EFFECTS OF SURFACE TREATMENT OF ALUMINA NANOFIBERS ON MECHANICAL AND THERMAL PROPERTIES OF POLYESTER E-GLASS COMPOSITES

Harish S.P. Kallagunta[1,2], Jitendra S. Tate[1,2,*], Gary W. Beall[1,3], Subash Panta[2], Maria Beldeque[2], Muzaffer Akanda[2], Rafiqul Islam[2]

Texas State University 601 University Drive, San Marcos, TX, 78666
[1]Materials Science, Engineering and Commercialization Program, Texas State University
[2]Ingram School of Engineering, Texas State University
[3]Department of Chemistry and Biochemistry, Texas State University
JT31@txstate.edu; 512-245-1826

ABSTRACT:

Dispersibility of the nanoparticles in the matrix is one of the critical factors influencing various properties of nanocomposites. To address the issue of agglomeration in nanocomposites and negate the effects on properties, surface treatment of nanoparticles is seen as the viable solution. This study is focused on the influence of alumina nanofiber (Nafen^{TM}) loadings and surface treatment of the alumina nanofibers (Nafen^{TM}) on thermal and mechanical properties of polyester/E-Glass composites. Loading levels of 0.25wt%, 0.5wt%,0.75wt% and 1wt% for both untreated alumina nanofibers and silane surface treated alumina nanofibers were selected and are dispersed into polyester resin using three-roll mill. Transmission Electron Microscopy (TEM) revealed that at higher loading levels, the alumina nanofibers tend to agglomerate. Thermogravimetric analysis was performed on the unreinforced nanocomposite coupons and the increase in thermal degradation behavior with addition of nanoparticles. Silane treatment of alumina nanofibers have seemed to improve the lower onset degradation temperature. Static mechanical studies were performed. The mechanical behavior improved with addition of alumina nanofibers. Major improvement in properties is observed at 0.25wt% and 1wt% silane treated alumina nanofiber modified glass polymer composites.

1. INTRODUCTION

Glass fiber composites have become cost-effective solution in the industrial composite market against high cost carbon fiber composites which are limited to aerospace applications. Unsaturated polyester resin infused glass fiber composite due to its good mechanical performance and low cost have gained prominence. Addition of nanoparticles to these composites have proven to be more effective in enhancing the mechanical performance without drastically increasing the cost. It was noticed that ceramic nanoparticle inclusion into thermosetting polymers improves the resistance of composites to wear, self-cleaning, self-healing and good adhesive property [1, 2]. Ionic and covalent bonding characteristic of ceramic nanoparticles offers good thermal conductivity, electrical resistivity and low dielectric constant, which are major concerns for miniaturization and high computing speed [3-5]. Nanoscale precipitation and point defects introduction through incorporation of nanoparticles can provide excellent thermal insulation properties which find applications in subsea, construction and piping industry[6, 7]. The nano-porosity and electrostatic attraction offered by alumina nanofibers with high surface areas is seen as a great potential for water treatment in order to reduce the contaminants through adsorption. However, much research is needed in order to assess its impact on environment and human health [8, 9]. Alumina nanofibers can find applications where high thermal stability is desired. The high thermal stability often can

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lead to improved fire resistance and char formation offering thermal protection to composites at affordable cost.

Glass fiber reinforced polymer nanocomposites have been researched for a variety of applications that require impact resistance, flame retardancy, and improved fatigue performance. Research utilizing different nanoparticles to enhance the mechanical properties have been of focus in designing for improved fracture toughness. Silica nanoparticles are studied for their effectiveness in increasing the mechanical properties when added to the glass fiber reinforced epoxy composites and are relatively inexpensive, nontoxic, biocompatible, highly thermal resistant and effective as mechanical reinforcements[10]. In another study Tate et al [11] investigated the influence of 6, 7 and 8wt% nanosilica on glass fiber reinforced epoxy composites in static mechanical and fatigue loading. It was observed that 6wt% nanosilica loading has greater effect on tensile strength, percentage elongation and interlaminar shear strength. 6wt% loaded nanosilica epoxy glass fiber composites showed a modulus loss of 45% as compared to 65% modulus loss of neat specimen. In another study by [12],[13] 20wt% and 25wt% loading of nanosilica, the Youngs modulus showed an increase of 23% and 78% respectively. Rostamiyan's research group in their study compared the effects of Nano-clay and Nano-silica on flexural properties. The group found that the hybridization of glass fiber with epoxy, nanosilica and nanoclay showed promising results with little variance [14]. Addition of other micro particles along with the nanomaterials has also been studied to evaluate their effect on the impact strength and interfacial adhesion of the nanocomposites. Tate et al, conducted experiments on glass fiber epoxy composites modified with 6.5wt% rubber microparticles in combination with 8.1wt% Nano-silica and reported an effective impact energy dissipation to surrounding area [15]. Nano alumina with particle morphology are also studied by various researchers. Zheng et al [16] reported that enhancement in impact strength by 84% is observed with addition of 20wt% alumina nanoparticle loading. Afrouzian et al [17] studied the effects of nanosilica on the tensile, flexural and ballistic impact properties of glass fiber epoxy composite laminates and found significant effect on mechanical behavior of the composite laminates under study. The virgin specimens and composites modified with loadings of 0.5wt%, 1wt% and 3wt% of nanosilica are compared to each other and lowest loading of nanosilica is found to outperform the remaining by 47%. Alumina nanofibers which are relatively new form of nanomaterials has very limited studies performed on their effectiveness in strengthening the polymer matrix for fiber reinforced composite materials. Some of the studies conducted on alumina nanofibers are: Mechanical, thermal stability and flammability characteristics of PA6 thermoplastics modified with alumina nanofibers by Wu et al.,[18] reported a significant effect of alumina nanofiber modification on the mechanical properties although there is no change in thermal and flammability properties. Rakitin.A and Grodensky [19], reported an increase in mechanical property by 72% by adding alumina nanofibers to the epoxy resin. Previous studies have indicated positive effect of alumina nanofiber in glass fiber epoxy composites under low impact loading at 2.5wt%[20]. Silane surface treatment using chloro-silane proved ineffective and inhibited ballistic impact property at 0.25wt% and 0.5wt% of silane treated alumina nanofiber loading[21].

The objective of the present study is to evaluate the mechanical performance of alumina nanofiber modification of unsaturated polyester reinforced with woven E-glass fibers. In addition to low density and fiber morphology which offers the benefits of tailoring the aspect ratio, it has been considered economical than other forms of ceramic nanoparticles in terms of manufacturability and monetary cost. Alumina nanofiber at four different loading levels (0.25wt%, 0.5wt%, 0.75wt%

and 1wt%) is studied to evaluate the variation in static properties. The effect of surface treatment of alumina nanofibers on the thermal stability and dispersion is studied along with its effect on the fracture behavior of the glass polyester composite material system.

2. MATERIALS AND MANUFACTURING

2.1 Material System

The material system used for this study comprises of 0/90 woven E-Glass fabric from Owens corning having GSM 888.0 as the reinforcement and unsaturated polyester resin SIL47DA-2949 with a viscosity of 396 cP from Interplastic Corporation as the matrix. Alumina nanofibers with minimal aspect ratio 18-20 under trade name Nafen^{TM} from ANF Technology are used as nanofillers. We use BPO as the curing agent with heat as an external stimulator. One set of composites were modified with untreated alumina nanofibers and the second set were modified with vinyl silane coupling agent treated alumina nanofibers. Loading levels of 0.25wt%, 0.5wt%, 0.75wt% and 1wt% were used for both sets of composites laminates. The glass polyester composites modified with untreated alumina nanofibers are here from referred to as UT-ANF/GPC and the composites modified with vinyl silane treated alumina nanofibers as VT-ANF/GPC.

2.2 Fabrication of Laminates

2.2.1 Dispersion using calendaring approach

Three roll milling was used for dispersing Nafen^{TM} into the polyester resin. In every pass, by adjusting the gap setting between the rollers a high shear force is achieved to reduce the clumps and clusters formed. The gap between the rollers is set at 25µm, 15µm, and 7.5µm respectively for three passes. Vinyl silane coupling agent treated alumina nanofibers are also dispersed using the same approach.



Figure 1: Three roll mill and illustration of dispersion using calendaring approach (Courtesy: www.exaktusa.com)

2.2.2 Fabrication of composite laminates and sample extraction

The E-glass fabrics were passed through a resin bath which allowed uniform wetting of the fabric. Fourteen layers were stacked and consolidated by using a pressure of 1.43 MPa and a platen heat of 82°C in the hydraulic compression press. The laminate was then post-cured for three hours at 110 °C in the programmable oven. Samples are cut according to the ASTM standards using a water jet to prevent edge delamination.

3. EXPERIMENTATION

3.1 TEM Imaging

In order to evaluate the dispersion state of alumina nanofibers in the polyester, Transmission Electron Microscopy was performed on the polymer coupons. The thin sections of polymer modified with alumina nanofiber were obtained using ultramicrotome which were placed on copper grids. The grids were then viewed in a high-resolution transmission electron microscope.

3.2 Thermogravimetric (TGA) Analysis

TGA analysis was performed to at 10°C/min ramp rate from room temperature up to 600°C in nitrogen atmosphere using a Discovery SDT650 from TA instruments. An air flow rate of 50mL/min was used.

3.3 Static Mechanical Testing

The static testing on the untreated and vinyl treated Alumina nanofiber (ANF) modified Glass Polyester Composites (GPC) is performed using MTS 810 Servo Hydraulic System. Table 1 gives an illustration of the static tests performed on the Glass Polyester Composites (GPC) modified with untreated and surface treated Alumina nanofibers (ANF).

Table 1: Static test standards and parameters performed on ANF-GPC

ASTM Standard	Property	Sample Dimensions	Loading Rate
D 3039	Tension	10in x 1in x 't'	2 mm/min
D 7264	Flexure	5.5in x 0.5in x 't'	1 mm/min
D 2344	Short Beam Shear Test	6t x 2t x t	1mm/min

3.4 SEM Imaging

SEM imaging was performed using Helios Nanolab 400 to analyze the failure modes at 10.0kV and a current range between 0.69amp and 2.7amp. The samples were previously treated with EMS150T ES sputter coater to add a layer of carbon of at least 20nm.

4. RESULTS AND DISCUSSION

4.1 TEM Analysis

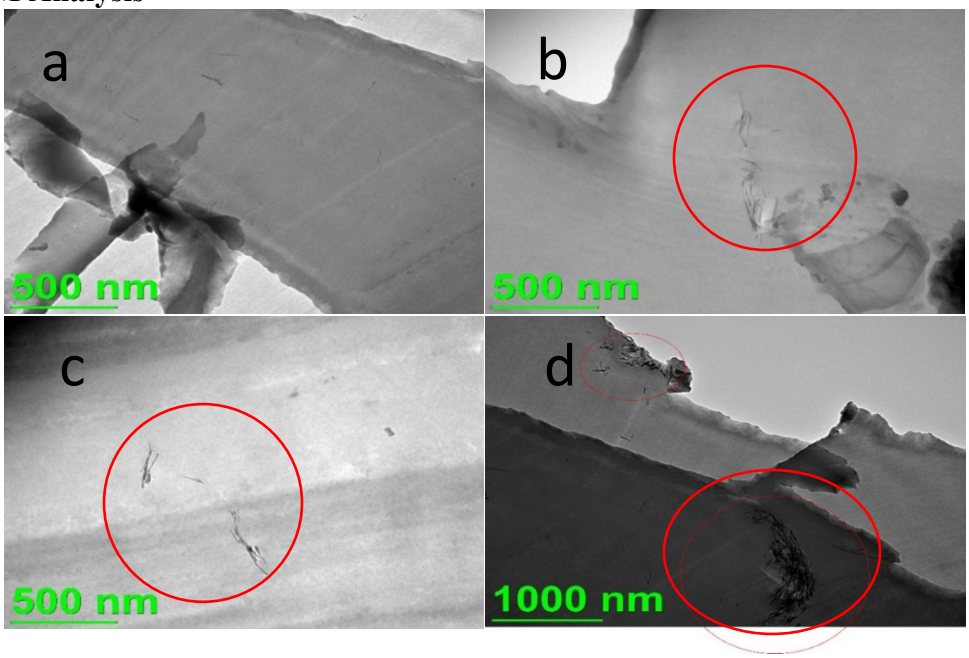


Figure 2: TEM images showing dispersion of Untreated ANF in UPE at loading levels of (a) 0.25wt%, (b) 0.5wt%, (c) 0.75wt% and (d) 1wt%

The TEM images revealed agglomeration of ANF nanoparticles in UPE at higher loading levels, whereas a decent quality of dispersion of ANF nanoparticles in lower loadings is observed. It was observed that ANF nanoparticles retained their fiber like morphology even after subjecting to high shear forces via three roll milling. Although individual ANF nanoparticles were visible, it was observed that the fibers are not completely de-bundled and distributed. The lack of de-bundling might be because of absence of any surface treatment. It would be interesting to see the effect of surface treatment of ANF on the de-bundling and distribution aspect of the nanoparticles during dispersion.

4.2 Thermogravimetric (TGA) Analysis

The TGA results on the formulations are reported in fig 3 and the results are as expected from addition of ANF. As compared to neat polyester, ANF modified polyester showed increase in thermal stability. The weight loss from the TGA curves of the vinyl silane surface treated is more than the untreated silane surface treated, as this is due to the combustion of the silane agent. The decomposition temperatures calculated from the thermographs are summarized in table 2. With the increase of loading content, the peak mass loss temperature increased. The lower onset degradation temperature was enhanced with surface treatment of alumina nanofiber due to promoted bonding between polyester resin and VT-ANF. However, the degradation temperature at higher temperatures as indicated by 50wt% mass loss temperature is common in polymer systems.

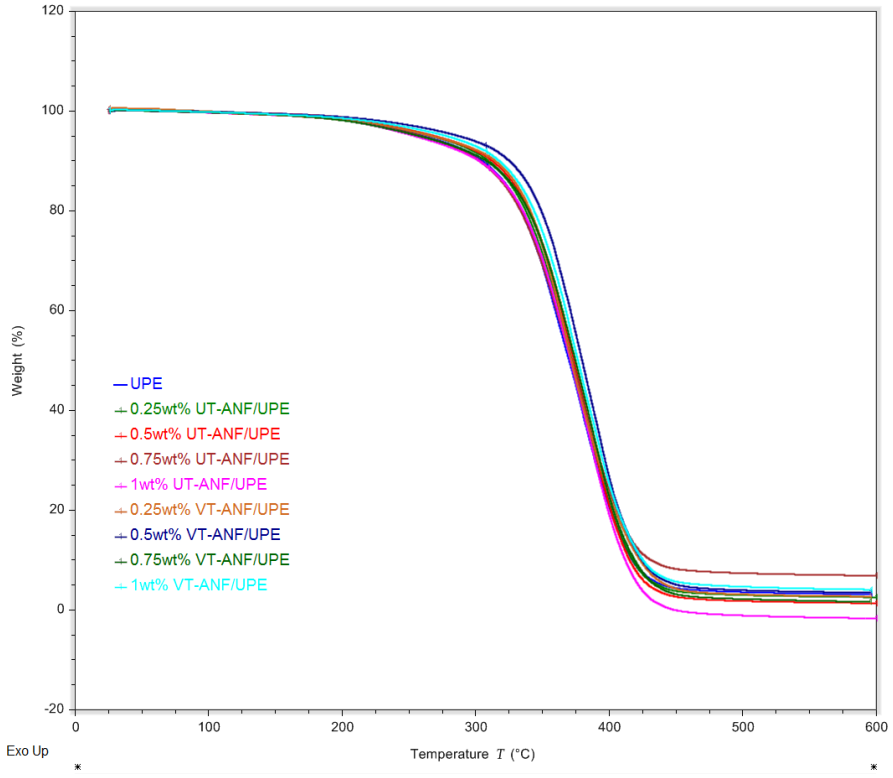


Figure 3: TGA thermographs showing dispersion of Untreated ANF in UPE at loading levels of (a) 0.25wt%, (b) 0.5wt%, (c) 0.75wt% and (d) 1wt%

Table 2: Summary of decomposition temperatures and residual mass

Sample	T_{dec} 10wt%		T_{dec} 50wt%		Peak mass loss temperature	
	°C		°C		°C	
	UT-ANF/UPE	VT-ANF/UPE	UT-ANF/UPE	VT-ANF/UPE	UT-ANF/UPE	VT-ANF/UPE
0wt%	305.30		370.24		374.65	
0.25wt%	309.94	315.22	372.35	371.92	382.03	373.05
0.50wt%	313.37	327.12	374.09	380.05	384.71	393.15
0.75wt%	303.32	308.61	372.11	374.76	381.37	393.84
1.00wt%	307.29	319.20	370.91	376.83	385.44	392.28

4.3 Static Mechanical Properties

A summary of mechanical test results on the glass polyester composites modified with untreated and vinyl silane treated ANF tabulated in table 3 and the percentage improvement in properties is shown in table 4. The ANF modification proven to enhance the mechanical properties even if it is untreated or vinyl silane surface treated. It seemed the addition of silane treated alumina nanofibers to the glass polyester composites had shown no greater change and the improvement is consistent at all the loading levels. However, with the untreated alumina nanofiber modified composites there seem to be a trend. The tensile properties improved at 0.25wt%, seen a decrement at 0.5wt% and 0.75wt% and then improved at 1wt%. Similar behavior is observed in flexure properties.

As observed from the percentage improvement in properties as summarized in table 4, the maximum of improvement in properties is seen in vinyl silane treated alumina nanofibers modified glass polyester composites, with all the properties peaking at 0.25wt% and 1wt% loading level of VT-ANF/GPC.

Table 3: Results of static mechanical test of ANF/GPC (Standard deviations are in parenthesis)

Tests	Control	0.25 wt%		0.5wt%		0.75wt%		1wt%	
		UT	VT	UT	VT	UT	VT	UT	VT
Tensile Strength (MPa)	410.82 (28.51)	521.32 (19.11)	507.45 (11.88)	506.25 (14.88)	508.23 (8.18)	453.18 (16.02)	520.35 (0.223)	524.60 (5.56)	520.44 (7.26)
Tensile Modulus (GPa)	18.62 (2.86)	20.64 (0.54)	22.69 (1.51)	17.52 (0.64)	25.25 (2.29)	18.87 (1.41)	23.24 (1.92)	21.78 (0.32)	24.88 (1.57)
% Elongation	6.72 (0.50)	4.96 (0.41)	3.03 (0.79)	4.09 (0.18)	2.90 (1.08)	4.63 (0.22)	1.86 (0.14)	5.43 (0.73)	5.50 (1.98)
Equilibrium Toughness (MJ/m^3)	7.08	10.29	10.22	10.02	10.07	8.70	10.44	10.24	9.95
Flexure Strength (MPa)	112.52 (17.14)	117.11 (14.91)	126.18 (13.05)	93.67 (3.44)	119.45 (3.10)	115.51 (12.86)	121.60 (12.25)	133.53 (14.87)	129.69 (3.47)

Flexure Modulus (GPa)	22.82 (7.59)	24.46 (2.36)	28.02 (4.98)	21.25 (1.05)	26.20 (1.88)	23.21 (1.51)	27.98 (4.05)	29.24 (1.56)	30.98 (2.79)
Short Beam Strength (MPa)	12.61 (0.85)	13.40 (1.29)	14.93 (2.84)	10.45 (0.85)	11.94 (1.08)	12.69 (2.16)	10.72 (1.85)	10.66 (1.37)	13.68 (2.63)

Table 4: Percentage improvement in static mechanical properties of ANF/GPC

Tests	**Control**	**0.25 wt%**		**0.5wt%**		**0.75wt%**		**1wt%**	
		UT	**VT**	**UT**	**VT**	**UT**	**VT**	**UT**	**VT**
Tensile Strength	-	26.90	23.52	23.23	23.71	10.31	26.66	27.70	26.88
Tensile Modulus	-	10.85	21.86	-5.91	35.61	1.34	24.81	16.97	33.62
% Elongation	-	-26.19	-54.91	-39.14	-56.85	-31.10	-72.32	-19.20	-18.15
Equilibrium Toughness	-	45.34	44.35	41.53	42.23	22.88	47.76	44.63	40.54
Flexure Strength	-	4.08	12.14	-16.5	6.16	2.66	8.07	18.67	15.26
Flexure Modulus	-	7.19	22.79	-6.88	14.81	1.71	22.61	28.13	35.76
ILSS	-	6.26	18.40	-17.13	-5.31	0.63	-14.99	-15.46	8.49

The tensile strength properties of both sets of composites as compared to control composites showed an improvement of approximately 20%. However, the tensile modulus of VT-ANF/GPC set of composites are significantly higher than the UT-ANF/GPC. The equilibrium toughness, which is a measure of the work done on the sample before failure is at an average of 43% for both set of composites. The major change associated with the silane treatment of the alumina nanofibers is the interlaminar shear strength, which peaked at 0.25wt% loading level. It was observed that the VT-ANF/GPC due to the presence silane-coupling agent contributed to higher bonding strengths and improved ILSS property by 18.40%.

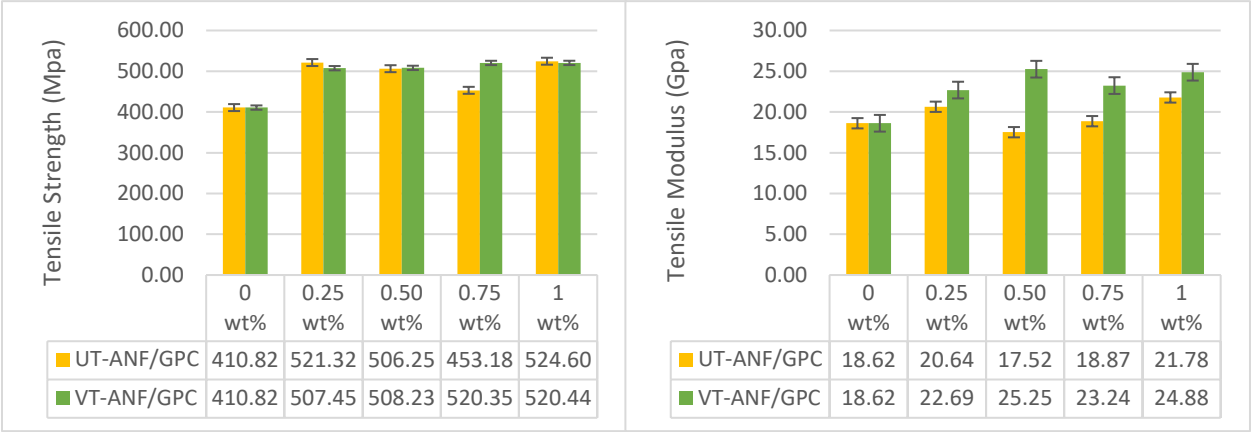


Figure 4: A summary of comparison of tensile strength and tensile modulus of UT-ANF/GPC and VT-ANF/GPC at 0.25wt%, 0.5wt%, 0.75wt% and 1wt%

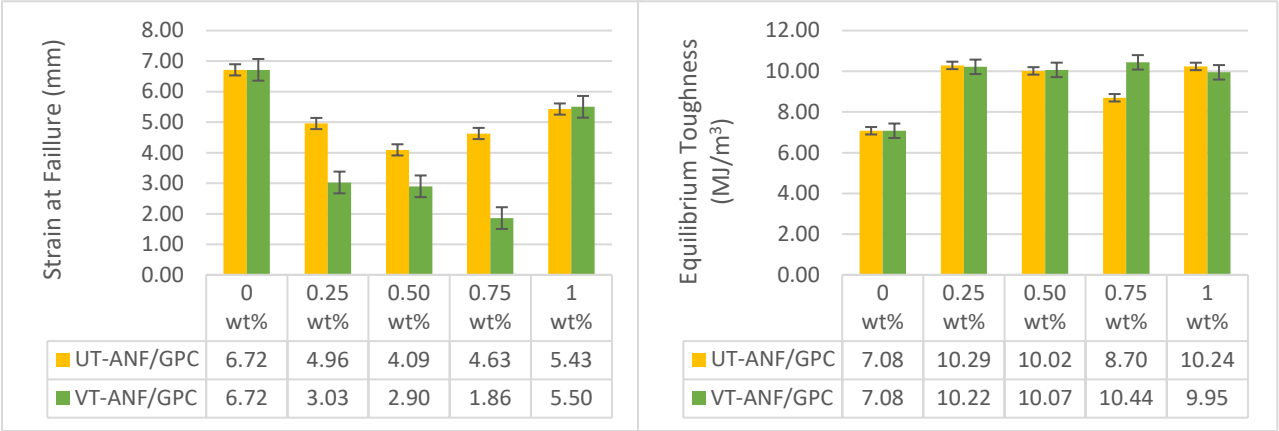


Figure 5: A summary of comparison of strain at failure and equilibrium toughness of UT-ANF/GPC and VT-ANF/GPC at 0.25wt%, 0.5wt%, 0.75wt% and 1wt%

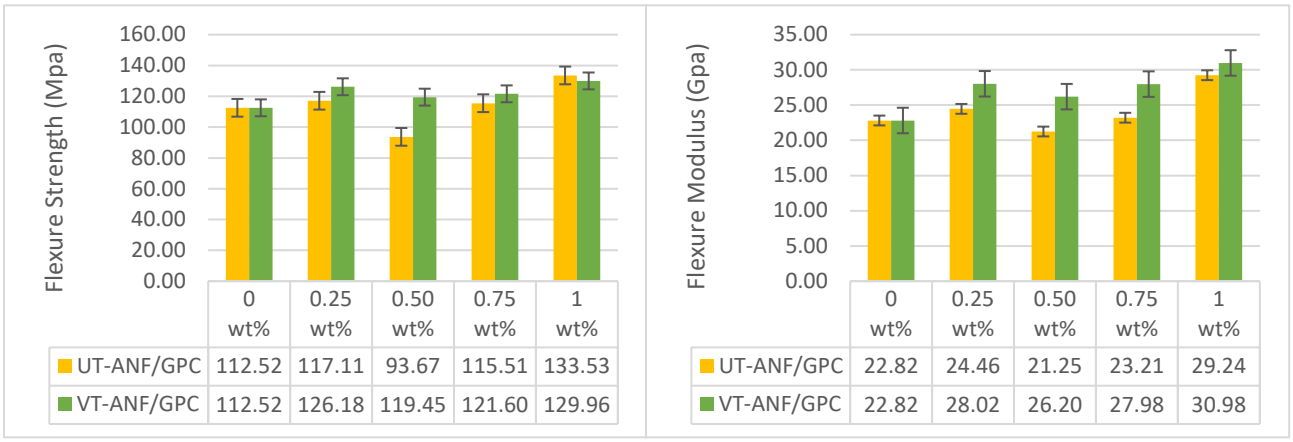


Figure 6: A summary of comparison of Flexure Strength and Flexure Modulus of UT-ANF/GPC and VT-ANF/GPC at 0.25wt%, 0.5wt%, 0.75wt% and 1wt%

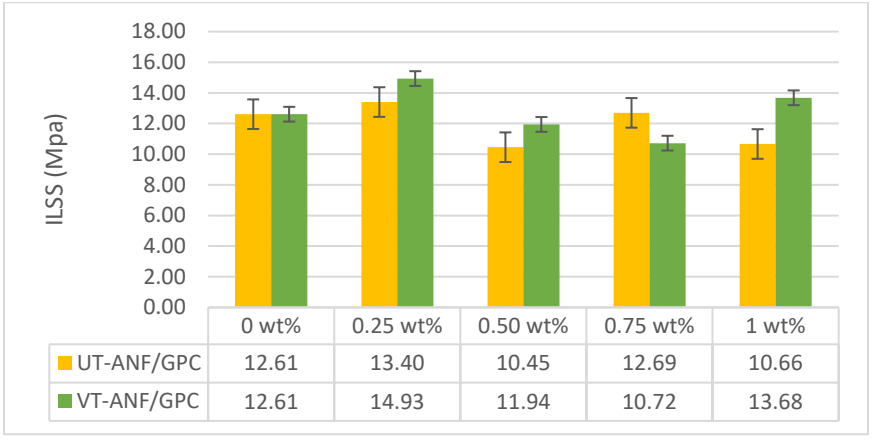


Figure 7: A comparison of ILSS of UT-ANF/GPC and VT-ANF/GPC at 0.25wt%, 0.5wt%, 0.75wt% and 1wt%

4.4 SEM Analysis

Figure 8 and Figure 9 shows the cross-sectional SEM images of samples after testing to understand the damage induced. The UT-ANF/GPC composite SEM images are shown in Figure 7, and they reveal fiber matrix debonding, fiber breakage, delamination, and cracks which are quite expected as it is a complete failure. Although the failure and damage are clearly visible, the interaction of UT-ANF modified polyester with the glass fibers cannot be clearly established. In case of VT-ANF/GPC, more of fiber pull out is prevalent and the fiber matrix debonding is less as compared to UT-ANF/GPC. AS for the short beam specimens considered here, the degree of bonding in each sample varied.

The morphology of fracture surfaces as seen in the figures 8 and 9 revealed that the composites are behaving more brittle than ductile which is expected from the glass reinforced polyester composites which is as seen from the morphologies of the fiber breakage. The surface topography of the silane treated alumina nanofiber filled composites showed smooth surfaces, with little fiber breakages and cleavages showing fiber pullout. The amount of resin still bonded to the fibers as seen in figure 9, reveals the enhancement in fiber adhesion to the matrix. In case of both sets of composites, the fiber is visible after the failure, meaning that there is uneven coating of fibers during hand layup. The SEM images reveal that failure at the interface is predominant than the rupture of the fibers.

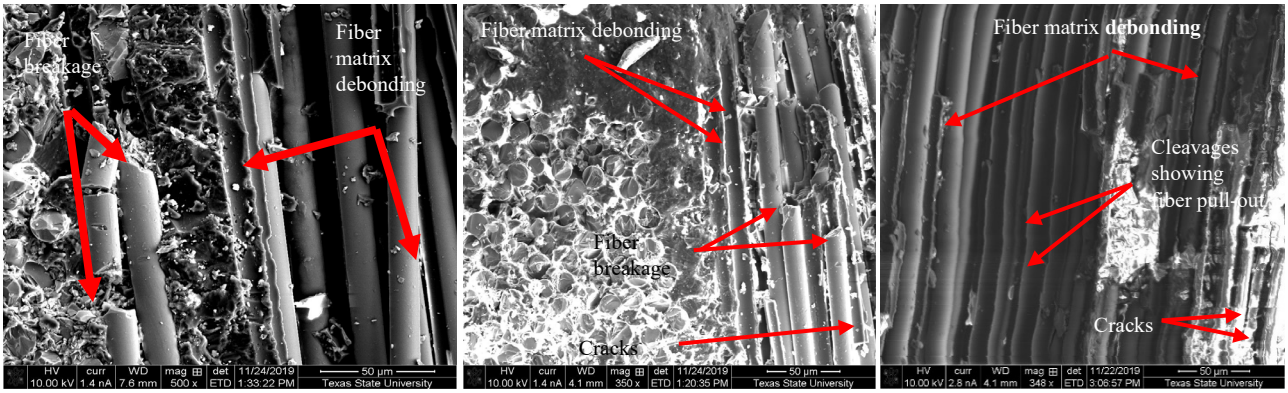


Figure 8: SEM images showing the fracture in the UT-ANF/GPC composites after testing

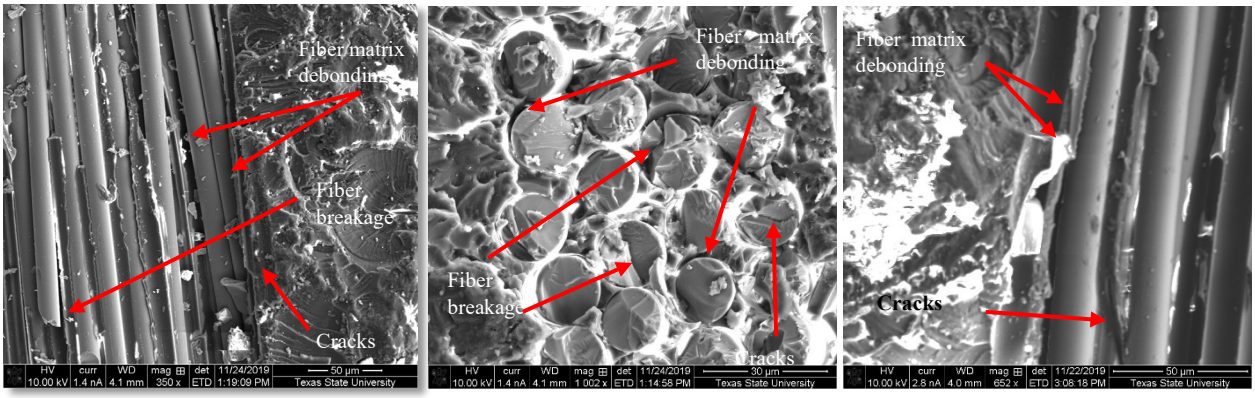


Figure 9: SEM images showing the fracture in the VT-ANF/GPC composites after testing

CONCLUSION

Transmission electron microscopy, thermo gravimetry analysis, tensile, flexure, interlaminar shear stress and scanning electron microscopy on the glass epoxy composites modified with untreated as received alumina nanofibers and silane treated alumina nanofibers is studied and compared. Observations in terms of dispersion, thermal stability, mechanical performance and fracture analysis on tested samples are reported in this article. TEM Images showed agglomeration at higher loadings but decent dispersion at lower loading. TGA thermograms revealed the improvement in thermal stability with increased loading level as measured by the peak mass loss. Silane treated alumina nanofiber peak mass loss temperature is at the maximum. The property improvement is obvious in alumina nanofibers. However, the major of the property values enhanced at 0.25wt% and 1wt% silane treated alumina nanofiber modified GPC. Interlaminar shear stress which is an important measure for strength against delamination has seen an improvement by 18.40% at 0.25wt% loading level.

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