**Mechanical properties and failure mechanisms of novel resin-infused thermoplastic and conventional thermoset 3D fabric composites**

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# Abstract

 This paper presents an extensive comparison of the mechanical properties and failure mechanisms of a recently developed thermoplastic (Elium ®) 3D fabric-reinforced composite (3D-FRC) with the conventional thermoset (epoxy) matrix 3D-FRC. Experiments involved tensile tests, compression tests, V-notch shear tests, and short beam shear tests for specimens produced through vacuum-assisted resin infusion in each case. These tests were used for the determination of in-plane elastic constants, failure strengths and for investigating the failure mechanisms. A micro-mechanical model validated against these experiments was used to predict the remaining orthotropic elastic constants. This work furthers our understanding of the mechanics of infusible thermoplastic-based 3D composites as a new class of emerging materials and provides useful data which substantiates that this unconventional thermoplastic resin, which is also easier to recycle and uses similar manufacturing processes, can be a suitable replacement for conventional thermoset resins.

**Keywords:** 3-Dimensional reinforcement, thermoplastic resin (Elium ®), thermoset resin, mechanical properties, failure mechanisms, micro-mechanics modelling.

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# List of abbreviations

Fibre Reinforced Composite (FRC); Interlaminar Shear Strength (ILSS); Vacuum-Assisted Resin Transfer Moulding (VARTM); Resin Transfer Moulding (RTM); Scanning Electron Micrographs (SEM); Methyl Methacrylate (MMA); Digital Image Correlation (DIC); Representative Volume Element (RVE); Co-efficient of Variance (COV), Micro Computed Tomography (Micro-CT); American Society for Testing and Materials (ASTM).

# 1. Introduction

The mechanical properties of FRC are strongly dependent on both, the resin system (thermoplastic or thermoset) and fabric architecture (2D or 3D reinforcement). The 2D-FRC’s (unidirectional and bi-directional) are known for their excellent in-plane properties; however, they possess poor delamination resistance [1-4]. To overcome this limitation, 3D-FRCs were developed, which have through-the thickness reinforcement yarn to increase interlaminar fracture toughness. This results in increased damage tolerance, impact and delamination resistance [5-11]. At present, 3D-FRC are mostly manufactured using liquid resin moulding processes such as infusion and RTM with thermoset epoxy resin for aerospace and wind energy applications [12]. The thermoset resins, however, are not recyclable or reusable in the true sense and therefore, there is growing emphasis on using thermoplastic polymers, which are easy to recycle [13]. Conventional thermoplastic polymers such as (Polypropylene and Polyphenylene sulphide) are typically manufactured using hot compression moulding processes (hot press-curing) as these are not liquid at room temperature. Recent studies of using these to manufacture 3D-FRC have reported poor fibre/matrix interface properties [14-17] and low interlaminar shear strength (ILSS) due to matrix defects (large voids) that appeared during the fabrication process [18] of the thermoplastic-based 3D composites.

Most of these problems result from the combination of the two facts; firstly, 3D-FRC’s are considerably thick (a single lamina can be over 45 mm thick). Secondly, the thermoplastic matrix (such as Polypropylene) is considerably viscous even at higher processing temperatures. Increasing the temperature and pressure further during the manufacture can reduce the extent of dry regions having poor fibre wet-out [14]; however, the process remains highly sensitive to the process parameters such as consolidation time, moulding temperature and pressure, etc., and imprecise control can significantly enhance the void content in the final product [19]. If one was to overcome these obstacles, the overall cost of manufacturing increases considerably as this cost increase is not simply the increase in associated energy consumption but includes the cost of tooling that is required for withstanding these higher temperatures and pressures and the enhanced control systems. This cost increase renders the manufacture of large thermoplastic 3D composite parts that are produced in relatively smaller volumes uneconomical. Thus, there is a strong need to develop novel thermoplastic polymers that remained liquid at room temperature and can thus be manufactured using the existing processes being used for thermoset 3D composites. This gap was recently partly filled by the introduction of an acrylic (MMA based) thermoplastic reactive resin system Elium®, which is liquid at room temperature. This newly developed liquid thermoplastic resin opens new ways to manufacture thermoplastic-based 3D-FRC using conventional processes such as vacuum-assisted resin transfer moulding (VARTM) and RTM [20-24].

In terms of mechanical properties of 3D-FRC, considerable research has been conducted in predicting the tensile [6, 25-28], compressive [6, 29-32], in-plane shear [27, 31, 33-35] and inter-laminar shear strength [36-39] of thermoset based 3D composites. Generally, the tensile stress/strain curve of 3D-FRC shows linear behaviour prior to damage initiation strain, which is followed by the nonlinear region until complete failure [6, 25-27]. The nonlinear response in the tensile stress/strain curve is due to the waviness in 3D fabric architectures, which is introduced by the through-thickness reinforcement. The in-plane elastic modulus of the 3D fabric composites is influenced primarily by the proportion of yarn in the warp and the fill (weft) direction, thus for example Tan et al. [40] studied the tensile behaviour of 3D orthogonal FRC and found that tensile strength and modulus were higher along the fill direction as compared to warp direction due to a higher proportion of yarn in the fill direction. The through-thickness reinforcement also has some influence on the in-plane properties, thus Brandt et al. [6] found that increasing through-thickness reinforcement, produce higher yarn waviness, which decreases the tensile modulus and increases the failure strain (nonlinear softening) of 3D-FRC. Cox et al. [41] found that this nonlinear softening under tensile load is due to the alignment of longitudinal yarns along the loading direction, followed by the matrix cracking, fibre failure and fibre pull-out, resulting in the brittle failure [42]. In terms of compressive properties, 3D-FRC failed due to defected zones (voids and cracks), followed by the fibre kinking across the width of the specimens [32, 43]. It was found that the waviness caused by the through-thickness reinforcement makes 3D-FRC more susceptible to kinking and fibre micro-buckling [44]. Das et al. [30] studied the compressive response of 3D-FRC and found that the compressive ductility is around 10% as compared to 2 to 4% in the case of 2D-FRC. The compressive ductility is also known as strain to failure, which is the compressive strain value, after which the material losses its load-carrying capability. The in-plane shear behaviour of 3D-FRC is highly nonlinear (due to plastic deformation and strain hardening) and exhibited higher failure strain, as compared to laminated composites [35]. Guo et al. [45] reported that the main damage modes under in-plane shear are interface debonding and tow splitting. The in-plane shear properties are highly dependent on the type of matrix and fibre, fibre-matrix interface, as well as unit cell size. Thus, for example, Qin et al. [34] used the Iosipescu shear test to determine the in-plane shear behaviour of C/C 3D-composites with a 2.4 mm unit cell and reported 19% failure strains. Whereas, Warren et al. [31] used the V-notched shear rail test for Carbon / Epoxy 3D composites having 9.8 mm unit cell and found that 3D composite failed at 10% strains. The through-thickness reinforcement contributes to increasing the shear strength and failure strain, for example, Munoz et al. [35] used bias off-axis specimen (±45°) to study the effect of through-thickness reinforcement on the shear properties of thermoset based 3D composite and found that the inter-ply damage propagation was significantly reduced.

Thermoplastic-based 3D composites have been studied by relatively few authors. [15, 17, 18]. Bandaru et al. [15] studied the tensile/compressive behaviour of polypropylene-based 3D hybrid composite. The authors found that the matrix yielding, fibre buckling, and breakage were the main failure modes. The authors also studied the in-plane shear behaviour of thermoplastic-based 3D composite using the Iosipescu test [17]. The thermoplastic generally exhibits higher failure strain than thermosets; studies with thermoplastic matrices have reported lower than expected maximum failure strain for 3D composites due to a poor fibre-matrix interface. For example, the maximum failure strain for the polypropylene matrix-based hybrid 3D composites was 8% due to poor fibre/matrix interface. Similarly, Archer et al. [18] studied the ILSS of thermoplastic 3D composites. The authors concluded that the ILSS was reduced due to matrix defects (large voids) that appeared during the fabrication process. Traditional thermoplastic matrices require high-temperature processing and even at these higher temperatures, the matrix viscosity can be significantly high, especially for thick 3D fabric, causing poor fibre wet-out and weak interfacial properties. Thus, there has not been much interest in these composites. Liquid thermoplastic-based composites can potentially solve this problem however to the best of the author's knowledge, no comprehensive study is available for the tensile, compressive, in-plane shear, and ILSS of liquid thermoplastic-based 3D composites and their failure mechanisms.

Regarding this new liquid thermoplastic resin system (Elium), in recent years a few studies have reported the evaluation of Elium based 2D fabric composites for in-plane mechanical properties [22, 24, 46, 47] and few for the out-of-plane response (such as low-velocity impact) [20, 48-51]. The evaluation and comparison of the same for 3D composites are, however, severely lacking. In this regard, recently, the authors of this study have presented a comparison of impact damage tolerance for the resin-infused thermoplastic and thermoset 3D-FRC [8]. To the best of the author's knowledge, however, no study has yet been reported for the full evaluation and comparison of the mechanical properties and failure mechanisms of 3D-FRC’s composites resin-infused with thermoplastic (Elium®) and thermoset. This clearly highlights the need for rigorous benchmarking as well as developing an understanding of the fundamental failure mechanisms of this novel thermoplastic-based 3D-FRC.

Following these directions, this paper aims to benchmark the performance of resin-infused thermoplastic-based 3D composite manufactured using novel acrylic thermoplastic resin (Elium®) with a conventional thermoset (epoxy) based 3D composite. In this regard, the tensile, compression, V-notch shear, and short beam shear test was conducted on both 3D composites. The mechanical properties obtained from these tests, i.e., tensile/compressive strength, tensile/compressive modulus, tensile/compressive failure strains, Poisson’s ratio, in-plane shear modulus/strength, and interlaminar shear strength, were compared to understand the effect of resin toughness on the mechanical properties of 3D composites. Furthermore, a comprehensive macro and micro-damage characterization have been performed to identify damage modes and failure mechanisms using a collection of digital images and scanning electron micrographs (SEM) of damaged specimens. Finally, the experimental results are also compared with the analytical prediction based on the elastic mechanic's approach, i.e., the volume averaging method. From this study, reliable data of both 3D-FRCs have been produced that can be used in numerical simulation and design for different applications.

# 2. Materials and methods

# 2.1. Material used

The 3D fabric used in this study is 3D orthogonal E-glass woven fabric (3D-9871) obtained from TexTech® Industries, USA as shown in the schematic diagram in Fig. 1(a). The areal weight of the fabric is 5200 gm-2, and the overall thickness of a single layer is 4.3 mm. The fabric consists of three layers of yarn along the warp direction (machine direction) and four layers of yarns along the fill direction (cross-machine direction), (see Fig. 1(a)). The fabric has 49% fibres along the warp and fill directions to maintain the same in-plane properties; whereas, only 2% of fibres are present in the third direction to bind the layers together. In this study, the 3D-FRC panels were fabricated using both thermoplastic and thermoset resin systems, i.e., acrylic thermoplastic liquid resin Elium® 188x0 from Arkema and thermoset epoxy resin system Epolam® 5015/5015 from Axson. Elium® 188x0 comprises mainly methyl methacrylate (MMA > 50%), acrylic copolymers (>10%), and a proprietary polymer (2-propeonic acid). These are mixed with a peroxide initiator to initiate the polymerization process at room temperature. The percentage of peroxide may vary between 2% (slow polymerization) to 4% (fast polymerization), depending upon the requirement. However, in this study, to fabricate thermoplastic composite, the percentage of Elium and peroxide initiator was set as 100:2.3 to achieve medium polymerization time (2.3 grams of peroxide is mixed with 100 grams of Elium® resin). Meanwhile, to fabricate thermoset composite, the epoxy and hardener ratio used was 100:30 by weight [52]. Further details about the cure kinetics of Elium resin have been recently described in the reference [8]. The glass transition temperature of Elium 188x0 and Epolam 5015 is 106 °C and 85 °C, respectively [46, 53]. This indicates that the resin-infused 3D-FRC can operate safely at sufficiently high temperatures. The maximum operating temperature of epoxy-based composites, cured at room temperature is up to 120°C [54]. Thus, in terms of service temperatures, the performance of Elium® is comparable to other epoxy matrices.

# 2.2. Fabrication process

Both thermoplastic and thermoset based 3D-FRCs were manufactured using a vacuum-assisted resin transfer moulding (VARTM) process. The mixed viscosity of Elium® 188x0 is 200 mPa.s, and Epolam® 5015 is 210 mPa.s, which is ideal for the VARTM process. The resins systems (Elium®/peroxide and epoxy/hardener) were mixed carefully for two minutes to get a homogenous mixture. Before starting the infusion process, the epoxy resin was degassed for 25 minutes to remove air bubbles. Since the pot life of thermoplastic resin is only 60-70 minutes, it was degassed for 15 minutes, and the resin pot is covered with a lid to control air bubbles formation. The infusion was carried out in a clean environment, and the fabric was dried in an oven for 2 hours at 125°C prior to the VARTM process; this was to remove any trapped moisture. Apart from this, no specific sizing or fabric treatment was required for either resin. A rectangular panel 400 mm in length and 500 mm in width was fabricated. Fig. 1(b) shows the VARTM of 3D FRC. In the case of thermoplastic-based 3D-FRC, the VARTM process was carried out at 100 mbar to avoid resin boiling during infusion, and the infusion process was completed in 25 minutes. After the VARTM process, the panels were left at room temperature for four hours to complete the polymerization process, followed by the post-curing in an oven at 80°C for eight hours to achieve maximum mechanical properties. Whereas in the case of thermoset based 3D-FRC, the VARTM process was carried out at 450 mbar, and the infusion was completed in 7 minutes. After curing at room temperature for twelve hours, the thermoset 3D-FRC panels were post-cured in an oven at 80°C for eight hours. Fig. 1(b) also shows the infusion time. In both cases, the infusion rate was fast at the start of the infusion process, and it slows down as the impregnated length increased.

The in-situ polymerization in Elium® resin depends on the initial viscosity of the resin, amount of catalyst (peroxide) mixed with monomer (MMA) and processing temperature. The low initial viscosity of Elium® resin is effective for the proper impregnation of fabric at room temperature. The in-situ polymerization can be accelerated by increasing the amount of catalyst (2% (slow polymerization) to 4% (fast polymerization)), or processing temperature (6 minutes at 80 °C). During this process, the viscosity of resin increases due to the formation of polymethyl methacrylate or PMMA. This increase in the molecular size, increases the viscosity and consequently, the resin flow front becomes stationary when the viscosity reaches its upper limit, i.e., ~ 800 mPa·s. Therefore, in the industrial environment, all these parameters must be optimized to get proper impregnation of fabric and desired product quality.

# 2.3. Physical parameters of the cured panel

Fig. 1(c) shows the fabricated thermoplastic and thermoset 3D-FRC panels, respectively. After the panels were fully cured, they were checked for defected regions and physical parameters such as density, fibre volume fraction, and thickness of the panel were calculated/measured. The fibre volume fraction of fabricated panels was measured using the burn-off method according to ASTM D3171-15, and the density was measured using the water displacement method according to ASTM D792-08. Ten samples were cut from different panels, and the average void content, density, and fibre volume fractions and thickness were measured. These are listed in Table 1. As can be seen from this table, the average volume fraction was approximately 52%. In the case of the thermoplastic composite, the void content was less than 3%; in comparison, in the thermoset composite, the void content was less than 1%. This is attributed to the higher volatile emission for the thermoplastic (Elium) during the condensation polymerization reaction. This can also lead to more variation in measured fibre volume fraction for the thermoplastic-based composite. However, in this study, with low vacuum pressure and using carefully measured quantities of peroxide for the cure reaction, the variation in volume fraction was limited to be within ±1.5%. The specimens were cut from panels using a water-based diamond tip disc cutter, which gives an excellent surface finish. Seven samples were prepared for each test to get a minimum of five valid mechanical tests. After cutting, the samples were conditioned for 24 hours at 23 ºC and 50% relative humidity to ensure that consistent temperature and relative humidity condition for each sample prior to testing, as required by the standard [55, 56].

# 2.4. Mechanical testing of 3D composites

Tensile, compression, V-notch shear, and short beam shear tests were performed on both types of 3D composites. In the case of tensile, compression, and short beam shear tests, the specimens were tested along both warp and fill direction, whereas, in the case of the in-plane shear test, the specimens were tested along fill direction. Fig. 2 shows a schematic representation of the 3D-FRC specimens along with the dimensions and boundary conditions for each type of mechanical property test used in this study. These samples were cut according to the relevant ASTM standards. The properties determined from these tests and the testing standards used were, a) tensile strength, tensile modulus, failure strains, and Poisson’s ratio from the tensile test ASTM D3039 [55], b) compression test, compression strength, modulus, and failure strains from compression test ASTM D6641 [56], c) in-plane shear modulus and strength from a V-notch shear test ASTM D7078 [57], and d) inter-laminar shear strength from a short beam shear test ASTM D2344 [58].

The resulting strains due to applied load were determined using strain gauges, extensometer, and digital image correlation (DIC), as shown in Fig.2. Strain gauges were used to determine Poisson’s ratio, compressive failure strains, compressive modulus, and shear modulus; an extensometer was used to determine tensile failure strain and tensile modulus; whilst the DIC was used to measure shear strength and strains. In this research work, 350 Ohm’s KFRPB series (KYOWA) strain gauges were used for strain measurement. 5 mm long grid length (15 mm-base lengths) strain gauges were used for the tensile and V-notch shear test (see Fig. 2(a) and (c)), which can cover almost two yarns of 3D orthogonal woven composites. Whereas, due to the smaller gauge length of compression test specimens, a 2 mm long grid length (10 mm base length) strain gauges were used (see Fig. 2(b)). In order to measure tensile failure strains, Epsilon® 50 mm displacement extensometer was used. All the experiments were performed at controlled room temperature i.e., 23-25 °C. In the following section, the test setup and detailed procedures for different tests are discussed.

# 2.4.1. Tensile testing

The tensile tests were performed according to ASTM D3039 test protocol [55], using the ZwickRoell hydraulic-driven load frame. The load frame was instrumented with a 50 kN load cell to record the force experienced by the specimen at each time step. Fig. 3(a) shows the test setup for the in-plane tensile test of 3D-FRC. A displacement controlled load rate of 1 mm/minute was applied, and resulting strains were measured using an extensometer (to measure failure strain and tensile modulus) and strain gauges (to measure Poisson’s ratio). The justification for using the extensometer to measure failure strain was that Dai et al. [5] observed the failure/debonding of strain gauges before the specimen reached its final failure. The dimensions of the specimens were 250 mm x 25 mm x 4 mm, with a 150 mm gauge length (see Fig. 2(a)). Five samples were tested for each configuration, i.e., along the warp and fill directions. The specimens were bonded with end tabs, which were fabricated using bi-directional carbon plain weave using the same vacuum infusion process. The nominal thickness of the tabs was 2 mm. The tabs were bonded on the specimens using aerospace-grade adhesive PT 326 from Paramount, USA. The tensile tests were performed along both warp and fill directions.

# 2.4.2. Compression test

The compression tests were carried out following the ASTM D6641 standard [56], using a ZwickRoell hydraulic-driven load frame, which is equipped with a 100 kN load cell. The load frame was also equipped with spherically seated platens to eliminate alignment issues between the load frame and fixture, as shown in Fig. 3(b). The ASTM D6641 fixture is a combined loading fixture, which can simultaneously apply end and shear loads. The dimensions of the specimens were 140 mm x 25 mm x 4 mm (see Fig. 2(b)), with a 13 mm gauge length, and five samples each were tested along the warp and fill directions. The compression test specimens were bonded with end tabs using the same procedure as discussed in section 2.4.1. The compressive strains were measured using strain gauges glued to the specimen.

**2.4.3. V-notch shear test**

 The in-plane shear properties of 3D composites were determined using a V-notch shear test, according to ASTM standard D7078 [57]. Three specimens were tested for each thermoplastic and thermoset 3D composite. The V-notched shear test is suitable for the 3D composite due to its large unit cell (10.8 x 3.7 x 4 mm). The tests were conducted on a ZwickRoell hydraulic-driven load frame, which is equipped with a 100 kN load cell. The load was applied in the form of crosshead displacement at a rate of 1 mm/min, and resulting strains were recorded using strain gauges and Digital Image Correlation (DIC). Fig. 3(c) shows the experimental setup for the in-plane shear test. The V-notched shear test specimens geometry was machined using a milling machine following the dimensions recommended in the standard and are shown in the schematic Fig. 2(c). The surface of coupons was roughened using sandpaper to ensure proper grip in the fixture. The V-notched samples were placed in the fixture and aligned, while the bolts were tightened with 54 Nm of torque. Two strain gauges were bonded at the centre of the coupon, on the bottom surface to measure strains in the ±45º direction, i.e. “$ε\_{-45°}$” and “$ε\_{+45°}$” see Fig. 2(c) and the shear strains were determined by taking their average i.e. $γ\_{12}= \left|ε\_{+45°}\right|+\left|ε\_{-45°}\right|$ as defined by ASTM standard D7078 [57]. The average shear stress was calculated by dividing the applied load *“P”* with the cross-sectional area *“A,”* i.e., $τ\_{12}= P/\left(L × t\right)$, where, *“L”* is the length of the ligament and *“t”* is the thickness of the coupon (see Fig. 2(c)).

In addition to strain gauges, 2D-DIC was used to measure strain fields on the face opposite to the one where strain gauges were mounted (i.e., back face in Fig. 2(c)). The surface of the coupon was prepared for DIC measurement by first spraying the surface with flat white paint, and then random speckles were made with flat black paint, as shown in Fig. 2(c). The video was recorded with a single Cannon Legria® 8-megapixel digital camera. These images were post-processed using GOM® correlate software to evaluate shear strains.

**2.4.4. Short beam shear test**

The inter-laminar shear strength of thermoplastic and thermoset based 3D composites were determined as per ASTM D2344 [58]. The specimens were loaded on a GoTech® electromechanical load frame, equipped with a 5 kN load cell. Fig. 3(d) shows the experimental setup for the short beam shear test. The tests were performed on a three-point bending fixture with a span-thickness ratio of 5.0 (fixed span 20 mm), see Fig. 2(d). Twenty rectangular specimens (each sample is 45 mm x 25.4 mm x 4mm in dimension) were tested along warp and weft direction. The specimens were loaded at a load rate of 1.0 mm/minute up to a central displacement of 2.5 mm to determine the failure and post-failure behaviour of both 3D composites. The specimen is supported by two rollers to initiate failure by interlaminar shear. The performance of both materials was evaluated through inter-laminar shear strength, calculated using the formula standard recommended formula, i.e., ILSS = 0.75*P/wt*, where “P”, “w” and “t” represent peak load, specimen width, and thickness, respectively.

# 2.5. Damage evaluation methods

A detailed fractography has been carried out to understand the failure mechanisms in both 3D composites. These failure mechanisms were characterized by macro and micro failure mechanisms. The macro damage morphologies of failed specimens were analysed through a collection of digital images obtained after mechanical tests. Meanwhile, the micro failure mechanisms were evaluated using SEM. Both 3D composites were fabricated using glass fibres, which is an insulator; therefore, the surface of specimens was coated with a 40 nm gold coating to make them conductive.

**2.6. Prediction of effective properties using elastic mechanics approach**

The effective properties (elastic constants) of both 3D composites were predicted using the volume averaging method [32, 59, 60]. It consists of a two-step homogenization scheme, i.e., micro-meso and meso-macro homogenization. The overview of the volume averaging method is shown in Fig. 4. This method required realistic internal geometry details (cross-sectional area of yarns and RVE dimensions), which were obtained from micro- CT (micro-computed tomography) of the fabricated 3D composite. Fig. 5(a)-(c) shows the cross-sections of warp yarn, fill yarn and z-binder yarn obtained through micro-CT, whereas, Fig. 5(d) shows the RVE, defined based on the micro-CT analysis. The effective properties of 3D-FRC were evaluated using elastic constants of constituents (fibre and matrix) and their volume proportion in the RVE. The cross-sectional details obtained from micro-CT (Fig. 5(a)-(c)), were used to evaluate the cross-sectional area $A\_{i}$, fibre volume fraction $V\_{f,i}$ and volume proportion $V\_{p,i/m}$ of each constituent, given by Eqn (1)-(3).

|  |  |
| --- | --- |
| $$V\_{f,i}=\frac{T\_{i}}{ρ\_{i}A\_{i}}$$ | *i = warp,fill and z-binder yarn* (1) |
| $V\_{i}=l\_{i}A\_{i}$ , $V\_{m}=V\_{REV}-\sum\_{}^{}V\_{i}$ | (2) |
| $$V\_{p,i/m}=\frac{V\_{i}}{V\_{RVE}}$$ | (3) |

where, $l\_{i}$,$ ρ\_{i}$, $T\_{i}$, $V\_{i}$, $V\_{m}$ and $V\_{RVE}$ represents length, density, Tex property, volume of each impregnated yarn and RVE volume, respectively. In the first step, micro-meso homogenization was performed, where elastic constants of impregnated yarns were evaluated in the local coordinate system (1,2,3) through elastic constants of fibre and matrix and the fibre volume fraction of impregnated yarns using the micro-mechanics model proposed by Chamis [61], given by Eqn. (4). The elastic constants of fibre (E-glass) and matrix (Elium or Epolam) are given in Table 2.

|  |  |
| --- | --- |
| $$\left\{\begin{matrix}\begin{matrix}E\_{11}=V\_{f}E\_{11,f}+\left(1-V\_{f}\right)E\_{m} \\E\_{22}=E\_{33}=\frac{E\_{m}}{1-\sqrt{V\_{f}}(1-\frac{E\_{m}}{E\_{22,f}}) } \\G\_{12}=G\_{13}=\frac{G\_{m}}{1-\sqrt{V\_{f}}(1-\frac{G\_{m}}{G\_{12,f}}) }\end{matrix}\\\begin{matrix}G\_{23}=\frac{G\_{m}}{1-\sqrt{V\_{f}}(1-\frac{G\_{m}}{G\_{23,f}}) } \\v\_{12}=V\_{f}v\_{f}+\left(1-V\_{f}\right)v\_{m} \\v\_{23}=\frac{E\_{22}}{2G\_{23}}-1 \end{matrix}\end{matrix}\right.$$ | (4) |

where, “$V\_{f}$”,”$ v\_{f}$” “$E\_{f}$” and “$G\_{f}$” represent the fibre volume fraction, Poisson’s ratio, modulus of elasticity, and modulus of rigidity of the fibres. The constants “$ v\_{m}$” “$E\_{m}$” and “$G\_{m}$” represent the Poisson’s ratio, modulus of elasticity and modulus of rigidity of the matrix and the constants “$E\_{11}$”, “$E\_{22}$”, “$E\_{33}$”, “$G\_{12}$”, “$G\_{13}$”, “$G\_{23}$”, “$v\_{12}$”, “$v\_{23}$” represents the effective modulus of elasticity, modulus of rigidity, and Poisson’s ratio of the impregnated yarn in a local coordinate system. These local stiffness matrixes (warp, fill, and z-binder) were transformed with respect to the global coordinate system (x, y, z) to get stiffness matrixes in the global coordinate system. In the second step, meso-macro homogenization was performed, where transformed stiffness matrixes in the global coordinate system “$\left[\overbar{Q}\_{i}\right]$” and volume proportion of individual yarns in the RVE “$V\_{pi }$” were used to determine the global stiffness matrix of 3D composites “$\left[Q\_{RVE}\right]$”, see Eqn. (5). The effective properties of 3D composites were then calculated by taking the inverse of the global stiffness matrix to get a global compliance matrix, see Eqn. (6) and (7).

|  |  |
| --- | --- |
| $\left[Q\_{RVE}\right]=\sum\_{}^{}V\_{pi }\left[\overbar{Q}\_{i}\right]$ (i = warp, fill, z-binder and matrix) | (5) |
| $$\left[S\_{RVE}\right]= \left[Q\_{RVE}\right]^{-1}$$ | (6) |

|  |  |  |  |
| --- | --- | --- | --- |
| $$E\_{x } = \frac{1}{S\_{11}}$$ | $$E\_{y }= \frac{1}{S\_{22}}$$ | $$E\_{z } = \frac{1}{S\_{33}}$$ |  |
| $$γ\_{xy }= -\frac{S\_{21}}{S\_{11}}$$ | $$γ\_{xz }= -\frac{S\_{31}}{S\_{11}}$$ | $$γ\_{yz }= -\frac{S\_{32}}{S\_{22}}$$ |  |
| $$G\_{yz }= \frac{1}{S\_{44}}$$ | $$G\_{zx }= \frac{1}{S\_{55}}$$ | $$G\_{xy }= \frac{1}{S\_{66}}$$ |  (7)  |

# 3. Results

# 3.1. In-plane tensile test

The experimental results obtained from in-plane tensile tests, i.e., tensile modulus ($E\_{11}^{T}$,$ E\_{22}^{T}$), ultimate tensile strength ($X^{T},Y^{T}$), and failure strains ($ԑ\_{11}^{T},ԑ\_{22}^{T}$) of both thermoplastic and thermoset based 3D-FRC are summarised in Table 3. Each value in the table represents the average value of five tested samples; whereas, the values in the bracket show the coefficient of variance (COV) in the results. The thermoplastic-based 3D-FRC exhibited the highest strength, i.e., 478 MPa, when loaded along warp direction. While the thermoset based 3D-FRC possesses the lowest strength, i.e., 414 MPa when loaded along fill direction. The thermoplastic fill loaded specimens show the lowest modulus, i.e., 24.9 GPa; in comparison, the thermoset warp loaded specimen showed the highest modulus, i.e., 26.3 GPa. These elastic moduli show excellent correlation with the predicted elastic moduli through the micro-mechanical modelling approach, with a maximum deviation of 2.6% (see Table 4).

Fig. 6(a) shows the average tensile stress/strain curves of both thermoplastic and thermoset based 3D-FRC. The results highlight that the tensile strength and failure strains of thermoplastic-based 3D-FRC are higher; in contrast, their tensile modulus is slightly lower than thermoset based 3D-FRC. Also, the tensile strength along warp loaded specimens is higher than the tensile strength along with fill loaded specimens. The tensile modulus was measured separately along the warp and fill directions to provide estimates of both longitudinal and transverse moduli. The stress/strain curves exhibited linear response up to the damage initiation strains. The average damage initiation strains for the thermoset ($ε\_{el}^{TS}$) and thermoplastic ($ε\_{el}^{TP}$) specimen were 0.5% and 0.75% strains, respectively. After this point, the nonlinear region starts; however, the stiffness only reduces slightly in the nonlinear regime indicting that such reduction in stiffness was primarily due to micro-matrix damage mechanisms that are explained subsequently in the discussion section. Such a nonlinear region in the tensile stress/strain curves has also been reported for thermoset composites by Warren et al. [31] and Callus et al. [26]; however, such mechanisms have not been explained for liquid thermoplastic resins based 3D-FRC.

Fig. 6(b)-(d) shows a comparison between normalized strength, stiffness, and failure strains along the warp and fill direction of thermoplastic and thermoset 3D-FRC. Normalizing the strength, stiffness, and failure strain allows us to evaluate both materials and gives better visual comparison under tensile loads. All the values were normalized with thermoplastic warp specimens strength, failure strains, and modulus. The experimental results indicate that, overall, the thermoplastic-based 3D-FRC possesses 15.5% higher tensile strength and 21% higher failure strains (see Fig. 6(b) and (d)) than thermoset based 3D-FRC. The thermoplastic-based 3D-FRC loaded along warp direction gives 6.5% and 11% higher tensile strength and failure strain than thermoset based 3D-FRC, respectively. However, their tensile modulus is 3.5% lower as compared to thermoset based 3D-FRC (see Fig. 6(c)). On the other hand, thermoplastic-based 3D-FRC loaded along fill direction shows 11% higher tensile strength and 15% higher failure strain, respectively. However, their tensile modulus is 4.5% lower than thermoset based 3D-FRC.

The Poisson’s ratio in both 3D-FRC was evaluated using two strain gauges. These were bonded in the middle of the gauge section, one along the 0° degree (loading direction) and 90° degree (transverse direction) to get longitudinal “$ε\_{1}$” and transverse “$ε\_{2}$” strains. These longitudinal and transverse strains were used to evaluate the Poisson’s ratio in both 3D-FRC. The Poisson’s ratio was only evaluated in the linear range. The in-plane Poisson’s ratio was measured along with both directions, i.e. “$ v\_{12}$” and “$ v\_{21}$”. Table 3. shows a comparison of Poisson’s ratio in 3D-FRC along both directions. The values in the parenthesis represent the coefficient of variance in the data. One possible reason for the large variation in the Poisson’s ratio could be that the strain gauge used for the tensile tests, which has a 5 mm grid length, is not enough to cover the whole unit cell of 3D-FRC (10.8 mm x 7.4 mm in dimension). Thus, the measured strains were actually local strains based on the location of the strain gauge within the unit cell. In addition to this, the surface variabilities, i.e., resin pockets between fill yarns and interlacing of z-binder, could be one of the reasons for higher variability in the measured Poisson’s ratio. The Poisson’s ratio “$ v\_{12}$” predicted through the micro-mechanical model for thermoplastic and thermoset 3D composites are within 7.1% and 6.5% deviations with experimental data (see Table 4).

**.2. In-plane compressive test**

Table 5 summarises the key results from the compression test. Each value in the table represents the average value of five tested specimens. The tables show the compressive modulus ($E\_{11}^{C}$,$ E\_{22}^{C}$), strength ($X^{C},Y^{C}$) and failure strains ($ԑ\_{11}^{C},ԑ\_{22}^{C}$) of both 3D-FRC. The values in the parenthesis show the COV in the results. In the in-plane compression tests, the alignment of the specimen within the fixture and the alignment of the test fixture with the testing machine is very critical to avoid the global buckling of the specimen. In order to eliminate alignment issues between the test fixture and the testing machine, a spherically seated platen was used. To ensure alignment of a specimen within the test fixture, two strain gauges were mounted on the opposite face of the specimen to calculate percentage bending, as suggested by the ASTM standard D6641 [56], i.e., percentage bending $=((ε\_{1}-ε\_{2})/(ε\_{1}+ε\_{2}))×100$. The maximum percentage bending in the specimens was below 7%, which was within the bounds in comparison with the ASTM standard which allows a maximum of 10% bending. In addition, the specimens failed in the gauge section as per the acceptable modes in ASTM standard D6641.

Overall the 3D-FRC loaded along fill direction exhibited higher compressive strength (and failure strain) as compared to warp loaded specimens. The stress/strain curves of both thermoplastic and thermoset 3D-FRC are shown in Fig. 7(a). This figure indicates that the thermoplastic and thermoset based 3D FRC shows linear behaviour up to 0.5 % and 0.25 % compressive strains, respectively. The compressive modulus reported in Table 5 was measured within this linear region for each case. After this point, the nonlinear deformation starts, which can be as a result of damage accumulation or local plastic deformation (especially in the case of thermoplastic – more on this in the discussion section). The thermoset warp 3DFRC samples show the least nonlinear stiffness reduction and fail abruptly in a brittle manner, while the thermoplastic weft 3DFRC samples show the highest level of progressive nonlinear stiffness reduction and highest strain to failure.

The thermoplastic-based 3D-FRC shows the highest compressive strength, i.e., 357 MPa, and lowest COV, i.e., 2.2%, when loaded along the fill direction. In contrast, the thermoset based 3D-FRC possesses the lowest compression strength (and failure strain), i.e., 257 MPa, and the highest COV, i.e., 10%. In both types of 3D-FRC, the compressive modulus values along warp loaded specimens were higher as compared to fill loaded specimens. The thermoset based 3D-FRC possesses the highest compressive modulus, i.e., 33.4 GPa when loaded along warp direction. On the other hand, the thermoplastic-based 3D-FRC shows the lowest compressive modulus, i.e., 26.4 GPa, when loaded along fill direction. The thermoplastic-based 3D-FRC shows the highest failure strains, that is., 1.70 % with 11.7 % COV, as compared to 1.16 % with 11% COV when loaded along the fill direction. All these results are discussed further with reference to failure modes in the discussion section.

Fig. 7(b)-(d) shows the comparison between normalized compression strength, modulus, and failure strains of thermoplastic and thermoset based 3D-FRC. The strength, modulus, and failure strain were normalized with thermoplastic-based 3D-FRC strength, modulus, and failure strain loaded along fill direction. Fig. 7(b) shows the normalized compression strength comparison. The thermoplastic-based 3D-FRC exhibited 19% higher compression strength as compared to thermoset based 3D-FRC when loaded along fill direction. However, thermoset based 3D-FRC possesses a 23% higher compressive modulus, as shown in Fig. 7(c), when loaded along warp direction. Fig. 7(d) shows the comparison between the failure strain of both types of 3D-FRC. The thermoplastic-based 3D-FRC possesses 46% higher failure strains as compared to thermoset based 3D-FRC loaded along fill direction.

**3.3. V-notch shear test**

It is well known that 3D-FRC shows a large strain to failure in shear. It is typically observed that at large shear strains, the strain gauge readings become less reliable if problems such as gauge detachment occur during the test. Techniques such as DIC allow for the measurement of both small and large strains and can be more accurate for large strains due to their non-contact nature [62]. Thus it was decided to use DIC to measure the full strain field until failure. However, for reliable DIC measurement, the DIC setup and test results should be validated. This was achieved by comparing the strain measurements from strain gauges and from DIC for small strains, i.e. until first signs of gauge detachment appeared (Both strain gauges measured strains up to ~18000 micro strains (1.8%); afterwards, they started to detach from the surface due to large deformations in the specimen).

Fig. 8(a) shows one such validation case where the strain field obtained from DIC for the representative test case is shown. As expected, the highest shear strains were observed directly inline, underneath the V-notched zone. Shear strain extracted from the strains strain gauges (±45º) (shown in Fig. 2(c)) and that extracted from the corresponding location on the opposite face from the DIC are compared in Fig. 8(b). This shows that the shear strains obtained from DIC and strain gauges matched well up to 1.8% strains (equivalent shear stress at 1.8% strain was 41 MPa). DIC measured up to 24% and 21.5% shear strains in thermoplastic and thermoset composites, respectively. Fig. 8(c) shows the comparison of load/displacement curves. The area under the load/displacement curves indicates the amount of energy dissipated during the process. The thermoplastic composite dissipates 32% higher energy as compared to the thermoset composite. The thermoset composite reached a peak load of 6.7 kN at 4.5 mm displacement; in contrast, the thermoplastic composites reached a peak load of 6.9 kN at 6.3 mm. This indicates that in thermoplastic composites, peak load was reached at ~29% higher displacement. In terms of peak displacement, the thermoplastic composite fails at 8 mm displacement, which is 35% higher than the thermoset composite.

The corresponding shear stress/strain curves of both 3D composites are depicted in Fig. 8(d). The shear modulus and shear strength were determined from stress/strain curves. The shear modulus was determined from the slope of stress/strain curves in the linear region (using shear strain range between 1500 to 4500 micro-strains) [57], i.e., up to point “a” in Fig. 8(d). Whereas the shear strength was measured by translating the linear portion of the stress/strain curve along the strain axis by 2%. The line is then extended until it intersects the shear stress/strain curve, which gives the shear strength value (see Fig. 8(d)) [63]. It is worth noticing that the thermoset composites show higher shear modulus and pseudo yield strength; however, the thermoplastic composite shows maximum shear strength, i.e., ~55 MPa (represented by the point “d” in Fig. 8(d)).

The results obtained from the in-plane shear test are summarised in Table 6. The initial shear modulus of the thermoplastic composite is (4.4 GPa), which is ~6% lower as compared to the thermoset composite (4.7 GPa). The predicted shear moduli through the micro-mechanical modelling approach for the thermoplastic and thermoset composites are 4.69 GPa and 4.82 GPa, respectively (see Table 4), which shows a good correspondence with the experimental results. The thermoplastic composite shows a higher deviation (5.1%), which may be due to higher void content. Overall, the predicted elastic constants through this approach show reasonable accuracy and therefore this approach can be used for predicting and comparing elastic constants at various volume fractions and can also be used to generate a full set of orthotropic input material constants for 3D finite element analysis of composite structures made from these 3D composites. Thus, in Table 4 full set of predicted orthotropic material constants are enumerated using this validated micro-mechanics approach.

**3.4. Inter-laminar shear test**

For each 3D composite, a load-displacement curve was selected that represents an average of five curves, as shown in Fig. 9. Both materials show linear response until the onset of damage, followed by the nonlinear region. The fill loaded specimens show the highest peak load, bending stiffness, and the lowest displacement at peak load. Among fill loaded specimens, the thermoplastic composite shows the highest peak load of 4.3 kN. After reaching the peak load, both thermoplastic and thermoset composite showed up to 15% and 20% load drop, respectively. However, the load level remains almost constant over the remainder of the test. The warp loaded specimens show higher nonlinearity, displacement at peak load, and the lowest bending stiffness. Among warp loaded specimens, the thermoset composite shows a higher peak load (3.4 kN) and undergoes a sudden load drop of 30-40%; in contrast, the thermoplastic composite shows a gradual load drop of 5-15%. Hence, the fill loaded thermoplastic composite achieved the highest peak load (4.3 kN); whereas, the warp loaded thermoplastic composites show the lowest peak load (3.3 kN) and the highest displacement (1.9 mm) among all four configurations. From load/displacement histories, the ILSS was calculated, as discussed in section 2.5.4. The average ILSS of both thermoplastic and thermoset composite is summarised in Table 7. The fill loaded thermoplastic composite shows the highest ILSS (32.2 MPa), which is 20% higher than fill loaded thermoset composite (25.8 MPa) and 29% higher than warp loaded thermoplastic composite. The ILSS of warp/fill loaded thermoset composite is close to each other, with a difference of 4.2%.

**4. Discussion on results**

# 4.1. In-plane tensile behaviour

The proportional ratios of warp yarn, fill yarn, and z-yarn in the 3D orthogonal woven fabric used in this study were 1:1:0.04, which indicates a very similar percentage of fibre contents along the warp and fill direction. Since tensile modulus is fibre dominated property, so theoretically, it should results in the same elastic modulus along both directions; thus, we see in Fig. 6 that the tensile stress/strain curves show almost identical behaviour along warp and weft for the starting portion of the curve; however, it diverges slightly and shows some nonlinearity for higher strain regions (0.5-2.5%), with a minor reduction in the effective Young’s modulus. This reduction in stiffness can be hypothesized to be partially caused by the matrix cracking and partially by the fibres straightening effects, as demonstrated earlier by Warren et al. [31] for toughened thermoset composites.

The tensile strength along warp loaded specimens was higher than the fill loaded specimens. One of the main reasons for this strength reduction along the fill direction is the interlacing of z-yarn (z-crown), which produces undulation along the fill yarn direction on the top and bottom surface of the 3D fabric as shown by the schematic diagram in Fig. 10(a). Such z-crown produces high stress-concentrated localized regions with the resin-rich pockets at these locations. These pockets are most susceptible to damage initiation (see Fig. 10(a)). In thermosets, this leads to the initiation of extensive matrix cracking, as shown in Fig. 10(e) zone-i. This extensive cracking makes the fabric unstable, and thus the final failure occurred at zone-ii in Fig. 10(e). This extensive fibre rupture of both fill and warp yarns at the lowest ultimate failure strains, as given in Fig. 4. On the other hand, for thermoplastic fill loaded specimens in Fig. 10(d) at zone-i, the evidence of localized matrix plastic yielding around the fill yarn can be seen; this leads to a more stable stretched orientation of the failure, see Fig. 10(d) at zone-ii and thus, the thermoplastic 3D-FRC achieves a higher strain to failure then both the warp and weft cases of thermoset 3D-FRC. The warp loaded thermoplastic 3D-FRC shows the highest strain to failure, and the failure zone in Fig. 10(b) clearly shows evidence of a much more stable fibre fracture aided by the tougher thermoplastic matrix and lack of stress concentrations as were seen for fill loaded specimens. Stress-concentration due to the z-crown are also logically expected to be more severe near the tab ends, and perhaps that is why all fill loaded specimens (both thermoplastic and thermoset) failed near the tab region while the warp loaded specimens invariably always failed near the middle of the gauge section during these tests.

Fig. 11 shows the micro-damages obtained through SEM images under tensile load. The SEM images show the difference in the fracture surfaces in both 3D composites. The smooth fracture surface of thermoset-based 3D-FRC indicates a weak fibre/matrix interface of the epoxy matrix, which transfers all the load to fibres and undergoes sudden failure, see Fig. 11(a)-(c). In contrast, the thermoplastic-based 3D-FRC shows a rough fracture surface with matrix still adhering to the portions of pulled out yarn, indicating a strong interface between glass fibres and thermoplastic matrix see Fig. 11(d)-(e), which leads to the whole yarn-straining instead of individual fibres pulling out, this then leads to relatively higher strain to final failure with fibre pull out failure happening very late in the failure process. The ductile (Fig. 11(f)) and brittle (Fig. 11(c)) interface characteristics observed from micrographs were consistent with the macrographic observation from in-plane tensile test specimens (Fig. 11). These characteristics elucidate the superiority of the tensile strength of novel thermoplastic-based 3D-FRC in comparison with conventional 3D-FRC.

**4.2. In-plane compressive behaviour**

After an initial linear region, both materials show a nonlinear response (Fig. 7). Focusing on the individual stress-strain curves, the thermoset, and thermoplastic 3D-FRCs, one can see a little variation of the stiffness (compression modulus) between warp and weft directions for the linear part of the stress-strain curves for 3D-FRC having the same matrix (i.e., either thermoplastic or thermoset). This was expected due to the similar areal density of glass fibre along both warp and weft directions, i.e., 2550 g.m-2. However, comparing the response of two 3D-FRC having dissimilar matrices, the stiffness of the thermoset 3D-FRC in both linear and nonlinear regions is higher than that of the thermoplastic 3D-FRC, and this has been quantified earlier in the results section. The primary reason for this in the linear region can be attributed to the difference in young’s modulus of the matrix, which for the thermoset epoxy matrix is typically higher than the thermoplastic Elium matrix [47].

In the nonlinear portion of the curves, however, we can observe a progressive drop in the stiffness for both composites, but the drop is neither in the same ratio for cases with different matrices nor for warp and weft directions of cases with a similar matrix. And we see that even for the cases having the same matrices (and thus having very similar modulus in the linear range), the curves start to diverge with increased compressive strain. The fill direction in all cases shows a higher degradation of the compressive modulus but overall higher strain to failure than the warp direction. This can be attributed to the different failure mechanisms in both these directions due to the presence of the z-binder yarn, which runs along the warp weaver but causes a further undulation for the fill yarn (see Fig. 1(a). and Fig. 10(a)). In Fig. 12 and 13, we show some evidence of these different failure mechanisms under compression loading for both types of 3D-FRC and for the different loading directions using digital images and SEM. These images show that the thermoplastic fill-loaded 3DFRC, which had the highest strain to failure at the point of final failure, shows clear evidence of extensive delamination between the warp layers (the z-binder is not effective in preventing this) and some local micro-buckling as well as a brooming failure of fill yarn (see. Fig. 12(c) and Fig. 13(g)-(i)). It does not show much matrix cracking within layers, which is attributed to the higher toughness of the thermoplastic Elium matrix. In comparison, the warp-loaded thermoplastic-based 3D-FRC shows little delamination, more kinking and brooming type failure, and some interlaminar matrix cracks which have not grown into full delamination for the warp loaded specimen (see. Fig. 12(d) and Fig. 13(e) and (f)). During the weaving process of 3D orthogonal woven fabric, warp yarns were kept under tension throughout the process, which results in aligned and straight warp yarns. This high degree of straightness and lesser possibility of delamination between the warp and weft layer due to the binder yarn means that warp loaded specimens retain their stiffness for higher loads, but then at peak loads due to local-kinking and micro-buckling, we get a crushing failure in warp yarns and matrix and a brooming type of failure in fill yarns. Thus, the ultimate load and strain are lower than the fill, loaded thermoplastic 3D-FRC specimens.

On the other hand, the thermoset based 3D-FRC shows no evidence of layer to layer delamination and primarily an inclined shear damage plane for both the warp and fill loaded specimens. In both directions, there is evidence of kink band formation and gross matrix shear at the macro-level (Fig. 12(a)-(b)). This is also verified at micro levels with a greater extent of kink-band fracture as well as micro-bucking in the warp yarns and some matrix shear cracks in fill yarns for the warp loaded specimens (Fig. 13(a) and (b)). Conversely, a greater extent of matrix cracking in the warp-yarns along with some kink-band fracture for the fill yarns in the fill loaded specimens (Fig. 13(c) and (d)). These findings for the thermoset-based 3D-FRC corroborate with the work done by Kuo et al. [64] and Cox et al. [43]. Similarly, this brooming type failure and delamination under in-plane compression were also reported in the previous work by Warren et al. [31] and Dai et al. [5]. However, for liquid resin-based thermoplastic composites, such a detailed exposition of damage modes were not available in the literature.

**4.3. In-plane shear behaviour**

The shear stress/strain curves of both 3D composites highlight four main regions, as shown in Fig. 8(d). The first region in the stress/strain curves is the linear region, where the shear strains increase linearly. The thermoplastic and thermoset composites show linear response up to ~0.7% and ~1% strain (represented by point “a” in Fig. 8(d)), respectively. In the second region, nonlinear shear deformation starts in both 3D composites, which strongly depends on the matrix properties. The nonlinear shear deformation in the thermoplastic and thermoset composites developed between 1.0% to 4% strains and 0.7% to 2.7% strains (point “b” in Fig. 8(d)), respectively. During this phase, the thermoplastic composite shows a 33% higher nonlinear deformation as compared to the thermoset composite. The shear modulus decreased rapidly in the case of thermoplastic composites as compare to thermoset composites (region “a-b”); for example, in thermoplastic composite at 2% strains, the initial value of shear modulus (4.45 GPa) was reduced to 1.8 GPa, whereas, in thermoset composites, it (4.72 GPa) reduced to 2.2 GPa.

 The nonlinear deformation is followed by the linear region. In this region, the shear strain increases linearly (represented by point “c” in Fig. 8(d)) due to the rotation of yarns and carry most of the axial loads. The fibre rotation is also called the scissoring effect (as shown in Fig. 14), which allows the specimen to undergo large deformation. During this phase, the load increases linearly due to large matrix deformation and progressive rotation of fibres and both composites behaviour can be characterised by a secondary reduced shear modulus. The reduction in the shear modulus of thermoset composite in this range (b-c) is slightly more severe than the thermoplastic. Finally, in the last phase, the load-carrying capacity of the composite decreases, and the specimens fail due to z-yarn failure and detachment of surface fill yarns between v-notch and warp yarn breakage, as shown in Fig. 14 (represented by point “e” in Fig. 8(d)).

Although the macro-shear stress-strain curves discussed above show some similarities in both 3D composites show microscopically different failure mechanisms under in-plane shear loads, as depicted in Fig. 14. The primary failure mechanisms in the thermoplastic composites appear to be a z-binder failure on the top and bottom surface between the v-notch region, detachment of surface fill yarn, and rotation of warp yarns. In contrast, the main failure mechanisms in the thermoset composite are a z-binder failure on the top and bottom surface between the v-notch region, matrix cracking, fill yarn failure, rotation of warp yarn, and failure in warp yarn. It is observed that after unloading, the warp yarn in the thermoplastic composite rotated back to their initial position; whereas, in the thermoset composite, the warp yarns permanently deformed (represented by a dashed line in Fig. 14). The micro-damages under in-plane shear are more closely shown in Fig. 15. The samples were cut between the gauge sections A-A and B-B, highlighted in Fig. 14(a) and (b), respectively. Due to the rotation of fibres during the hardening phase, both 3D composites show a different failure mechanism. The thermoset composites undergo interface debonding, matrix cracking, and delamination between yarns due to their weak interface (see Fig. 15(a)-(c)). In contrast, the thermoplastic composite undergoes nonlinear deformation without cracking, while their interface is still intact due to the toughened matrix (see Fig. 15(d)-(f)). This increases the overall load carrying capacity and integrity of the thermoplastic 3D composite.

**4.4. Inter-laminar shear behaviour**

The load/displacement curves highlight that the thermoplastic composites along the fill direction exhibit the highest peak load, whereas it shows the highest displacement before the peak load for the warp loaded specimens. Both warp and fill load thermoplastic specimens show less reduction in the load after damage initiation. After reaching the peak load, thermoplastic composites undergo a slow and stable load drop (see Fig. 9). In comparison, the thermoset composites undergo a rapid, unstable, and higher load drop. This difference in the load/displacement curves is due to different failure mechanisms under a three-point bending load. In the case of warp loaded specimens, the resin-rich pockets, as well as the fill yarns, are under maximum stress (on the top/bottom surface), which undergo plasticization/cracking depending on the matrix (ductile/brittle). Therefore, the warp loaded thermoplastic composite reached its elastic limit (~1.3 kN) at the lowest load and achieved the highest displacement to peak load (1.9 mm) among all composites.

The inter-laminar damage mechanisms were investigated through SEM images. Both 3D composites show significantly different failure mechanisms under transverse load, which are summarised in the schematic diagram, see Fig. 16(a). The main damage modes in the thermoplastic composites were matrix plasticization beneath the loading roller (in yarns and resin-rich pockets), yarn debonding, matrix drawing, and localized stable crack propagation between warp and fill yarn, as shown in Fig. 16(a) and Fig. 16(b)-(d). The stable crack propagation is due to matrix plastic deformation and higher fracture toughness of the thermoplastic matrix [8]. Due to this phenomenon, the thermoplastic-based 3D composites show a gradual and small load drop after reaching the peak value. In contrast, the primary failure modes in the thermoset composites were matrix cracking beneath the loading roller (in yarns and resin-rich pockets), fibre breakage, matrix cracks (intra-laminar cracks) in the yarn at the bottom of the specimen, and a large number of unstable crack propagation (debonding) between warp and fill yarns, see Fig. 16(a) and Fig. 16(e)-(g). The unstable crack propagation is due to its brittle nature and lowers the fracture toughness of the epoxy matrix, which facilitates crack propagation resulting in a rapid and large load drop after reaching the peak load due to fibre breakage. This rapid crack propagation and fibre breakage makes thermoset-based 3D composites unstable and decreases the load-carrying capacity by a large amount (30-40%) immediately after the damage onset.

In each case, the tests were stopped after 2.5 mm of loading roller displacement. It is interesting to note that despite the higher load-carrying capacity of the thermoplastic composites at the end of the test (Fig. 9), once the load was removed and fractography was carried out using SEM (Fig. 16), relatively larger and more prominent interlaminar cracks were seen of thermoplastic 3D-FRC (see Fig. 16(b) and (c)) as compared to the thermoset 3D-FRC which shows relatively closed interface cracks (see Fig. 16(e)-(g)). The reason for this is the plastic (permanent) deformation of the thermoplastic matrix, which means that despite the load removal, the final cracks remained visible. While due to the brittle cracking in the thermoset, although the damage was permanent, the cracks apparently closed after the load was removed.

The SEM images show debonding and crack propagation between warp and fill yarns (Fig. 16). Fig. 16(f) highlights the crack arrest phenomenon in 3D composites due to the presence of through-thickness reinforcement. The delamination cracks between warp and fill yarns were arrested by the z-binder and prevents further crack propagation. The thermoplastic composites show extensive localized matrix plasticization under the loading roller, with no visible damage at the bottom surface (which is in tension). In contrast, the thermoset composite undergoes significant matrix cracking under the loading roller and at the bottom surface of the specimens (along the specimen width) and z-yarn failure (only in warp loaded specimens).

**5. Conclusion**

A comprehensive assessment of the mechanical properties and failure mechanisms of novel resin-infused thermoplastic (Elium®) 3D composite and their comparison with conventional thermoset (epoxy) 3D composite has been presented using both experimental and numerical approaches. The tensile, compression, V-notch shear, and short beam shear tests were conducted, and the resulting strain-strain response was discussed in detail. The mechanical properties and failure and damage mechanisms were also compared in each case. A useful finding is that the thermoplastic 3D composite possesses 15.5% and 21% higher average tensile strength and failure strain, as compared to the thermoset counterpart. While under compressive loads, the performance of thermoplastic 3D composite was, even more, superior with 19% and 46% higher average compressive strength and failure strains, respectively. The in-plane shear behaviour of both 3D composites on the other hand was almost similar initially; however, the thermoplastic composites show higher nonlinear shear deformation and displacement at final failure. Furthermore, in terms of interlaminar shear strength, the thermoplastic composites demonstrate 20% higher strength than the thermoset counterpart. The fractographic analysis of the failure modes through the Scanning Electron Microscope (SEM) assisted in better understanding the mechanical behaviour of thermoplastic 3D composites and it is postulated that the improved mechanical properties of thermoplastic 3D composites are due to superior ductility, fracture toughness and strong fibre/matrix interface provided by the thermoplastic matrix. This study demonstrates that the new thermoplastic 3D composite is a suitable replacement for conventional thermoset 3D composites for composite applications due to their improved mechanical properties and recyclability. An additional benefit of the study is that it presents a reliable set of data in terms of elastic and strength constants, which can be used in the numerical simulation of these novel thermoplastic composites for various applications.

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The authors declare no conflict of interest with respect to the research or publication of this work.

 **Availability of data and material**

The data will be available at appropriate request.

 **Code availability**

The code will be available at appropriate request.

 **Authors' contributions**

**Syed Zulfiqar Hussain Shah:** Conceptualization, Methodology, Formal analysis, Investigation, Visualization, Writing - Original Draft; **Puteri Sri Melor Megat-Yusoff:** Supervision, Investigation, Project administration, Funding acquisition; **Saravanan Karuppanan:** Supervision, Investigation, Writing- Reviewing and Editing

**Rizwan Saeed Choudhry:** Supervision, Writing - Original Draft, Writing- Reviewing and Editing; **Faiz Ahmad:** Funding acquisition, Resources**; Zubair Sajid:** Resources

**Ethics approval**

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 **Consent to participate**

All authors agree with the submission of this manuscript to Applied Composite Materials.

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**Figures and Captions**

**Fig.1.** The 3D orthogonal woven fabric and fabrication process. (a) schematic diagram of 3D orthogonal woven fabric “3D-9871”, (b) vacuum-assisted resin infusion (VARI) process along with the infusion time and (c) fabricated thermoplastic and thermoset 3D composite panels through the VARI process.

**Fig.2.** Specimens along with dimensions, boundary conditions and strain measurement techniques for different mechanical tests. (a) in-plane tensile test specimen according to ASTM D3039 (two strain gauges (SG) were bonded in the gauge section in 0°/90° direction to measure longitudinal and transverse strains), (b) in-plane compression test specimen according to ASTM D6641 (a strain gauges (SG) was bonded in the gauge section in 0° direction to measure longitudinal strains), (c) V-notch shear test specimen according to ASTM D7078 (two strain gauges (SG) were bonded in the gauge section at the back face ( ±45° direction) and Digital Image Correlation (DIC) was used to at the front face to measure shear strains), and (d) short beam shear test specimen according to ASTM D2344 (span to thickness ratio 5).

**Fig.3.** Experimental setup for different mechanical tests performed according to ASTM standards. (a) tensile test, (b) in-plane compression test, (c) in-plane shear test and (d) short beam shear test (20 mm span was used in short beam shear test).

**Fig.4.** Flowchart of volume averaging method

**Fig.5.** Micro- CT and RVE of 3D orthogonal woven composites. (a) micro-CT cross-section of warp yarns, (b) micro-CT cross-section of fill yarns, (c) micro-CT cross-section of z-binder yarns, (d) 3D E-glass orthogonal woven fabric, (e) schematic diagram of 3D fabric along with RVE and (f) RVE of 3D orthogonal woven composites.

**Fig.6.** Comparison of in-plane tensile stress/strain curve, normalized tensile strength, normalized tensile modulus and normalized tensile failure strains of thermoplastic (TP) and thermoset (TS) 3D-FRC along the warp and fill direction. (a) comparison of average tensile stress/strain curve of thermoplastic and thermoset 3D composite, (b) comparison of normalized tensile strength along the warp and fill direction, (c) comparison of normalized tensile modulus along the warp and fill direction, and (d) comparison of normalized tensile failure strain along the warp and fill direction. The data were normalized with the thermoplastic warp loaded 3D composite.

**Fig.7.** Comparison of in-plane compressive stress/strain curves, normalized compressive strength, normalized compressive modulus and normalized compressive failure strains of thermoplastic (TP) and thermoset (TS) 3D-FRC along the warp and fill direction. (a) comparison of average compressive stress/strain curve of thermoplastic and thermoset 3D composite. (b) comparison of normalized compressive strength along the warp and fill direction, (c) comparison of normalized compressive modulus along the warp and fill direction, and (d) comparison of normalized compressive failure strain along the warp and fill direction. The data were normalized with the thermoplastic fill loaded 3D composite.

**Fig.8.** Validation of shear strains measured through strain gauges vs. DIC, comparison of load/deflection curve and shear stress/strain curve. (a) shear strains obtained from DIC measurement (2D shear strain field distribution in the gauge section) and strain gauges, (b) validation of shear strains measurement obtained through DIC. The DIC and strain gauge data matched well up to 1.8% strain, (c) load-displacement curves of thermoplastic and thermoset composites, points “*Pmax*” represent the peak load reached during the test, and (d) shear stress/ shear strain curve of thermoplastic and thermoset composite. The error bar represents the variation in the data at different shear strain values, i.e, 5%, 10%, 15% and 20%. The points “a”, “b”, “c”, ‘d” and “e” represents different stages in the shear stress/strain curves discussed in section.3.3. “*G12*” and “$σ\_{Y}$” represent the shear modulus and shear strength.

**Fig.9.** Comparison of inter-laminar shear strength of thermoplastic (TP) and thermoset (TS) 3D-FRC. The error bar represents the variation in the data at different displacement values, i.e., 1 mm, 1.5 mm and 2 mm.

**Fig.10.** Macro-damage mechanisms in the thermoplastic and thermoset 3D-FRC under tensile load. (a) schematic diagram of 3D composite along with two cross-sections A-A and B-B. (b) damage in thermoplastic 3D composite warp loaded specimen, (c) damage in thermoset 3D composite warp loaded specimen, (d) damage in thermoplastic 3D composite fill loaded specimen, and (e) damage in thermoset 3D composite fill loaded specimen.

**Fig.11.** Micro-damage mechanisms in thermoplastic and thermoset 3D-FRC under tensile load. Figure (a)-(c) shows damage modes in thermoplastic 3D composites. Figure (d)-(f) shows damage modes in thermoset 3D composite.

**Fig.12.** Macro-damages mechanisms in thermoplastic and thermoset 3D-FRC under compressive load (at the side face). (a) damage in thermoset 3D composite fill loaded specimen, (b) damage in thermoset 3D composite warp loaded specimen, (c) damage in thermoplastic 3D composite fill loaded specimen, and (d) damage in thermoplastic 3D composite warp loaded specimen.

**Fig.13.** Micro-damage mechanisms in the thermoplastic and thermoset 3D-FRC under compressive load. Figure (a) and (b) damage modes in thermoset 3D composite warp loaded specimens, (c) and (d) damage modes in thermoset 3D composite fill loaded specimens, (e) and (f) damage modes in thermoplastic 3D composite warp loaded specimen, (g)-(i) damage modes in thermoplastic 3D composite fill loaded specimen.

**Fig.14.** Macro failure mechanisms in the gauge section of thermoplastic and thermoset 3D-FRC under V-notch shear test. (a) failure mechanisms in thermoplastic 3D composites, and (b) failure mechanisms in thermoset 3D composites. Section A-A and B-B are the cutting planes for SEM analysis, shown in Fig.13.

**Fig.15.** Micro-damage mechanisms in 3D-FRC under in-plane shear load. Figure (a)-(c) shows damage modes in thermoset composites. Figure (d)-(f) shows damage modes in the thermoplastic 3D composite.

**Fig.16.** Micro-damage mechanisms in 3D-FRC under short beam shear test. (a) schematic diagram of the failure mechanisms in 3D composites. (b) damage modes in thermoplastic 3D composites fill loaded specimen, (c) damage modes in thermoplastic 3D composites warp loaded specimen, (d) matrix drawing and plastic deformation in thermoplastic 3D composites under out-of-plane bending load, (e) damage modes in thermoset 3D composites fill loaded specimen, (f) damage modes in thermoset 3D composite warp loaded specimens, and (g) brittle shear failure cusps formation due to week interface.

**Tables**

**Table 1.** Physical parameters of the cured panel (average of ten samples)

|  |  |  |
| --- | --- | --- |
| Parameters | 3D thermoplastic FRC | 3D thermoset FRC |
| Thickness (mm) | 4 ± 0.1 | 4 ± 0.1 |
| Fiber volume fraction (%) | 52 ± 1.5 | 52 ± 0.4 |
| Void content (%) | 2.7 ± 1 | <1 ± 0.3 |
| Density (g/cc) | 1.86 ± 0.02 | 1.92 ± 0.01 |

**Table 2.** Elastic constants of fibre and matrix. For fiber $(E\_{11,f}=E\_{22,f}$)

|  |  |  |  |
| --- | --- | --- | --- |
| Material Properties | E-glass fibre [59] | Elium® [65] | Epolam® 5015 [66] |
| Modulus of Elasticity (GPa) | 73 | 3.1 | 3.3 |
| Modulus of Rigidity (GPa) | 29.9 | 1.31 | 1.15 |
| Poisson’s Ratio  | 0.22 | 0.37 | 0.3 |

**Table 3.** Comparison of tensile properties (values in parenthesis represent the coefficient of variance in the data)

|  |  |  |  |
| --- | --- | --- | --- |
| Material/Property | Direction | 3D thermoplastic FRC | 3D thermoset FRC |
| Strength (MPa) | Warp ($X^{T}$) | 478 (3.5) | 450 (4.1) |
| Fill ($Y^{T}$) | 458 (2.4) | 414 (9.7) |
| Modulus(GPa) | Warp ($E\_{11}^{T}$) | 25.4 (2.5) | 26.3 (2.8) |
| Fill ($E\_{22}^{T}$) | 24.9 (3.7) | 26 (2.0) |
| Failure strain (%) | Warp ($ԑ\_{11}^{T}$) | 2.48 (8.8) | 2.23 (5.0) |
| Fill ($ԑ\_{22}^{T}$) | 2.35 (4.3) | 2.05 (6.4) |
| Poisson ratio | $$v\_{12}$$ | 0.13 (8.5) | 0.11 (7.6) |
|  | $$v\_{21}$$ | 0.16 (10.5) | 0.14 (15) |

**Table 4.** Comparison of analytical and experimental results. Value in the parathesis represents the coefficient of variance in the data.

|  |  |  |
| --- | --- | --- |
| Engineering Elastic constants | 3D thermoplastic composite | 3D thermoset composite |
| Experimental | Analytical | Error (%) | Experimental | Analytical | Error (%) |
| Longitudinal modulus “Ex” (GPa) | 25.4 (2.5) | 25.0 | 1.5 | 26.3 (2.8) | 25.6 | 2.6 |
| Transverse modulus “Ey” (GPa) | 24.9 (3.7) | 25.2 | 1.2 | 26.0 (2.0) | 26.1 | 0.4 |
| Transverse modulus “Ez” (GPa) | ------ | 13.8 | ------ | ------ | 13.3 | ------ |
| Poisson’s ratio in xy-plane “vxy” | 0.13 (8.5) | 0.14 | 7.1 | 0.11 (7.6) | 0.18 | 6.5 |
| Poisson’s ratio in xz-plane “vxz” | ------ | 0.33 | ------ | ------ | 0.27 | ------ |
| Poisson’s ratio in yz-plane “vyz” | ------ | 0.32 | ------ | ------ | 0.28 | ------ |
| Shear modulus “Gxy” (GPa) | 4.45 (0.7) | 4.69 | 5.1 | 4.72 (0.6) | 4.82 | 2.5 |
| Shear modulus “Gxz” (GPa) | ------ | 4.69 | ------ | ------ | 4.82 | ------ |
| Shear modulus “Gyz” (GPa) | ------ | 4.69 | ------ | ------ | 4.82 | ------ |

**Table 5.** Comparison of compression properties (values in parenthesis represent the coefficient of variance in the data)

|  |  |  |  |
| --- | --- | --- | --- |
| Material/Property | Direction | 3D thermoplastic FRC | 3D thermoset FRC |
| Strength (MPa) | Warp ($X^{C}$) | 293 (9.6) | 257 (10) |
| Fill ($Y^{C}$) | 357 (2.5) | 300 (7.5) |
| Modulus (GPa) | Warp ($E\_{11}^{C}$) | 27.1 (7.6) | 33.4 (7.2) |
| Fill ($E\_{22}^{C}$) | 26.4 (9.3) | 31 (10.5) |
| Failure strain (%) | Warp ($ԑ\_{11}^{C}$) | 1.21 (5.5) | 0.81 (9.1) |
| Fill ($ԑ\_{22}^{C}$) | 1.70 (10) | 1.16 (8.7) |

**Table 6.** Comparison of in-plane shear properties (values in parenthesis represent the coefficient of variance in the data)

|  |  |  |
| --- | --- | --- |
| Material/Property | 3D thermoplastic FRC | 3D thermoset FRC |
| Shear strength $"S\_{12}" $(MPa) | 40 (1.0) | 44.7 (1.7) |
| Shear modulus $"G\_{12}" $(GPa) | 4.45 (0.7) | 4.72 (0.6) |

**Table 7.** Comparison of ILSS (values in parenthesis represent the coefficient of variance in the data)

|  |  |  |  |
| --- | --- | --- | --- |
| Material/Property | Direction | 3D thermoplastic FRC | 3D thermoset FRC |
| ILSS (MPa) | Warp ($X\_{ILSS}$) | 24.6 (1.6) | 26.9 (4.2) |
| Fill ($Y\_{ILSS}$) | 32.0 (9.0) | 25.8 (5.5) |