Mechanical Characterization of Core Shell Rubber Particles Modified Vinyl Ester and Glass Fiber Reinforced Composites

Swayam Shree[1], Jitendra S. Tate[1,*], and Abhay Deshpande[2]

[1,*]Ingram School of Engineering, Texas State University
601 University Dr, San Marcos, Texas 78666-4616
Phone: 512-245-1826; Email: jt31@txstate.edu

[2]Evonik Corporation, Polymer & Construction Specialties,
7801 Whitepine Rd, Richmond, VA 23237 USA
*Phone: 512-245-4872; Fax: 512-245-7771; Email: JT31@txstate.edu

ABSTRACT

Glass fiber reinforced Vinyl Composites are wide spread application in the marine and tanks manufacturing industry because of their superior chemical resistance and increased strength over polyester resins. However due to the nature of their cure, vinyl ester resins result in brittle composites and suffer dimensional warping after curing process. This limits their applications where components are subjected to impact and cyclic loading. Introduction of rubber content is known to improve the toughness and strain to failure of thermosetting matrices like vinyl ester. Glass fiber reinforced vinyl ester composites will be manufactured with a vinyl ester-based Core Shell Rubber Particles (CSRP) added as a toughener at three different loading levels. To achieve proper dispersion the Core Shell Rubber Particles were added to the resin matrix using a planetary centrifugal mixer. Composite panels were manufactured using Vacuum Assisted Resin Transfer Molding (VARTM). Tensile, flexural, in-plane shear, compression and Interlaminar shear strength properties of the modified composites were evaluated as per the ASTM standards and compared to unmodified composites.

Keywords: Core shell rubber, Fatigue, VARTM, Polymer Matrix Composites

1. INTRODUCTION

Polymer matrix composites are increasingly being used aerospace, architecture, automotive, energy, infrastructure, marine, pipe and tanks and transportation industry [1]. Compared to traditional engineering materials like metal alloys and solid solutions reinforced composites provide better mechanical strength and stiffness [2]. In composite materials the constituent matrix and reinforcement remain distinct and maintain their individual structures therefore, these materials have complex, multi stage failure mechanisms that provides them with higher strength. This leads to lighter load bearing components that have higher longevity. Major adaptors of composite materials are the aerospace and automotive industry [3].

Although, carbon fiber epoxy composites have been researched extensively for their mechanical characterization other polymer composites have not been researched to the same extent. Because of the higher cost of carbon fiber reinforcements their application is only limited to high performance applications where weight reduction is of highest importance. Glass fiber reinforcements are an inexpensive alternative to carbon fibers because of their lower cost [4]. Glass fiber composites have improved compressive strength compared to carbon fibers when used as reinforcement [5].

One disadvantage of glass fibers is that they suffer from stress corrosion [6]. However, when paired with an appropriate matrix this disadvantage can be countered. Marine and wind energy industries account for the highest usage of glass fiber reinforced composites [7]. Vinyl esters are the matrices of choice in these application because of their excellent resistance to water organic solvents and alkalis which helps glass fibers fight stress corrosion. Vinyl ester also provide good resistance against fire. Also because of their lower density vinyl ester composites can replace much heavier metal alloy parts [8].
Vinyl esters are unsaturated polymers that are usually made by reacting an epoxy with monocarboxylic acids. These resins have longer double bonded vinyl groups that provides the resin more flexibility. This enables vinyl ester composites to withstand impact deformations and repeated flexing without forming cracks. Because of the double bonded vinyl group these resins have roughly twice the toughness of polyester resins. However, vinyl esters react and build molecular weight to a solid waxy resin that is not usable. Therefore, styrene is added to these polymers to keep the resin in a liquid state [9].

Even though vinyl esters have higher flexibility compared to epoxies and polyesters, vinyl ester composites still are brittle in nature. The double bonded nature also creates issues during curing which results in shrinkages during curing process [10]. Extensive research has been done to find methods to increase the toughness of polymer composites. Research shows that introduction of a rubber medium like liquid rubber[11], [12] and core shell rubber particles [13] improves the toughness and percentage elongation of reinforced composites. Core shell rubber particles are a relatively new additive. Core shell rubber particles (CSRP) are spherical with one copolymer surrounding another. Inner copolymer is mainly-butadiene/styrene, polybutadiene, siloxane, acrylic, etc. Outer shell is made up of polymers that are compatible with the resin it is being added to. Unlike liquid rubber like CTBN which goes through a phase separation during the curing process [14] core shell rubber particles are preformed particles. Therefore, to achieve the same amount of toughness less amount of core shell rubber particles is needed in comparison to liquid rubber [15-17].

Most of the efforts into rubber toughening of matrices has focused on epoxies and not a lot of research has been done into the effect of core shell rubber particles on vinyl ester composites. This research is conducted to study and characterize the effect of core shell rubber modified vinyl ester-glass fiber composites which will ultimately lead to development of material systems more suited for marine, wind turbine and pipe and tank industry for parts that have improved impact and equilibrium toughness and with higher longevity under fluctuating load conditions.

1.1 Matrix

For this research Derakane 510A-40 from Ashland was selected as the polymer matrix. The resin is a brominated epoxy vinyl ester resin that provides high degree of chemical resistance and fire retardance. The bromine content of the resin provides better toughness and fatigue resistance in comparison to standard epoxy vinyl ester resins. It has a low viscosity of 400 mPas which makes it suitable for lay-up, pultrusion and resin transfer molding processes [18].

1.2 Curing Agents

Two curing agents were selected for this research. The first one is Luperox® DDM-9 from Sigma Aldrich which is a 35 wt% solution of Methyl ethyl ketone peroxide (MEKP) in trimethyl pentanediol diisoburate. MEKP initialtes the crosslinking of unsaturated bonds in vinyl esters [19]. The presence of a diisoburate helps in reducing the sensitivity to shock of the MEKP. The second curing agent used was 6% cobalt Napthenate from Sigma Aldrich. It as the advantage of being soluble in non-polar substrates and acts as a catalyst to the crosslinking reaction initiated by MEKP [20].

1.3 Reinforcement

The reinforcement for this research was $0^0/90^0$ stitch bonded non-crimp glass fabric from Saertex. These fibers are suitable for Resin Transfer Molding, Compression and pultrusion processes and are compatible with vinyl ester resin systems. Non-crimp fabrics have greater load bearing capacity compared to woven fabrics. Composites manufactured using these resins facilitate ideal transmission of component of generated forces and therefore provide higher component loading when compared to standard reinforcement textiles. The $0^0/90^0$ orientation provides strength in both axial and transverse direction as compared to uniaxial oriented fibers that provide strength in only one direction [21].

1.4 Additive

The additive chosen for this research was Albidur VE 3940 from Evonik industries. This additive is a 40% concentrate of high-performance elastomer that are based on vinyl esters. The elastomer is present in the form of core shell rubber particles that have a size of around 3 micrometers. The addition Albidur VE 3940 improves toughness with minimal loss in modulus and glass transition temperature [22].

2. MANUFACTURING

2.1 Matrix Manipulation

The Planetary Centrifugal Mixer THINKYTM ARV-130 was used to disperse the additive and mix the curing agents with the matrix and eliminate sub-micron level air bubbles simultaneously. The THINKYTM ARV-130 keeps the material container at a 45º angle and provides both rotational and revolutionary action under vacuum pressure reduction. The revolution speed is set at twice the rotational speed of the cup holder [23].

In this experiment, four different composites were made. One was a control composite that had no core shell rubber particles, one had 2 wt% core shell rubber particles added, one had 5 wt% core shell rubber particles added, and the final one had 10 wt% core shell rubber particles added to it. To manufacture these composites, first the appropriate amount of core shell rubber particles was

added to the vinyl ester resin and mixed using the planetary mixer at 2000 rpm under 4kPa vacuum for 5 minutes. Then, one by one the curing agents were added, and hand stirred to prevent any unstable exothermic reaction and again mixed in the planetary mixture at 2000 rpm under 4kPa vacuum for 5 minutes.

2.2 Fabric Impregnation and Composite Curing

15"x15" composite panels were fabricated by impregnating a stack of four layers of $0^o/90^o$ stitched fabric with the resin systems by using vacuum assisted resin transfer molding. The resin line was passed through the center of the panels while two vacuum lines were placed of each side of the fabric stack parallel to the resin line. VARTM was chosen as the preferred manufacturing method because of its ability for consistent manufacturing of larger parts at a relatively lower cost and the good control over fiber-volume fraction it provides [24]. VARTM is also an energy efficient manufacturing process that provides lowest porosity on composites that result in parts with high flexural strength [25], [26]. After complete impregnation the composites were allowed to green cure at room temperature for 24 hours. After this the composites were removed from the mold and post cured in a programmable oven for six hours at 80^0C.

2.3 Sample Preparation

After the panel had cured samples for mechanical testing were cut from the composites using a abrasive water jet cutting machine. Water jet cutting avoids edge delamination if the composites. The tensile samples were sanded at the edges using a Dremel and tabbed using a glass fiber epoxy composite that had an average thickness of 1.2 mm. The tabs were attached to the tensile test samples using scotch weld epoxy adhesive. Tabbing was done to ensure failure during tests occurred only in the gage area.

3. TESTING

3.1 Static Mechanical Testing

Specimen from all four composites were tested for their tensile, compressive, flexure, shear and inter laminar shear strength. All the tests were conducted according to appropriate ASTM standards for composites. All tests were done on the MTS 810 Material Testing System. The samples varied in thickness from 3.0 mm to 3.4 mm.

Tensile tests were conducted according to ASTM D3039 – "Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials" [27]. The samples were then tabbed to ensure failure occurred in the gage area. These tabs were made from a fiberglass composite material and bonded to the end of the samples using Scotch-Weld Epoxy Adhesive DP 460 Off-White. An axial extensometer was used to measure axial strain. The ultimate tensile strength (UTS) and modulus for each sample was calculated from the collected data. Flexural tests were conducted according to ASTM D7264 – "Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials" [28]. The flexural strength and modulus of the samples was calculated from the collected data. The short beam test was done according to ASTM D2344 – "Standard Test Method for Short-Beam Strength of Polymer Matrix Composite Materials and Their Laminates" [29]. Inter-laminar shear strength was evaluated for the samples using the collected data. The shear test was conducted according to ASTM D7078 – "Standard

Test Method for Shear Properties of Composite Materials by V-Notched Rail Shear Method" [30]. The in-plane shear strength was calculated from the collected data. The compression test was done according to ASTM D6641 – "Standard Test Method for Compressive Properties of Polymer Matrix Composite Materials Using a Combined Loading Compression (CLC) Test Fixture" [31]. The compressive strength was calculated from the collected data.

4. RESULTS

4.1 Tensile Test Results

The tensile strength and tensile modulus have been presented in Figures 1 and 2, respectively. With the addition of core shell rubber particles, the tensile strength and modulus have decreased. However, it was observed that the 2 wt% panel had the worst results and the properties improved with the 5 wt% sample but again decreased in the 10 wt % samples. However, the control specimen had the best properties among all the panels. The control had a tensile strength of 473 MPa, the 2wt% specimen had a tensile strength of 328 MPa, the 5 wt% panel had a tensile strength of 440 MPa and the 10 wt% specimen had a tensile strength of 421 MPa. The modulus had the same trend with the control, 2 wt%, 5 wt% and 10 wt% specimen having modulus 23.43 GPa, 15.20 GPa, 21. 59 GPa and 20.99 GPa, respectively. Tensile strength and modulus decreased for all the core shell rubber modified composites.

The same trend was observed in the percentage elongation as the core shell rubber content was progressively increased. The control composites had a 2.61% elongation at break. The 2 wt% composites had a 2.34% elongation at break. The 5 wt% panel had a 2.46% elongation at break and the 10 wt% specimen had an elongation at break of 2.37%. These results are displayed in Figure 3.

4.2 Flexural results

Figures 1 and 2 display the results for flexural strength and modulus. Flexural properties decreased with the addition of core shell rubber particles. The control, 2 wt%, 5 wt% and 10 wt% specimen had flexural strengths of 743 MPa, 113 MPa, 500 MPa and 746 MPa respectively. It is noteworthy that as the concentration of core shell rubber particles increased from 2 wt% to 10 wt% the flexure strength increased as well with the 10 wt% panel outperforming the control panel. The control, 2 wt%, 5 wt% and 10 wt % panels had modulus of 22.5 GPa, 5.7 GPa, 17.3 GPa and 16.2 GPa, respectively. The 5 wt% panel performed the best among all modified composites, but the control had the highest modulus

4.3 Shear results

The results of the V notch In-plane rail shear test have been presented in Figure 4. Addition of core shell rubber particles had a detrimental effect on the shear property of the composites. Like tensile and flexural tests, the 2 wt% specimen had the worst property and the control had the best. The shear strength of the control, 2 wt%, 5 wt% and 10 wt% specimen was 58.22 MPa, 26.58 MPa, 54.7 MPa and 55.16 MPa, respectively.

4.4 ILSS results

The results of the short beam test have been presented in Figure 5. Addition of core shell rubber particles led to a decrease in the property of the composites with 2 wt% specimen having the worst properties and control specimen having the best properties. The control, 2 wt%, 5 wt% and 10 wt% panels had inter-laminar shear strength of 51.75 GPa, 35.88 GPa, 48.13 GPa and 46.06 GPa.

4.5 Compression test results

The results of the compression test have been presented in Figure 6. Addition of core shell rubber particles led to the decrease in compressive strength of the composites with 2 wt% specimen having the worst property and control having the best. The control, 2 wt%, 5 wt% and 10 wt% specimen had compressive strength of 247, 109, 229 and 219 MPa, respectively.

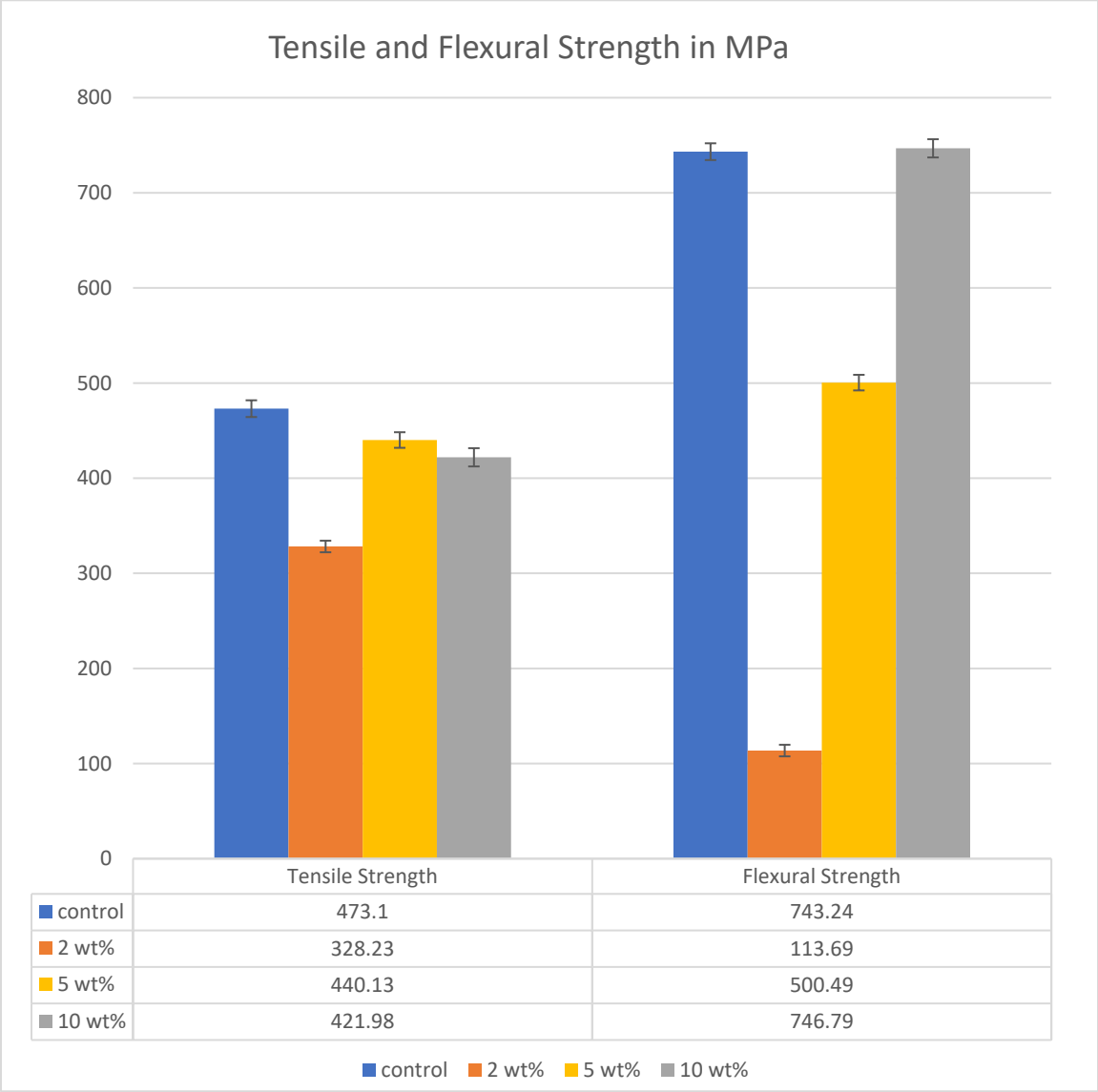

Figure 1: Results of tensile strength and Flexural Strength

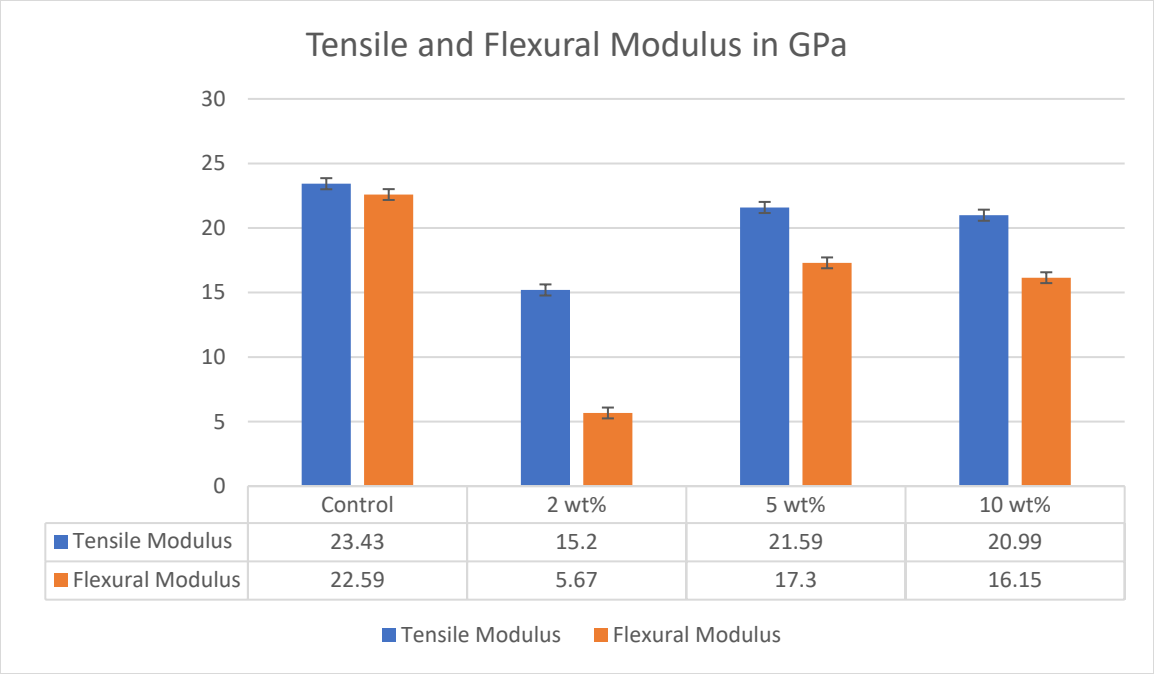

Figure 2: Results of Tensile and Flexural Modulus

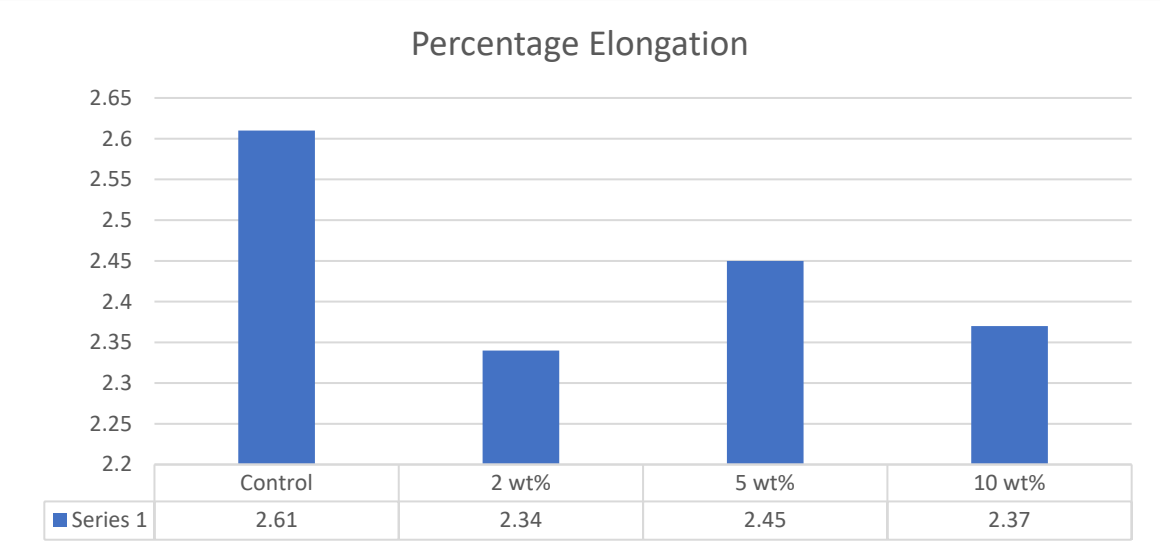

Figure 3: Percent elongation at break

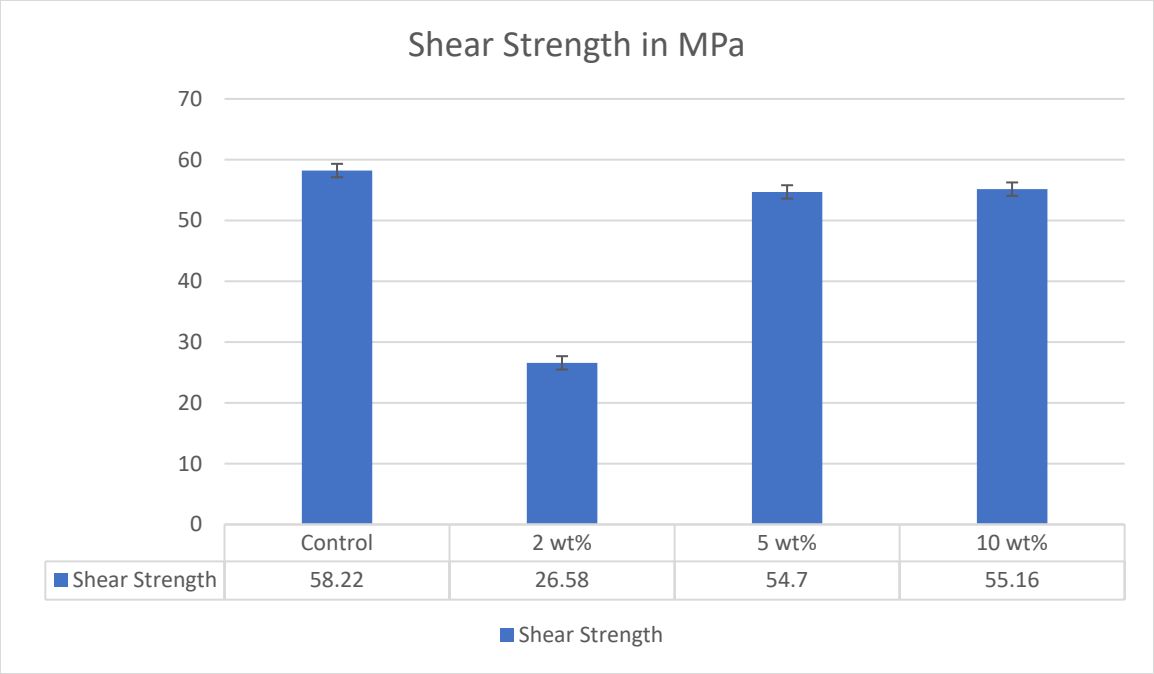

Figure 4: Result of V notch Rail shear test

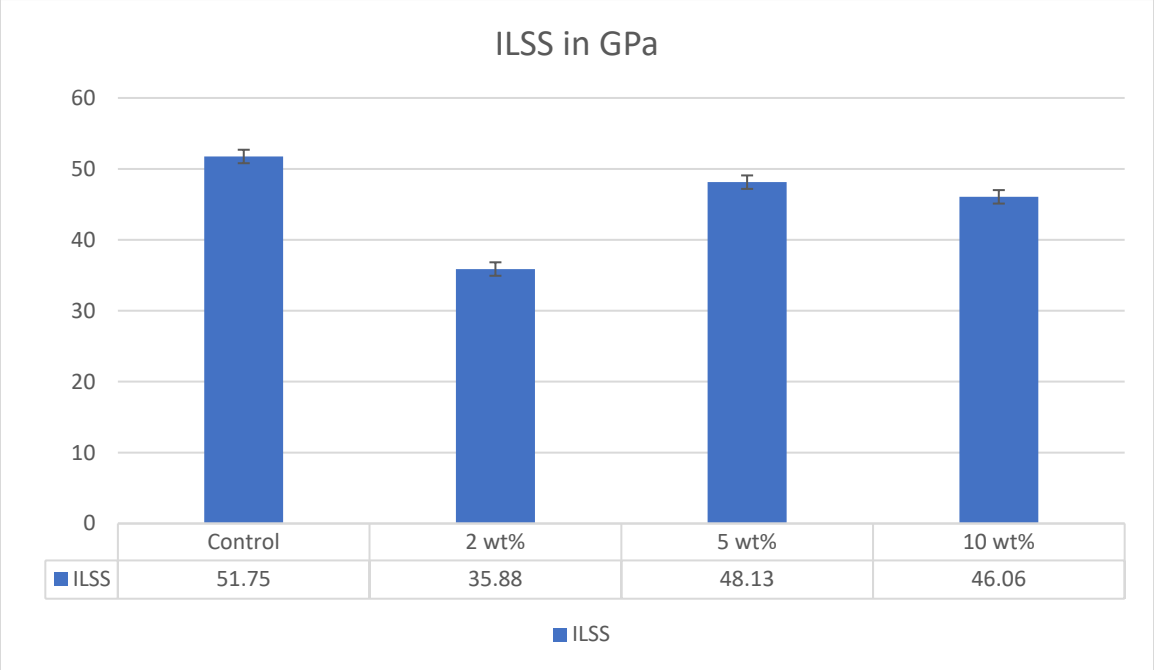

Figure 5: Result of Short Beam Test

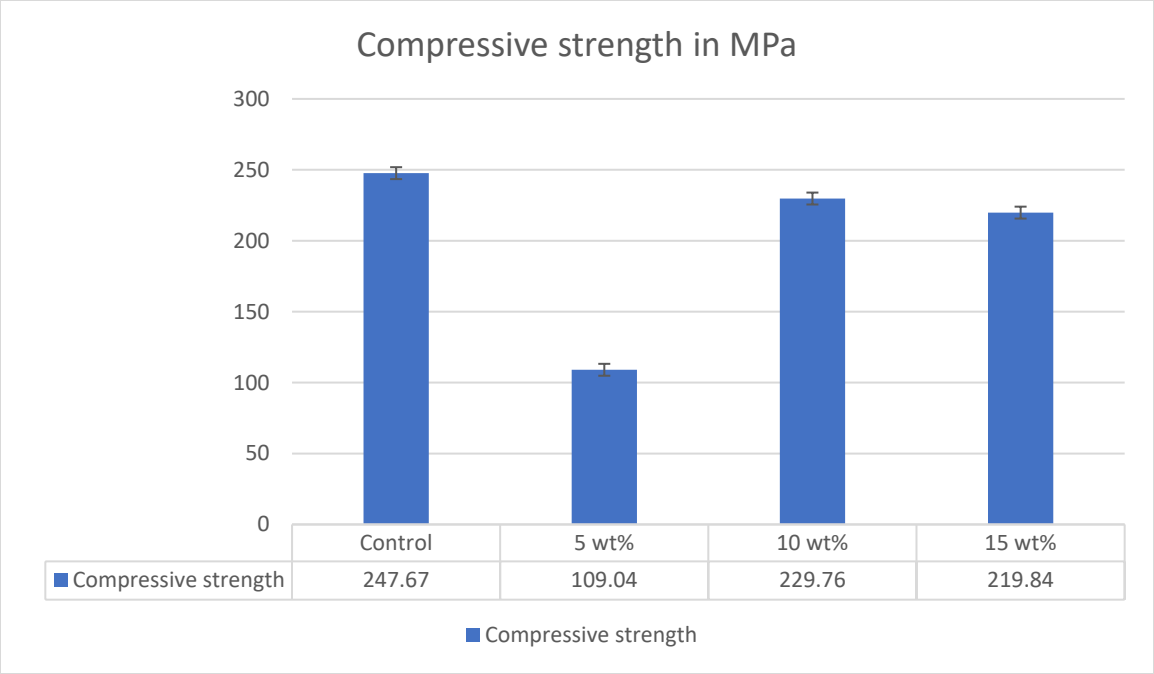

Figure 6: Result of compression Test

Panel / Property	Tensile Strength (MPa)	Tensile Modulus (GPa)	Flexure Strength (MPa)	Flexure Modulus (GPa)	Elongation at Break (%)	Shear Strength (MPa)	ILSS (GPa)	Compressive Strength (MPa)
Control	473.1 (8.8)	23.43 (0.43)	743.24 (13.5)	22.59 (0.42)	2.61 (0.048)	58.22 (1.1)	51.75 (0.95)	247.67 (4.2)
2 wt%	328.23 (6.1)	15.20 (0.39)	113.69 (9.2)	5.67 (0.39)	2.24 (0.046)	26.58 (0.76)	35.88 (0.62)	109.04 (2.9)
5 wt%	440.13 (8.2)	21.59 (0.41)	500.49 (14.1)	17.30 (0.41)	2.45 (0.05)	54.7 (1.2)	48.13 (1.1)	229.76 (4.5)
10 wt%	421.98 (9.6)	20.99 (0.45)	746.79 (15.3)	16.15 (0.40)	2.37 (0.051)	55.16 (0.9)	46.06 (1.02)	219.84 (4.3)

Table 1: Results of static mechanical Test

The standard deviations are in parenthesis

Addition core shell rubber particles led to a decrease in mechanical properties. This is expected as core shells are a rubber additive and addition of rubber leads to a decrease in strength and modulus. However, rubber addition toughens the composites which leads to an increase in the fracture toughness. The nature of core shell rubber particles provides a three-stage toughening. First the shell debonds from the matrix, this is followed by cavitation of the inner copolymer and finally the debonding of the core from the shell occurs, but experimental analysis has to be done to provide conclusive evidence of this.

In the tests the composite panel with 5 wt% CSRP had the least knock off in properties. At higher loading levels there might be some agglomeration of the particles which might explain the

decrease in strength and modulus of the 10 wt% composite panel. Results of flexure tests have a higher standard deviation compared to the rest of the tests. This might be because during bending the forces are concentrated in a small region. Composites like all brittle materials are very sensitive to localized defects. Therefore, depending on where the failure begins during tests different samples might fail at different loading levels.

5. CONCLUSIONS

Four different glass fiber reinforced vinyl ester composite panels were made with 0 wt%, 2 wt%, 5 wt% and 10 wt% of core shell rubber particles added to the resin system. Tensile, flexural, compressive, shear and short beam tests were carried out on samples cut from these composites. Overall 5wt% core shell rubber particle modified composite had the least degradation in static mechanical properties compared to 2wt% and 10wt% Core shell rubber particle modified composites. The 2 wt% panel had the worst static mechanical properties. Addition of rubber into epoxy matrices improves the toughening of the composites but it comes at the disadvantage of a reduction in elastic modulus. At a low loading level of 2 wt% it might have been the case that almost all of the core shell rubber particles led to plasticization therefore leading to the worst results.

The future work includes qualitative analysis of dispersion of core shell rubber particles in the composites using scanning electron microscopy and analyze damage mechanism in the composites using SEM. SEM may also be utilized to determine what is the dominant toughening mechanism of the core shell rubber particles.

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