the short beam shear strength method. The disadvantages of this method have been discussed in the previous section. The influence of selected environmental effects on the properties of the laminate was studied using dogbone-shaped specimens subjected to immersion in tap water at 4°C, 23°C and 50°C; the change in tensile properties was monitored for 60 days. Future work will involve exposure to more aggressive fluids such as deionised water and fluids which the laminate could possibly be exposed to in structural applications such as diesel, de-icing fluids and greases such as soaps of lithium/sodium.

2.3 Physical Characterisation

The glass transition temperature and the specific heat of the material were measured with Digital Scanning Calorimetry as per ASTM E1356-08 [27]. This test method involves continuously monitoring the difference in heat flow into a sample of the composite material and a reference material such as indium from a temperature range of 30°C to 200°C at a heating rate of 10°C/min.

The coefficient of thermal expansion (CTE) was calculated with the use of dual electrical resistance strain gauges, EA-06-125PC-350, with one strain gauge bonded to the composite material and the other to a reference material with low CTE value such as INVAR 36 with M-bond 200 adhesive supplied by Vishay Measurements Group UK. The strain gauges were wired to a P3500 strain indicator instrument and the composite and INVAR test pieces were subjected to a heating cycle between 25°C and 60°C in increasing steps of 5°C with a closed oven. The specimens were left at the step temperature for at least 30 minutes. A thermocouple was taped onto the composite material for precise temperature measurements. CTE was calculated as the ratio of difference in thermal outputs from the test pieces and the temperature range [28].

3. RESULTS

3.1 Tensile Testing

Unidirectional composites are generally characterised with straight-sided geometry and are supported with end tabs to transfer the load from the grips to the specimen. This concept had been applied with multi-layered laminates and woven laminates with good results, and it has been recognized as a standard for testing composites in the same way that dogbone geometries are accepted as the standard geometry for testing metals and polymers.

Metals are generally isotropic in nature while composites are inherently anisotropic. Thus, a continuous unidirectional composite specimen when tested with a dogbone geometry would result in fibre splitting in the specimen shoulder. The material characterised here however, exhibited 20% anisotropy in the 0 and 90 directions owing to the nature of the ELG carding machine to orient more fibres in the 90 direction of the mat. Selection of specimen geometry would not be of particular concern if the tensile failure modes of straight-sided composites were acceptable.

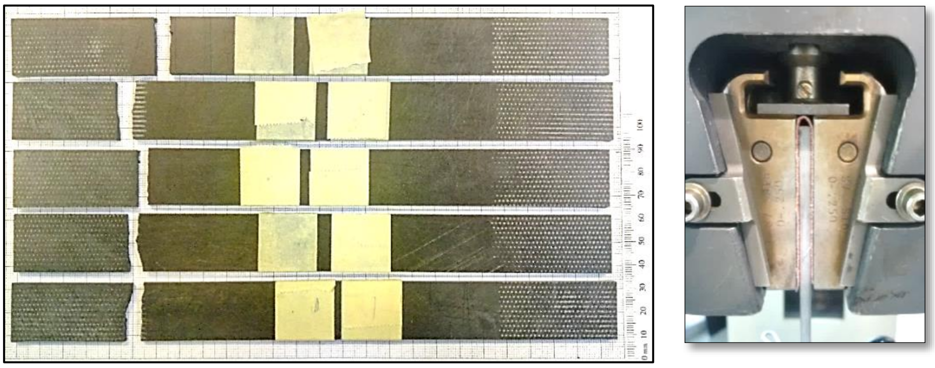


Figure 5 - Failure of straight-sided specimens near the grips (left) and use of emery cloth for tabbing in tensile wedge grips (right)

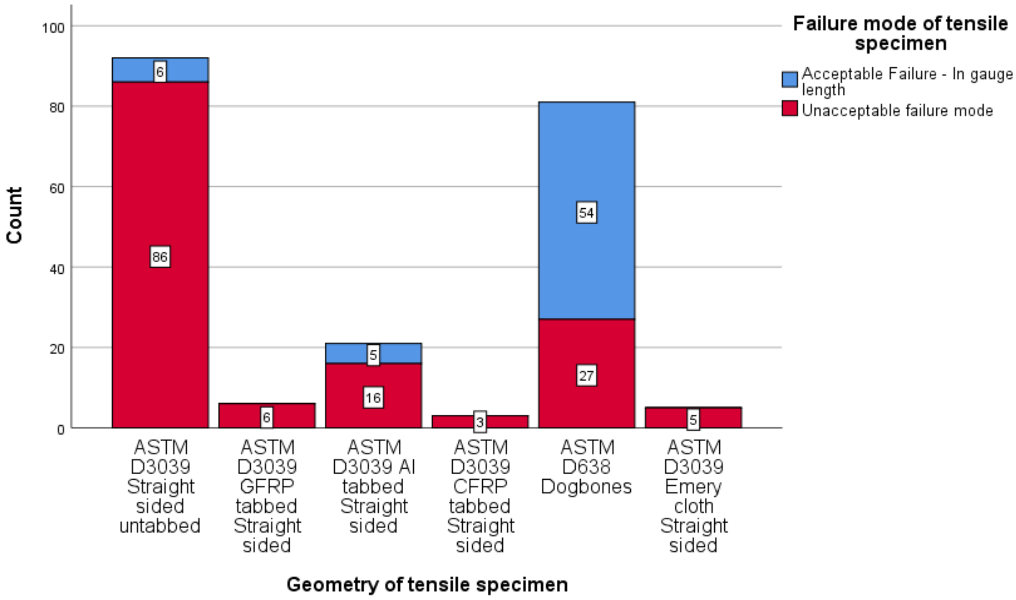


Figure 6 - Proportion of acceptable failures based on the geometry of tensile test specimen, graph obtained from IBM SPSS v25[18]

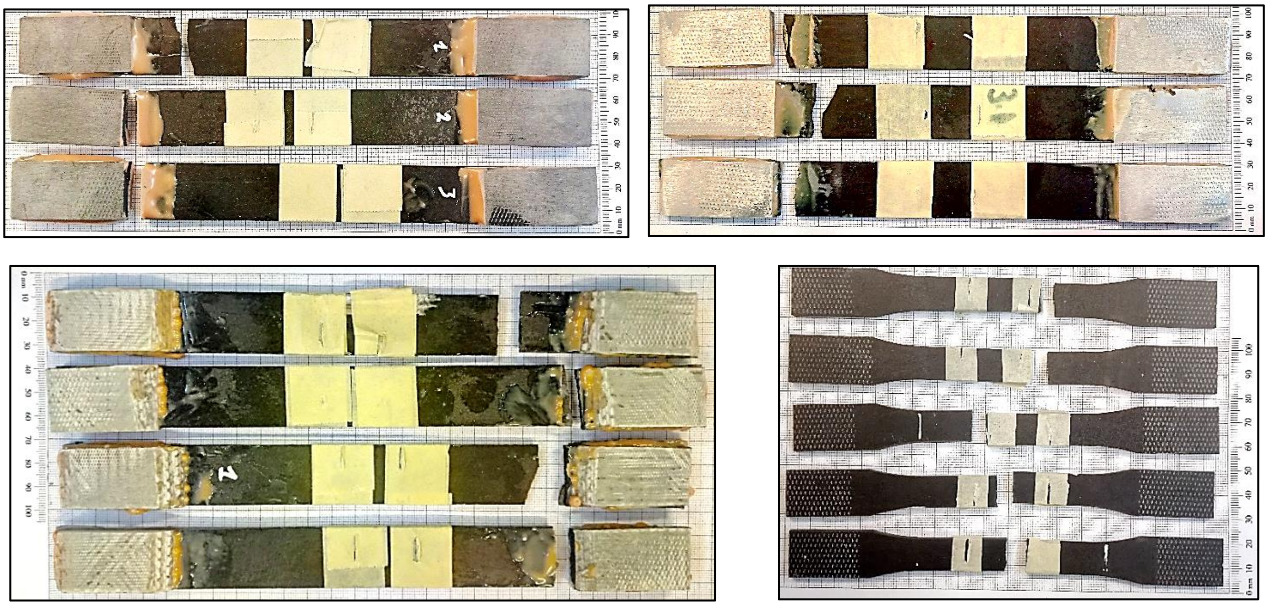


Figure 7 - Unacceptable failures in straight sided specimens with CFRP (top left), aluminium (top right) and GFRP (bottom left) end tabs and relatively successful gauge length failures with the use of dogbone specimens (bottom right)

Composite standards deem failures at the grips to be unacceptable because they are not representative of the strength of the material, and results are usually less than the actual tensile strength. The discontinuous, randomly oriented composite used here consistently failed at the grips with straight-sided geometries and the use of end tabs did not show any significant improvement. The adaptation of a dogbone geometry, traditionally used for characterisation of metals and polymers, was found to be fairly successful for discontinuous non-woven laminates. The tensile specimens failed within the gauge length more often and showed brittle failure with planar failure surfaces. This geometry allows the stress to be distributed evenly from the grips to the gauge length of the specimen. The improved distribution of stress across the dogbone specimens also resulted in increased tensile strength.

3.2 Shear Testing

The short beam shear test methods, and the various adaptations of the test, result in tensile failure on the bottom plane of the beam which is recognized as an unacceptable failure mode. The specimens do not show any signs of shear failure, despite manipulations to the span-to-thickness ratio, introduction of stress into the beam, concentration of stress through notches and increasing the overall thickness of the beam. This restricts the test method to be merely a quality check and the resulting data cannot be used as a design allowable. The alternative, which the literature mentions provides acceptable results, is that of Iosipescu shear testing with a V-notched specimen geometry.

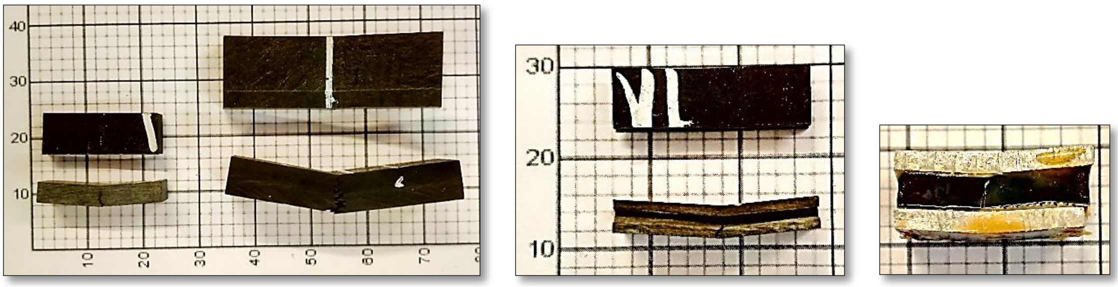


Figure 8 – Tensile failures of SBS specimens regardless of the specimen thickness or alternate geometries

3.3 Fatigue Tests

The laminates, when tested at a maximum alternating stress of 10% of the mean UTS, showed no visible signs of fatigue damage, including intralaminar cracks, which is generally noticeable by the naked eye. The specimens were then subjected to a tensile load and the fatigue strength of the material showed no significant variation from the mean UTS. The strain to failure of the specimens exhibited a 30% decrease, increasing the stiffness of the material. Further fatigue tests to determine an endurance limit, and to ultimately obtain a characteristic S-N curve, is in progress.

3.4 Physical Characterisation

The glass transition temperature and the specific heat capacity of the sample was calculated to be 116.2°C and 1.042 J/g°C respectively. The CTE of the material was determined to be 12.27 µstrain/°C in the 0° direction and 5.34 µstrain/°C in the 90° direction. This test method is reliable because the quantity measured is with respect to a material of known CTE. The CTE of a plain woven carbon fibre/epoxy composite was calculated to be 5.68 µstrain/°C and 8.33 µstrain/°C in the 0° and 90° directions respectively [29].

3.5 Fluid Exposure

The laminates have a moisture diffusivity constant of 1.58E-08 cm^2/s, realising the equilibrium moisture content at 1.75% at 75°C and 95% R.H. in an environmental chamber. The diffusivity constant for a T300 CF/1031 epoxy unidirectional composite at 80% RH and 75°C is 1.32E-08 cm^2/s and our results fall within the same range [30]. Exposure to fluid environments is of interest because of the plausible structural applications of the composite system. The specimens immersed in tap water at 4°C, 23°C and 50°C for a time period of 60 days showed a 10% decrease from the mean tensile strength. The strength remained fairly constant after this initial decrease.

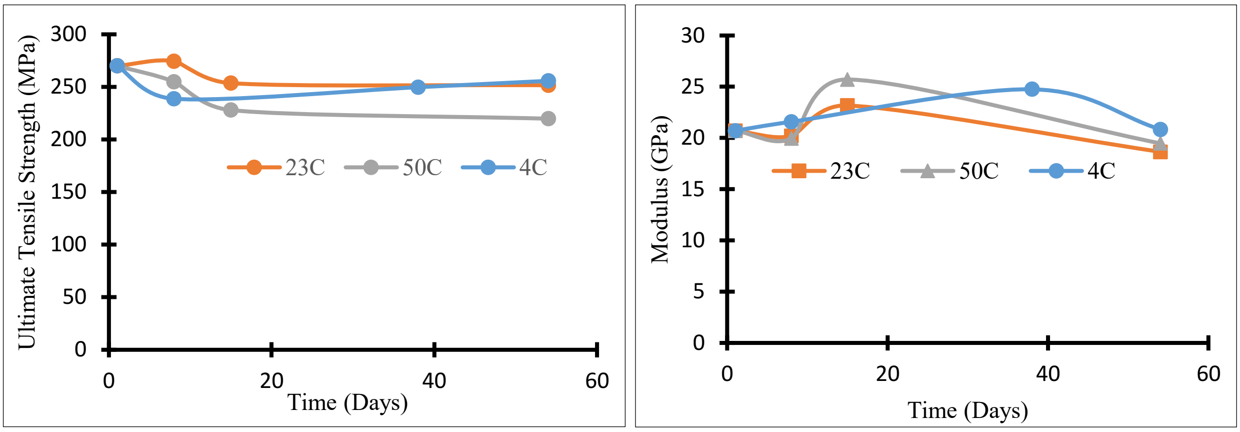


Figure 9 - 10% reduction in tensile strength due to tap water immersion (left); Influence of tap water immersion on tensile modulus (right)

4. CONCLUSIONS

i. A dogbone geometry is preferred for tensile testing of discontinuous non-woven composites, over the straight sided specimens recommended by the standards, because the material performance is more representative of the composite material system. The variability in results amongst the same specimens was lower with the dogbone geometry.

ii. The 3-point flexure test with a short beam did not produce shear failures in the composite material and can be used for qualitative analysis only. Alternate procedures such as the Iosipescu test is required for determining out of plane shear properties.

iii. High cycle fatigue at low loads did not have any significant influence on the fatigue strength of the non-woven composite material.

iv. Immersion in tap water at 4°C, 23°C and 50°C for 60 days reduced the tensile strength of the laminate by 10%.

Future Work:

i. Use of the Iosipescu test to determine in plane and out of plane shear.

ii. Establish the influence of selected fluid environments on the composite system

iii. Determine the fatigue endurance limit and possible use of NDT to study fatigue damage.

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