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Experimental Study on the Mechanical Properties of Glass Fibre Reinforced Epoxy at Elevated Temperature

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ABSTRACT

This paper presents the effects of elevated temperature on the mechanical response of a glass fibre reinforced epoxy (GFRE) composite. The mechanical properties taken into account are tensile, compression and shear. All tests are carried out at temperatures of 30°C, 70°C and 110°C, below the glass transition temperature of the resin. The properties along fibre direction and perpendicular to fibre direction are investigated, with two sets consisting of 0° and 90° fibre direction for tensile and compression tests. Stress-strain profiles at each temperature are firstly compared. Subsequently, the elastic modulus and the ultimate strength with respect to temperature are assessed. The results indicate that tensile properties remain relatively unaffected at 70°C but drop rapidly at 110°C. In addition, compressive properties decrease steadily from 30°C to 110°C, while shear properties are heavily degraded with increasing temperature. Fibre dominated properties have better heat resistance compared to matrix dominated properties due to matrix softening and weakening.

Keywords: Temperature; mechanical properties, glass fibre; composite.

INTRODUCTION

Composites have widely developed by researchers and industries alike throughout the last couple of decades. With the addition of fibre reinforcement combined with polymer matrix that formed fibre reinforced polymer (FRP), the properties of composites are greatly improved. FRP has brought a lot of benefits and possibilities into the picture, most notably the superior strength-to-weight ratio, stiffness and ease of manufacturing compared to conventional materials [1-3]. FRP has found its applications in construction, aeronautical, aerospace, oil and gas, marine, automotive and military fields. Fire safety is deemed as a very important aspect of modern transportation structures. Structural materials are required to exhibit sufficient fire resistance for maintaining structural integrity. These applications present many conditions that affect the FRP performance one of which is elevated temperature, where FRP is used at high-temperature environments [4, 5].

Resins used to form composites have specific glass transition temperature (T_g) where the matrix changes the physical behaviour from glassy to rubbery state and decomposition due to chemical reaction. Beyond T_g, the mechanical properties of FRP may be severely diminished and the structure was compromised for use [4, 6, 7]. Both fibre reinforcement and matrix take an important role in the temperature resistance of the composite. The thermo-mechanical behaviour of composite structures investigated analytically by Dimitrienko [4] indicated temperature and heating time are the main parameters that affect elastic moduli and strength of matrix while the only temperature affects the properties of fibres. On the other hand, the fibre volume fraction has no effect on the T_g of the composite [8].

Experimental and analytical methods were carried out by Correia et al. [1] on the tensile, compression and shear properties of glass fibre reinforced epoxy (GFRE) pultruded profiles at temperatures ranging from 20°C to 250°C. Their range of testing temperatures included the glass transition temperature region (136°C to 162°C) and also the decomposition region. For tensile tests, the stiffness of GFRE remained relatively unaffected by temperature even approaching a maximum of 250°C, while the tensile strength was reduced by the increase of temperature in a steady linear pattern. However, for compression and shear tests, temperature heavily affected the stiffness and strength of the GFRE. Even before T_g, the compressive and shear strength lose about half of the original values at ambient temperature, as there was a steep decrease prior to T_g for both cases. A more recent study by Hawileh et al. [9] compared the mechanical properties of different fibre reinforcement; glass (GFRP), carbon (CFRP) and hybrid of both (CG) at a temperature range of 25°C to 300°C. Their study also included the T_g and decomposition. It was concluded that GFRP specimens had retained the highest tensile strength compared to CFRP and CG, while CG maintained the highest elastic modulus, 91% compared to others. Aklilu et. al [10] studied on similar materials; GFRP, CFRP and hybrid at temperatures from 25°C to 140°C but on the interlaminar shear strength and stiffness. Their results indicate interlaminar shear strength is enhanced with a hybrid of carbon and glass fibres. The in-plane shear strength of GFRP was very recently analyzed by Rosa et al. [11] where they found significant degradation with respect to temperature. Ou and Zhu [12] also tested on GFRP properties, with different strain rates and temperatures ranging from -25°C to 100°C. They found a linear decreasing trend of modulus with respect to temperature and a bilinear decrease of strength where the tensile strength decreased faster at higher temperature zone.

A new type of basalt type (BFRP) composites was investigated on their thermo-mechanical properties compared to GFRP by Lu et al. [13], from room temperature to 200°C. Their results indicated BFRP showed much better-elevated temperature resistance than GFRP, but the T_g for BFRP is 166.9°C while for GFRP is 121.1°C. The mechanical properties of composite and metal hybrid was also investigated by Hu et al. [14] where a Polyetheretherketone-based polymer with high T_g was tested at room temperature, 120°C, 170°C and 220°C. The effect of loading rate on thermo-mechanical properties was also investigated by Kumar et al. [8] across temperatures of 25°C to 110°C. Effectiveness of fire-resistant paint on reducing the tensile degradation of GFRP and CFRP sheets were explored by Jarrah et al. [15]. There have been various analytical and mathematical techniques used to predict and examine mechanical of composites across a range of temperature [5-7, 16, 17]. From literature, it is apparent that there are limited recent elevated temperature studies on the fundamental properties; both fibre and matrix dominated tensile and compression properties, and in-plane shear properties of S2 grade GFRE which is intended for aeronautical and aerospace applications. Different orientation of fibre as in fibre dominated or matrix dominated properties could exhibit different behaviour under a range of temperatures.

The aim of this paper is to demonstrate the influence of elevated temperature towards the mechanical properties of unidirectional GFRE. Glass fibres are suitable to be used at a higher temperature because of their relatively high melting and softening point. Tensile, compression and shear properties in both fibre and matrix dominant directions are tested on standard coupon specimens based on ASTM that are exposed to stable-state temperatures of 30°C, 70°C and 110°C. The stress-strain curve, modulus, ultimate strength and failure modes are captured with the use of strain gages and subsequently analyzed. Then, the relationship between each property and temperature are plotted to capture the mechanical properties across the range of temperature. The results are then compared to characterize GFRE tensile, compressive and shear properties.

EXPERIMENTAL SETUP

The type of GFRE used in this study is unidirectional S-2 type glass fibre cured from individual prepregs of 0.5 fibre volume fraction with a single-ply thickness of 0.15mm. The prepregs are made of epoxy that have a glass transition temperature of 125°C and is supplied by X Plas Singapore. The tensile test, compression test and shear test were prepared with accordance to ASTM [18-20]. In order to acquire the properties of the GFRE in both fibre dominant and matrix dominant directions, two different groups of specimens were fabricated for both tensile and compression tests, 0° fibre direction for fibre dominant and 90° fibre direction for matrix dominant. Shear tests were conducted using one group of ±45° specimens tested through a tensile test to determine the in-plane shear response and properties. Therefore, the fibreglass prepreg layup is cross plied into ±45° relative to the loading axis. The details about the dimensions and set up for each type of test are shown in Table 1. All parts are formed into the desired shape according to each test requirements using a hot press machine, followed by regulating the prepreg binding at a stable condition of 120°C.

Table 1. Designation corresponding to each test and respective details [18-20].

	Type of test	ASTM	Fibre angle	Length, *l* (mm)	Width, *w* (mm)	Thickness, *t* (mm)	No. of plies	Rate (mm/ min)
T-0	Tensile	D3039	0°	250	15	~1.2	8	2
T-90			90°	175	25	~2.1	14	2
C-0	Compress-ion	D6641	0°	140	13	~2.7	18	1.3
C-90			90°	140	13	~2.7	18	1.3
S	Shear	D3518	±45°	250	25	~3	20	2

GFRE is relatively brittle material that might crack when gripped by testing machine, thus, to protect the specimens around the machine grips, woven type end tabs with a thickness of 1.5mm are used for all the specimens. The ends of the specimens are sanded using fine type sandpaper, followed by cleaning and dust removal with acetone. Right after, the end tabs are bonded using Araldite AW 4859 with curing cycle of 24 hours at room temperature, followed by 2 hours post-curing at 150°C. Strain gages were used on T-0, T-90 and S specimens illustrated in Figure 1 to record the strain values during the test. Kyowa strain gages and strain gauge adhesive type CN, suitable for use up to 110°C are used. The compression loading in ASTM D6641 is achieved using a combined loading compression test fixture, shown in Figure 2 where the specimen is clamped between two top and two bottom blocks to ensure uniform compression.

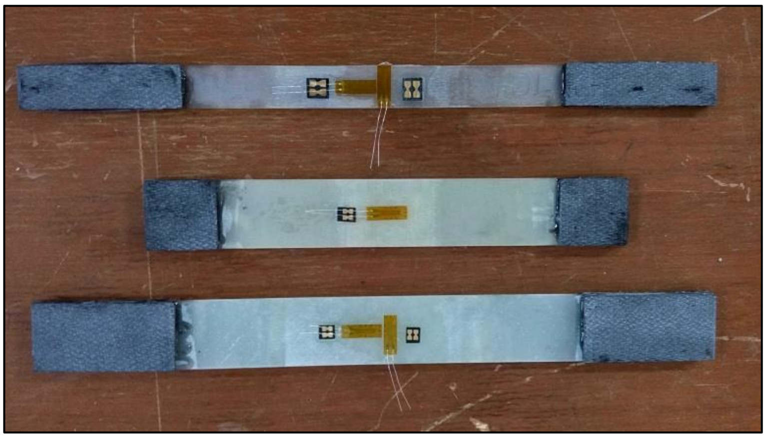


Figure 1. Specimens with tabs (in black) and strain gauges installed (from top to bottom: tensile 0°, tensile 90° and shear).

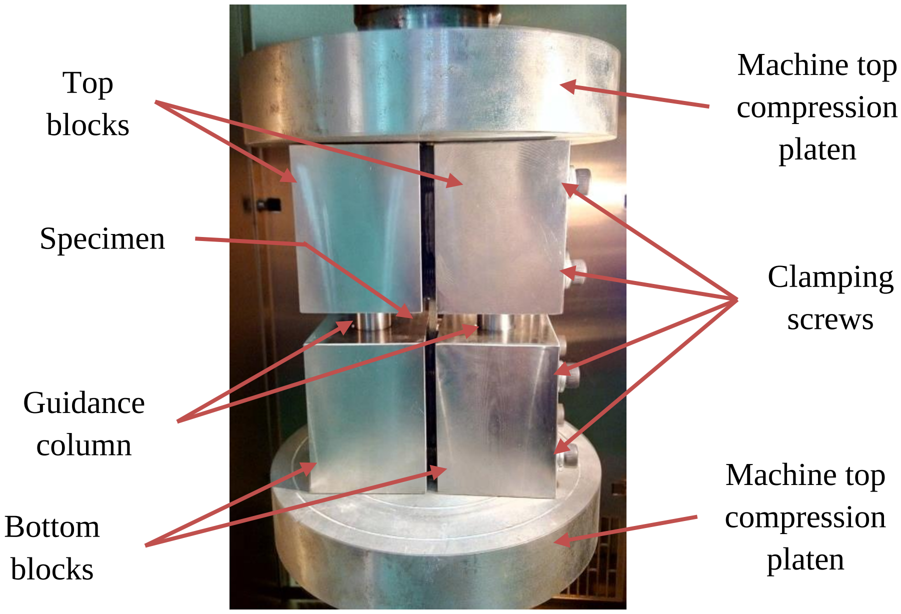


Figure 2. Compression jig setup with the specimen in between.

Quasi-static tests were carried out using universal testing machine Shimadzu AG-X plus with a maximum capacity of 100kN, and portable data logger was used in cases that use strain gauge. Constant steady-state temperature for the tests was done using thermostatic chamber TCE-N300 at 3 temperature settings that were lower than the glass transition temperature of the resin; 30°C, 70°C and 110°C were used for every test. For a controlled environment and eliminating the effects of thermal ageing, the chamber is first heated up to achieve the desired temperature, then only the specimen is set up within the chamber, followed by a brief of 5 minutes for the specimen and chamber environment to reach the desired temperature, and finally the testing machine is engaged to start the test with continuous heating throughout the test. An average minimum of three specimens is used for each temperature setting. From the tests, stress values are calculated from the machine while strain values are collected from the data logger. Both values are then compiled to plot the stress-strain profile. From there, the modulus of elasticity, ultimate strength and strain are obtained and compared across the temperature range. It is worth note that while the stress values in tensile and compression tests are calculated by the usual load per unit area, the shear stresses are determined from load per two units of area based on the ASTM [20] as shown in Eq. (1).

$$\tau_{12}=\frac{P}{2A} \tag{1}$$

where τ_{12i} is the shear stress, MPa; P is the load and A is the cross-sectional area. Shear strain is acquired from Eq. (2).

$$\gamma_{12}=\varepsilon_x-\varepsilon_y \tag{2}$$

where γ_{12} is the shear strain, ε_x is the longitudinal strain and ε_y is the lateral strain. Moreover, since ultimate failure typically does not occur within 5 % shear strain, the maximum shear strain is taken at 5 %. Shear modulus is then calculated from the difference in stress versus the difference in strain taken from around 2000 to 6000 $\mu\varepsilon$.

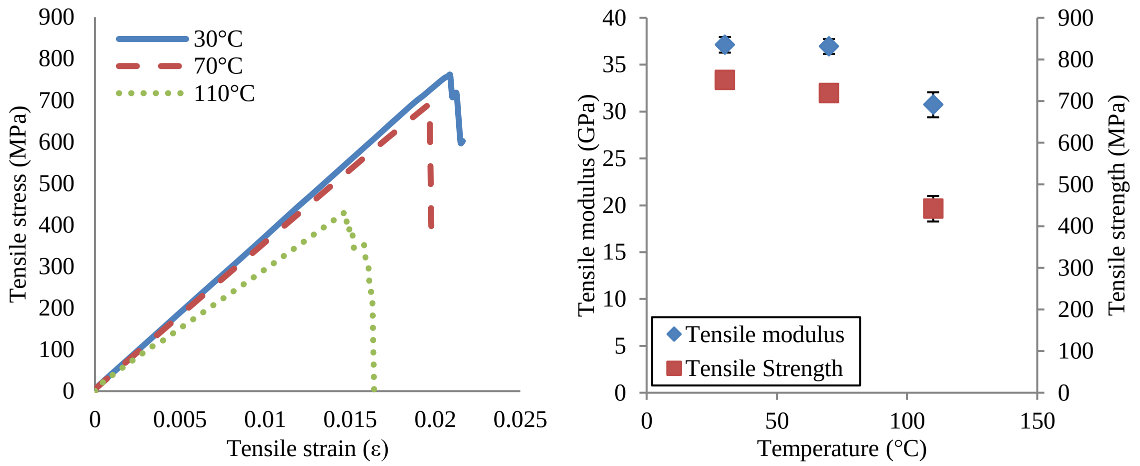
RESULTS AND DISCUSSION

Tensile Test

Figure 3(a) shows the comparison between each representative stress-strain curve at a temperature of 30°C, 70°C and 110°C for tensile specimens along fibre dominant direction. The degradation of the profile is generally on par with the stress-strain curve by Aydin [3] and Hawileh et al. [9], with little to no effect on the peak and stiffness at lower temperatures, but drops increasingly at temperatures above 100°C. Generally, the tensile stress-strain follows a linear curve followed by a sudden drop to zero, indicating brittle breakage. The stress-strain profile is affected by elevated temperature with a progressive reduction of the profile. It is worth noticing that for 70°C, the gradient and peak value is only slightly lowered when compared to 30°C, whereas it is much lower for 110°C.

This can be further illustrated when comparing elastic modulus and tensile strength in Figure 3(b). From 30°C to 70°C, the tensile modulus is only reduced by a meagre 0.48% while the strength decreased by 4.15%. However, the modulus and strength decreased by 17.23% and 41.15% relative to 30°C for 110°C. Both modulus and strength degradation follows a similar trend. This indicates that along fibre dominant direction, the tensile properties are mostly maintained for middle-temperature range, but the properties start to decline at higher temperature ranges when the fibres and matrix become weaker and softer, as described by Ou et al. [21]. Results are generally similar to the findings of Correia et al. [1], with a slight reduction of stiffness and a marginal reduction in strength.

The general failure mode of the sample after the tensile test can be observed in Figure 4, where the specimens did not fail across the transverse section but instead ruptured longitudinally along the axis. Observation of post-test samples for 30, 70 and 110°C show very similar failure, hence only one sample is displayed. Instead of the composite fracturing locally through fibre failure, the specimen fractured prematurely due to fibre/matrix debonding. The studies by Hawileh et al. [9], Correia et al. [1] and Ou and Zhu [12] showed a similar type of failure. Hence, the longitudinal tensile strength obtained herein is lower than the actual desired ultimate strength due to fibre failure.



(a) (b)

Figure 3. (a) Representative stress vs. strain curve comparison at different temperatures and (b) tensile modulus and tensile strength (with standard deviation) vs. temperature for T-0 specimens.

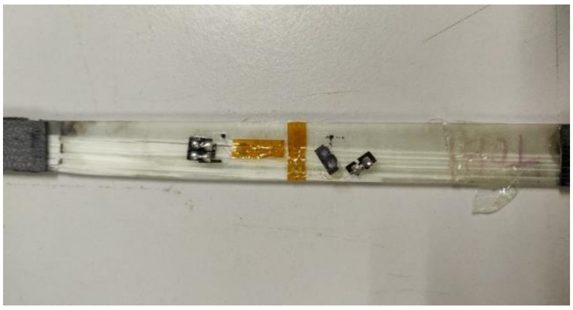


Figure 4. Failure mode of T-0 specimen.

Tensile test results for T-90 specimens, loaded in matrix dominant direction are shown in Figure 5. Similar to T-0 specimens, the decrease of gradient and drop of peak stress at higher temperature zone (110°C) is much significant compared to medium temperature zone (70°C). However, in this case, the effect of temperature on the modulus and strength is at a larger scale. The stress loaded in matrix dominant direction increases in a non-linear profile of decreasing gradient. The ductility of matrix increases as temperature increases, as shown by the increase in maximum strain.

Based on Figure 5(b), there is a 21.54% drop in tensile modulus and a 9.45% drop of tensile strength at 70°C, followed by 72.82% modulus reduction and 64.40% strength reduction. These signify the weaker resistance of matrix dominated loading, where the epoxy becomes softer and loses its strength more easily when introduced to heat compared to the fibres [15]. The comparison between specimens at the three temperatures after fracture can be seen in Figure 6. The straight transverse breaking of the specimen confirms that the failure is governed by matrix brittle fracture at all three temperatures. The nature of brittle failure explains why there is no evident drop of stress at the failure point in Figure 5(a). The specimens for 30°C failed but did not separate into two, as a part of the matrix is still connected. However, at 70 and 110°C, the specimens separated into two, due to the much weaker matrix.

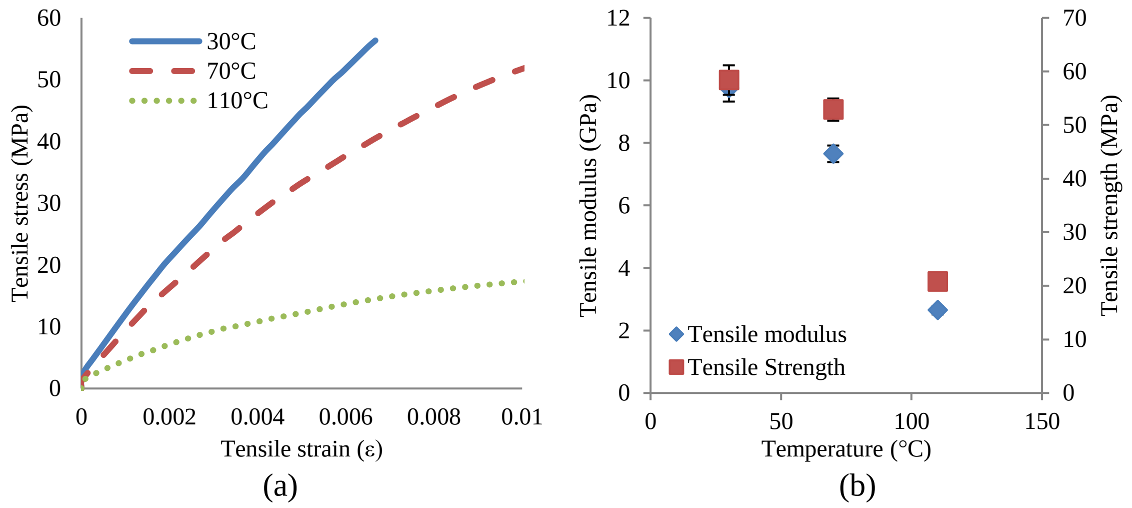


Figure 5. (a) Representative stress vs. strain curve comparison at different temperatures and (b) tensile modulus and tensile strength (with standard deviation) vs. temperature for T-90 specimens.

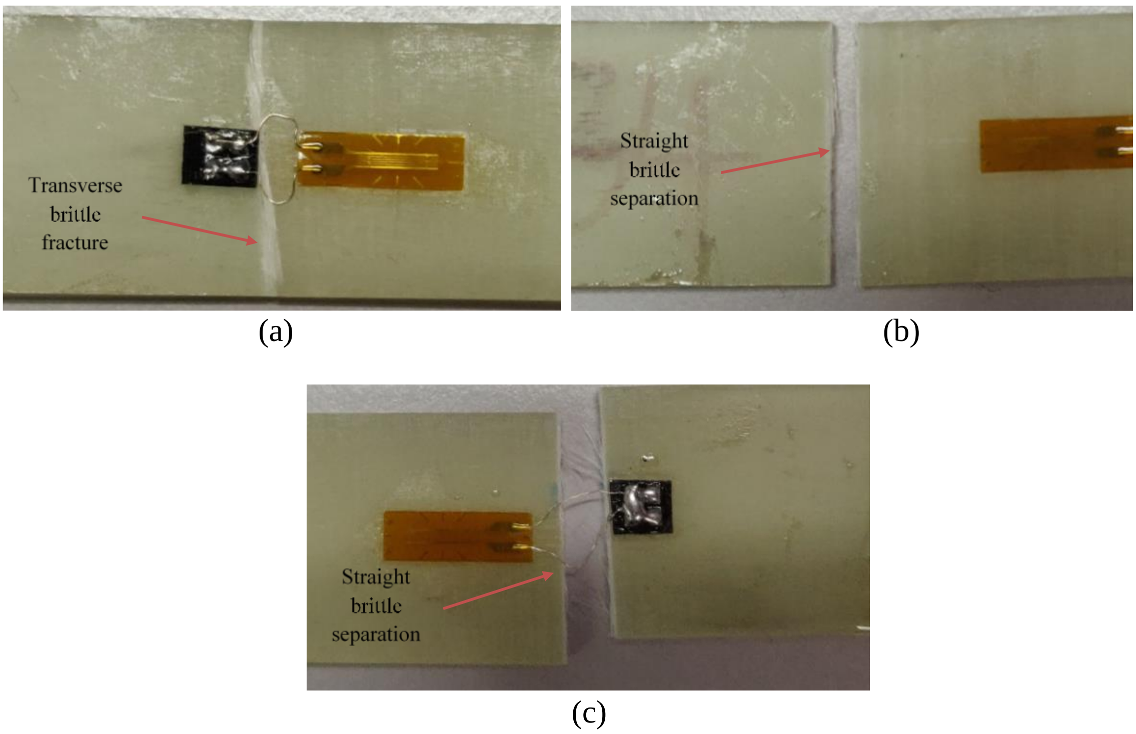


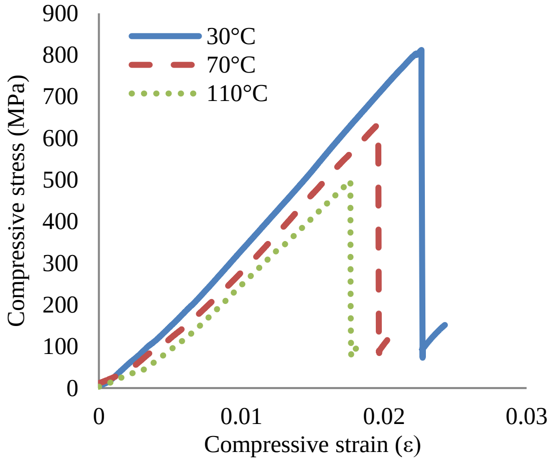
Figure 6. Failure mode of T-90 specimens tested at (a) 30°C, (b) 70°C and (c) 110°C.

Compression Test

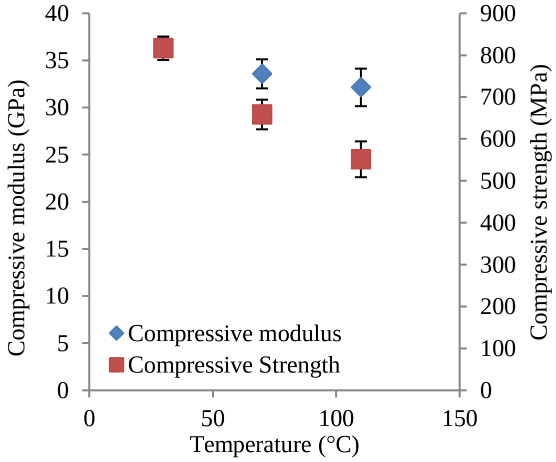
Figure 7(a) presents stress-strain curves while Figure 7(b) presents the compressive modulus and strength as a function of the temperature of compression tests for 0° fibre orientated specimens. It can be seen that the compressive stress is directly linear when plotted against strain values. Stress abruptly drops right after the peak stress value, which is once again attributed to the brittle nature of fibres.

For the cases of compression, the material properties degrade in a steady rate across all the temperatures, where the compressive modulus decreases by 8.08% at 70°C, and 12.03% at 110°C when compared with 30°C base temperature. On the other hand, compressive strength at 70°C and 110°C are 19.36% lower and 32.45% lower respectively. This behaviour shows that compression properties are less affected at the highest temperature zone and decreases linearly across the designated temperatures. Results for compression tests differ greatly from the findings of Correia et al. [1], where their compressive strength decreased significantly even before T_g. This could be due to the different setup for the test.

Differences of failure modes between compression 0° specimens tested at 30, 70 and 110°C are demonstrated in Figure 8. From general comparison, it appears that the failure is more severe for specimens at 30°C with a large degree of swelling at the failure location with outwards buckling and splitting of matrix caused by delamination as the fibres have much higher strength and did not fail. This swelling phenomenon decreases as the temperature increases. Such changes in the failure mode were also reported by Opelt et al. [22]. As the temperature increases, the decrease in matrix stiffness reduced the splitting and buckling failure mode in which the failure becomes more shear dominated.



(a)



(b)

Figure 7. (a) Representative stress vs. strain curve comparison at different temperatures and (b) compressive modulus and compressive strength (with standard deviation) vs. temperature for C-0 specimens.

Compressive stress-strain profile shown in Figure 9(a) provides an understanding of ductile behaviour of the material matrix that is similar to matrix dominant tensile behaviour, where it follows an initial linear trend that decreases in gradient as the stress approaches peak. The degradation of the properties however, follows a steady pace that is compressive loading. The compressive modulus at 70°C and 110°C is reduced by 13.65% and 23.78% respectively; while the compressive strength is reduced by 27.90% and 43.78% respectively. The more temperature dependent C-90 matrix dominant specimens caused all the reduction percentage to be slightly higher than for the less temperature dependent fibre dominant specimens in C-0.

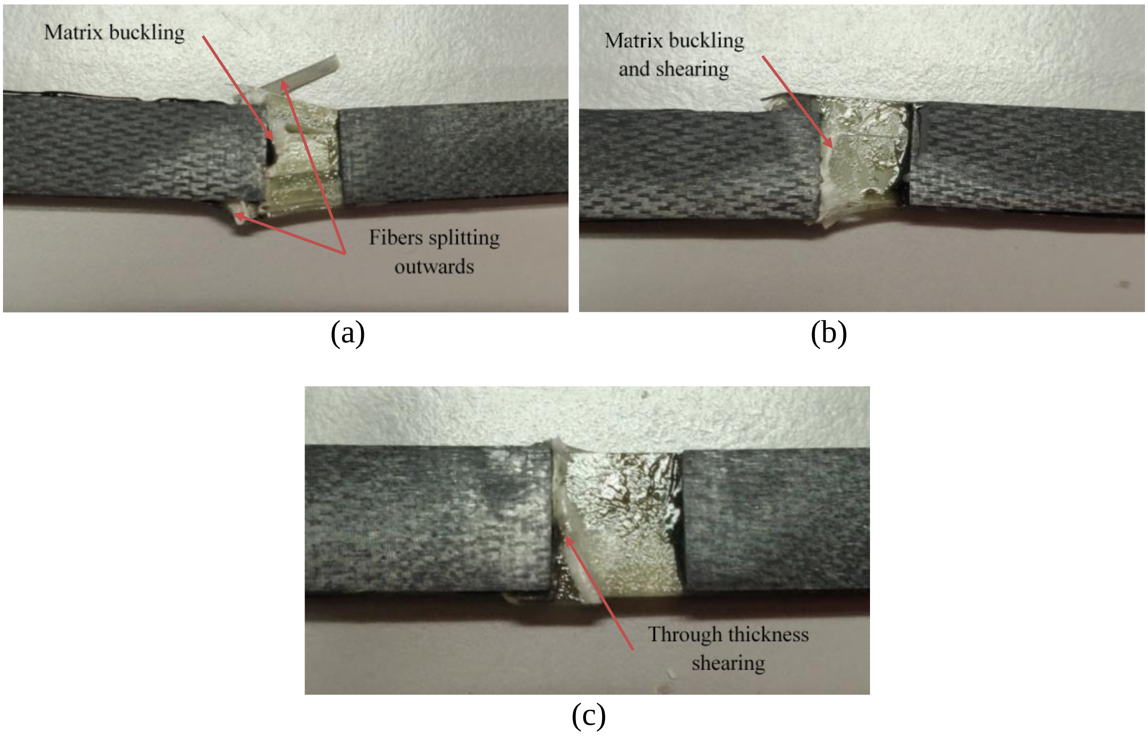


Figure 8. Failure mode of C-0 specimens tested at (a) 30°C, (b) 70°C and (c) 110°C.

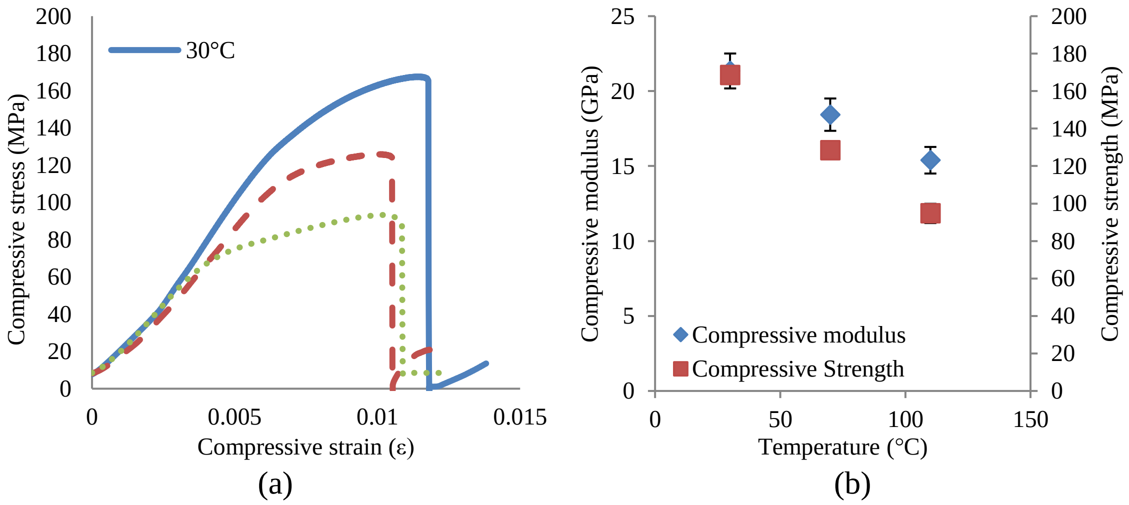


Figure 9. (a) Representative stress vs. strain curve comparison at different temperatures and (b) compressive modulus and compressive strength (with standard deviation) vs. temperature for C-90 specimens.

Figure 10 shows the typical failure mode of C-90 specimens across 30°C to 110°C, which are identical to each other. In this case, the failure is dominated by a matrix as the fibres are in the transverse direction. Hence, the failure is much more brittle and cut straight across the entire cross-section. The change from increasing to plateau in stress versus strain curve of Figure 9(a) is because the matrix cannot bear an additional increase of stress when it reaches the ultimate compressive stress, but the strain continues to increase in the form of through thickness-shear and sliding between the two broken sections. The failure mode did not change much with higher temperatures.

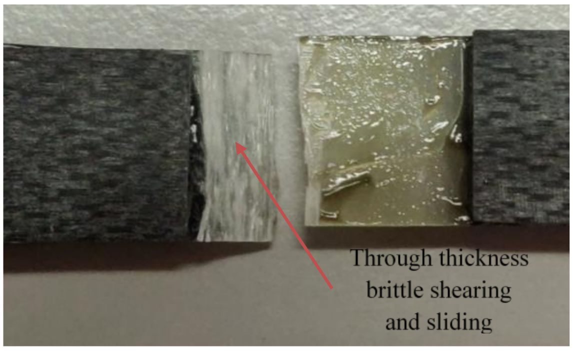


Figure 10. Failure mode of C-90 specimen.

Shear Test

Figure 11(a) illustrates the shear stress-strain curves of 30°C, 70°C and 110°C. The results show an immense deterioration of shear properties because of elevated temperature. The stress-strain profile demonstrates non-linear ductility similar to other matrix dominant tests.

Referring to Figure 11(b), the shear modulus loses 39.05% of its original value at 70°C and 97.49% when reaching 110°C. At the same time, the shear strength decreases by 44.62% and 93.11% at 70°C and 110°C respectively, where the specimen loses almost all the initial shear strength and shear stiffness compared to 30°C. However, it is worth noting that the decline of properties is at a constant degree with respect to temperature. This condition is very similar to the one reported by Rosa et al. [11] where the shear strength and modulus of their GFRP are also severely low (less than 30%) when nearing T_g.

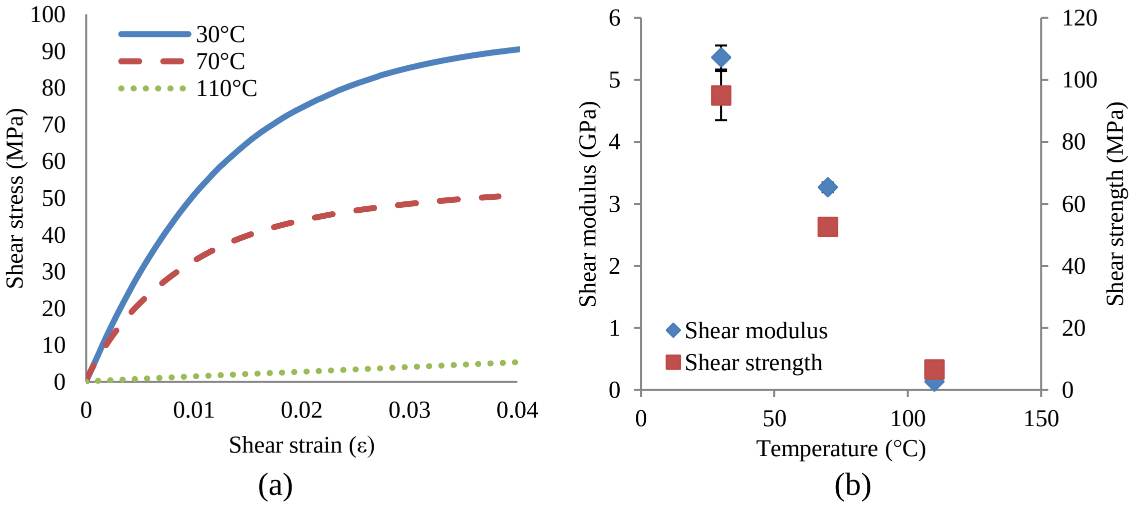


Figure 11. (a) Representative stress vs. strain curve comparison at different temperature and (b) compressive modulus and compressive strength (with standard deviation) vs. temperature for S specimens.

For in-plane shear tests, the specimen does not fail and therefore there is no failure mode since the samples are not in pure in-plane shear stress according to the ASTM [20]. The specimens can be stretched to a very high strain due to in-plane normal stresses, fibre scissoring and redistribution of load with a higher number of plies. When the temperature increases, the shear resistance between each ply matrix is substantially lost when the composite prepreg plies lose their adhesion with each other, causing a major drop of shear properties.

All in all, GFRE composite used in this study demonstrates its feasibility for applications at elevated temperature. Based on Table 2, unidirectional GFRE is most suitably used in compressive loading, secondly in tensile loading yet only at medium temperature range, and not recommended for applications involving shear loading. When comparing between tensile and compression tests, tensile properties are generally unaffected at 70°C, but greatly drops at 110°C when approaching glass transition temperature. Meanwhile, the compression type of loading is more stable even at the highest temperature range, whereas shear properties diminish greatly with temperature. Besides that, the orientation of fibres plays an important role in the tensile and compressive strength, where it is much stronger when loads are applied along fibre direction as opposed to perpendicularly. The fibre volume fraction in this study is 0.5, and it is possible to further improve the strength and stiffness by studying different fibre volume fraction on fibre and matrix dominated properties. It is also suggested to make improvements through coating with heat resistance or insulation material.

Table 2. Designation corresponding to each test and respective details.

Temperature (°C)	Tensile (MPa)		Compression (MPa)		Shear (MPa)
	0°	90°	0°	90°	-
30	750.67±12.78	58.40±2.75	816.53±27.99	168.51±2.88	94.93±7.93
70	719.48±13.32	52.88±2.08	658.42±35.41	128.44±3.05	52.57±0.77
110	441.77±30.67	20.79±0.52	551.52±42.78	94.74±5.15	6.54±0.08

CONCLUSION

This study presents an investigation of the mechanical properties of GFRE with the effect of temperature at 30°C, 70°C and 110°C via experimental method. Tension, compression and shear tests were carried out under quasi-static loading within a thermostatic chamber. The main conclusion of this present study can be listed as follows:

i. Temperature increase from 30°C to 70°C has an insignificant effect on tensile strength and modulus of GFRE, for both fibre dominant and matrix dominant loadings. However, when applying temperature of 110°C, which is close to the glass transition temperature, the strength drastically reduced such that the strength remaining in matrix dominant loading (35.60%) is much lower compared to fibre dominant loading (58.85%).
ii. Compressive modulus and strength were reduced to a limited extent where more than half of the strength and more than 70% of the stiffness are retained in both 0° and 90° fibre orientations.
iii. Shear modulus and strength are substantially reduced with increasing temperature. The shear strength and modulus become less than 10% of the original 30°C state.
iv. Tensile and compression properties, when loaded along the fibre direction are less degraded by the increase of temperature, as the fibre is more resistant towards heat, whereas matrix dominant loads are more susceptible to the effect of heat.
v. At 70°C, GFRE is suitable for use under tensile and compression loadings. At 110°C, it is suggested for compression loading use. It is not advisable for shear loading use at both elevated temperatures.

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