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Effect Of Electrospun Nanofibers On Flexural Properties Of Fiberglass Composites

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Effect of Electrospun Nanofibers on Flexural Properties of Fiberglass Composites

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Biographical Sketch

Fatima White was born on May 31, 1985. She was born in the city of Salisbury, North Carolina. She is the daughter of Charles Lee White Jr. and Cathy Ann King. Fatima is the mother of Jamari E. White and Majar J. White. She attended Elizabeth City State University in Elizabeth City, North Carolina where she received her Bachelor of Sciences, degree in Physics and Chemistry in 2007. Fatima White is a candidate for a M.S. in Nanoengineering.

Dedication

This thesis is dedicated to my parents Charles Lee White Jr. and Cathy Ann King.

Acknowledgements

I would like to express my sincere gratitude to my advisor Prof. Ajit D. Kelkar for the support, for his patience and motivation. My sincere thanks to the Department of Nanoengineering and NASA Kennedy Space Center for providing financial support to perform exciting research in the emerging field of nanoengineered composite materials. I would like to thank my fellow students Dattaji Shinde, Earl Martin and laboratory manager Dr. Evan Kimbro and Ms. Karen Courtney for all the assistance during the course of study.

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Abstract

In the present study, sintered electrospun TEOS nanofibers were interleaved in S2 fiberglass woven fabric layers, and composite panels were fabricated using the heated vacuum assisted resin transfer molding (H-VARTM) process. Cured panels were water jet cut to obtain the flexural test coupons. Flexural coupons were then tested using ASTM D7264 standard. The mechanical properties such as flexural strength, ultimate flexural failure strains, flexural modulus, and fiber volume fraction were measured. The S-2 fiberglass composite with the sintered TEOS electrospun nanofibers displayed lower flexural stiffness and strength as compared to the composites that were fabricated using S-2 fiberglass composite without the TEOS electrospun nanofibers. The present study also indicated that the composites fabricated with sintered TEOS electrospun nanofibers have larger failure strains as compared to the ones that were fabricated without the presence of electrospun nanofibers. The study indicates that the nanoengineered composites have better energy absorbing mechanism under flexural loading as compared to conventional fiberglass composites without presence of nanofibers.

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CHAPTER 1

Introduction

In composite manufacturing, both fibers and matrix play key role depending upon the end application. Chemical resistance, strength, heat sensitivity, elasticity are some of the characteristics of fibers which determine the end application and cost of the fibers [1, 2]. Generally there are two different types of fibers, natural fibers and synthetic fibers. Among various types of synthetic fibers, glass fibers and carbon fibers are the most commonly used fibers in the world today. Typically glass fibers exhibit greater advantages than that of the carbon fibers when ultimate structure involves flexural applications or energy absorbing applications [3, 4, and 5]. Typically glass fibers are more flexible than carbon fibers. Glass fibers are significantly tougher than carbon fibers. The most significant difference between the glass fibers and the carbon fibers is that the glass fibers are significantly less expensive to manufacture than carbon fiber. The two commonly used glass fibers for the structural applications include E-glass and S2 glass fibers. The S2 glass fibers have been used for many years because of the outstanding performance when used as reinforcing fibers in polymers. The unique properties of the S2 glass fibers such as temperature resistance, high strength, light weight and impact resistance makes the S2 glass fiber reinforced composites suitable for many structural applications including aerospace, automotive, defense etc. [6]. Some of these applications of the S2 glass fiber reinforced composites include small plane fuselage, secondary structural parts of the aircraft (floors, doors, seats), helmets, exterior automotive body panels(fender, hoods, and roof tops), load floors, snowboards, high speed racing boats etc. [7]. Most commonly used forms of S2 glass include roving, chopped, and yarned fibers [8]. The two phase S2 glass composites consist of matrix and the reinforcement [9]. In the present research thermoset epoxy resin

reinforced with S2 glass fibers was used to fabricate two phase composites. In addition, three phase composite panels were manufactured using an additional third phase of TEOS (Tetraethyl Orthosilicate) electrospun glass nanofibers. The objective of the present study was to study the effects of flexural loading on the behavior of electrospun nanofiber reinforced fiber glass composites.

Present work involved fabrication, processing and characterization of S2 fiberglass composites with and without presence of TEOS electrospun nanofibers. The characterization included measurement of various properties such as fiber volume fraction, flexural properties including flexural modulus and the load deflection behavior of the nanofiber modified composites.

The literature review indicates that burn test can be effectively used to determine the fiber volume fraction in composite materials. Abdalla et al [10] determined the fiber volume fraction ratio of filament wound glass and carbon fiber reinforced composites by using American Society for Testing and Materials (ASTM) D2584 (1968) standard of testing. In the present study the two and three phase composite panels were fabricated using Heated Vacuum Assisted Resin Transfer Molding (H-VARTM) and the fiber volume fraction of the H-VARTM fabricated composite panel was determined using the burn test. The volume fraction for the fibers (VF) and the volume fraction for the matrix (VM) in the present research was in the range of 51.5% ±1% for fibers and 48.5% ±1% for matrix.

The present research involves fabrication of TEOS nanofibers. Wilkes [11] utilized the electrospinning process to make the electrospun TEOS nanofibers. According to Wilkes [11] in electrospinning process, the molecular weight and distribution, the design of the polymer, and the electrospun solution properties such as viscosity and surface tension are the typical system

parameters that affect the quality of electrospun fibers. In addition the distance between the tip of the spinneret and the collector plate, flow rate, electric field, humidity and temperature in the laboratory can also significantly influence the quality of nanofibers.

Shendokar et al [12] used Differential Scanning Calorimetric (DSC) to relate the variation in silicon dioxide in the electrospun nanofibers with increasing sintering temperature. They used TEOS sol-gel to produce nanofibers. The electrospun nanofibers were heated at three different temperatures; 300 degrees C, 600 degrees C and 900 degrees C. They observed significant reduction in the diameter of TEOS nanofibers after the sintering. They fabricated composite panels using the electrospun TEOS nanofibers which were sintered at the three different temperatures 300 degrees C, 600 degrees C and 900 degrees C respectively. They performed Short Beam Shear Strength (SBS) Tests as per ASTM D2344 and modified Short Beam Strength Tests (MSBS) to determine the performance of the nanoengineered composite laminates. They observed that the composite panels fabricated using 900 degrees Celsius sintered nanofibers exhibited the highest short beam shear strength. They concluded that the strength of the TEOS electrospun nanofibers increases as the sintering temperature increases.

Shendokar et al [13] used the H-VARTM method to fabricate two phase nanocomposites. They concluded that the glass moldings cab be effectively used in the H-VARTM system to fabricate high quality two phase nanoengineered composites. They compared the behavior of nanoengineered two phase composites with two phase composites manufactured using microfibers under tensile loading. They concluded that two phase nanoengineered composites exhibited better load-deflection performance compared to the two composites fabricated using microfiber composites. They performed fractographic examination of the failed coupons and

concluded that microfibers acts as a stress risers in the matrix and have significantly less deflection under tensile loading as compared to two phase nanoengineered composites.

Kelkar et al [14] studied effects of electrospun fibers on the interlaminar properties of woven composites. They performed double cantilever beam tests (DCB) to measure the fracture toughness of the three phase nanoengineered composites. They concluded that addition of TEOS electrospun glass nanofibers significantly improves the fracture toughness of the fiberglass composites.

The literature review clearly indicates that very little work has been done in the area of flexural behavior of nanoengineered three phase composites comprising of thermoset epoxy resin, S2 glass fibers and TEOS electrospun nanofibers. The following chapter presents the materials that were used in the present study and details of electrospinning to manufacture TEOS glass nanofibers.

CHAPTER 2

Materials and Electrospinning of Nanofibers

This chapter presents the constituent materials that were used for the fabrication of nanoengineered fiberglass composites. The nanoengineered fiberglass composites were fabricated using S2-Glass plain weave fibers, thermoset epoxy resin known as EPON 862-W and TEOS electrospun nanofibers. The details of electrospinning process for manufacturing the TEOS nanofibers are provided in following section.

2.1 Electrospinning Process

Electrospinning process was introduced in 1934 by Formhals [15]. Electrospinning is one of the most effective and low cost processes for manufacturing nanofibers. The simple and versatile process of electrospinning enables to produce nanofibers from different polymer solutions [16, 17]. The present study focuses on the use of Tetraethylorthosilicate (TEOS) sol-gel solution to produce TEOS nanofibers. The success of getting the TEOS electrospun nanofibers depends upon the viscosity of the sol-gel solution, the humidity and temperature within the room setting and the aging conditions in which the solution aged in the appropriate timing. The first step of the electrospun manufacturing process involves preparation of sol gel solution. There are two solutions that are used in the preparation of sol-gel solution. The first solution consists of 95.5grams of Tetraethylorthosilicate (TEOS) and 10.425grams of Ethanol (EtOH). They are combined and then magnetically stirred together. The second part of the sol-gel solution is obtained by mixing 20.8 grams of Ethanol (EtOH), 5 drops of hydrochorolic acid (HCL), and 8.3 grams of deionized water. A titration pipette is then used to combine these two solutions. The second solution in the pipette is combined with the first solution to obtain the sol-gel required for electrospinning of TEOS nanofibers (see Figure 2.1). It is critical that crystals should not form

during the creation of sol-gel solution. In order to have a sol-gel solution that will not crystalize completely at the time of mixing both parts of the sol-gel process, it was observed that solution two needs to be titrated into solution one at the rate of one drop every 7 to 10 seconds. This takes typically 2-2.5 hours to obtain the sol-gel solution with adequate viscosity for successful electrospinning of the TEOS nanofibers.

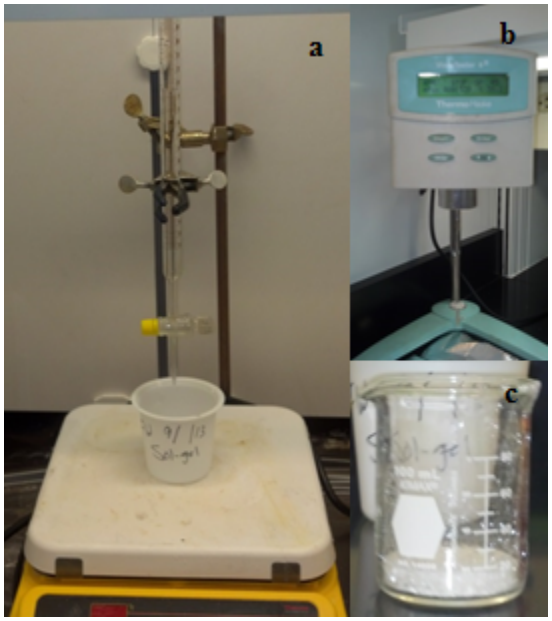

Figure 2.1. *a) Sol-Gel solution b) Viscosity meter*

The completed sol-gel solution was stored in a freezer and removed from the freezer whenever TEOS nanofibers fabrication using electrospinning was desired. This is typically done by taking out sol-gel solution out of the freezer and bringing it to room temperature until the viscosity of the solution is between 520-750 mPsa. The study indicated that, when the viscosity of the sol-gel solution was less than 500 mPsa the solution was to thin and did not produce good quality electrospun fibers. It was also observed that if the viscosity was above 750mPsa, the solution would turn into gel and will not produce electrospun fibers. In the present work the sol-

gel solution with the viscosity in the range of 520-750 mPsa was filled into a 30 ml syringe with a tip diameter of 50 mm and attached to a spinneret. The set up for electrospinning consisted of a programmable Model NE-1000 Multi-Phaser dispensing pump, FC series 120 Watt Regulated High Voltage DC Power Supply, spinneret and the collector plate [12].

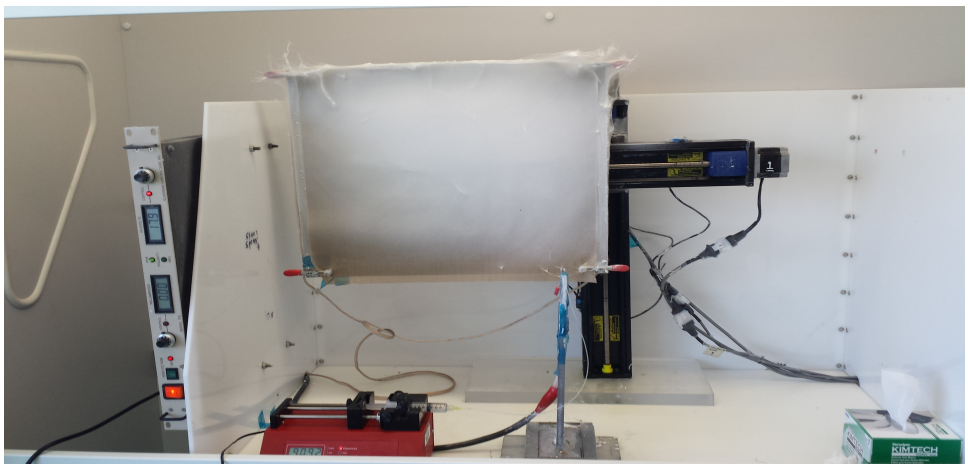

Figure 2.2. *Electrospinning setup*

Once the setup was complete, the electrospun fibers were collected on the collector plate which was covered with a Teflon sheet. This was done by dispensing the sol-gel at the rate of 2.0 ml for optimal nanofiber fabrication. In order for the fibers to be collected on the moving collector plate, a drop of the sol-gel solution at the tip of the spinneret must form an approximate 49.3 degree angle towards the collector plate and a whole angle width of approximately 98.6 degrees; this is known as the Taylor Cone [18] (see Figure 2.3).

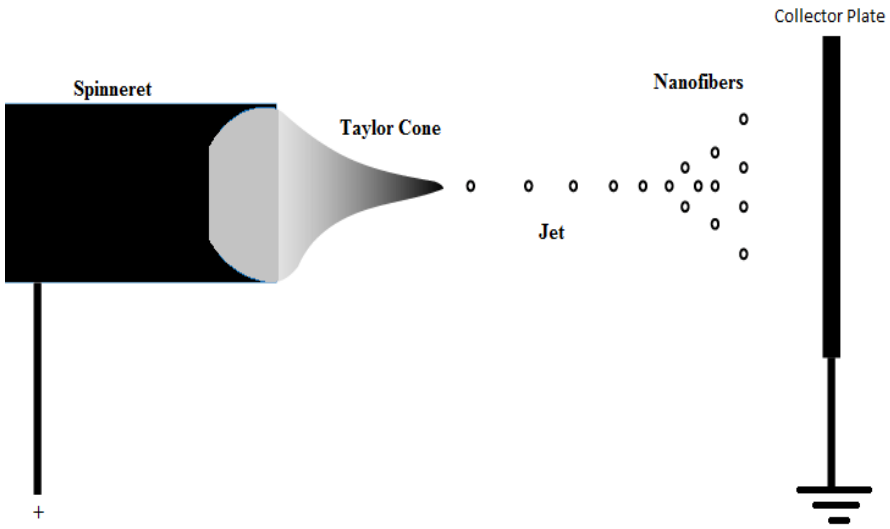

Figure 2.3. *Taylor Cone*

The Taylor Cone forms when the TEOS sol-gel solution changes shape because of the surface tension after the electric field has been applied. The surface tension of the sol-gel solution plus the potential difference from the collector plate causes the solution to form a conical shape at the tip of the spinneret where the jet will be formed and the plume of fibers will begin. The collector plate was grounded; and the tip of the spinneret was kept at a positive potential on the surface with a distance of 20.5 cm between the tip of the spinneret and the collector plate. Once the TEOS nanofibers were collected on a Teflon sheet, they were stored in in sealed plastic bags to prevent any damage or contamination. Figure 2.4 shows a collection of the electrospun nanofibers on the Teflon sheet after electrospinning.

Figure 2.4. *Electrospun TEOS nanofibers on a Teflon Sheet*

A sample of the electrospun fibers were taken and stored for characterization of the quality, diameter, and uniformity of the overall fibrous mat. This was achieved by The characterization was performed using the Scanning Electron Microscopy (SEM), where the electrospun nanofibers were coated with a five nanometer layer of gold palladium. The gold palladium was used to create a conductive surface to image due to the insulating nature of the TEOS nanofibers. Figure 2.5 shows a SEM micrograph of electrospun nanofibers.

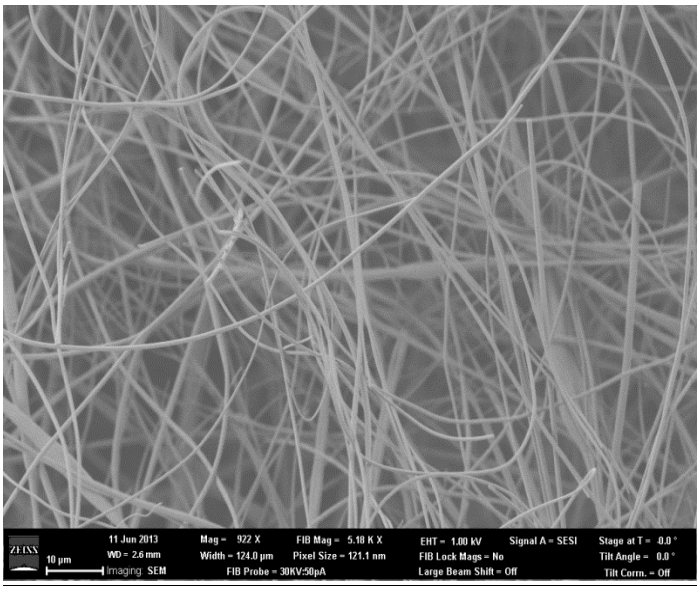

Figure 2.5. *Actual image of TEOS nanofibers under SEM*

2.2 Sintering of Electrospun TEOS Nanofibers

Typically the electrospun fibers are sintered so that the fibers would decrease the amount of ethanol saved into the fibers and they would become more solid material. Also the purpose of sintering is to decrease the diameter and increase the surface area of the TEOS electrospun nanofibers. The electrospun fiber mats were folded into squares and stacked one on top of the other and were sintered (see Figure 2.6) at 600 degrees C in Barnstead Thermodyne Inc. Furnace, model number 6000.

The oven was programmed to ramp for one hour to reach up to 600 degrees C from room temperature of 25 degrees C. After the ramping was complete, the dwelling was set at 600 degrees C for 6 hours and after it had dwelled for 6 hours at 600 degrees C, it was allowed to cool for 8 hours until it reached 25 degrees C. Sintering process helps to reduce residual ethanol from electrospun fibers. Sintering process helps to decrease the diameter of the electrospun fibers and increase the surface area (see Figure 2.7).

Figure 2.6. *TEOS electrospun nanofibers folded, stacked, and sintered at 600 degrees C*

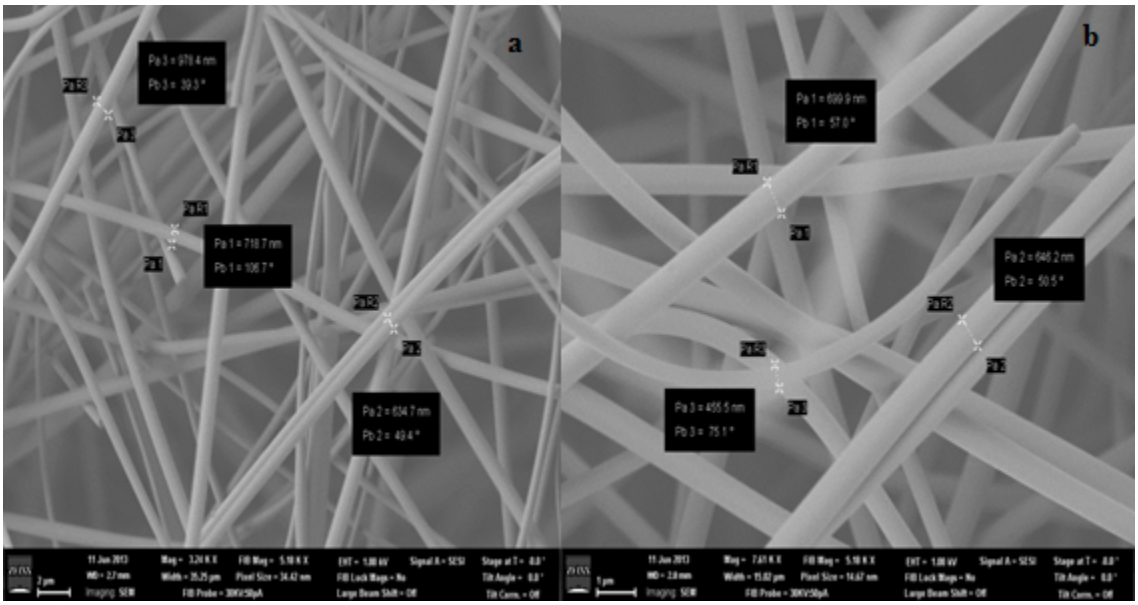

Figure 2.7. *SEM of decreasing diameter of TEOS electrospun nanofibers : a) before sintering b) after sintering*

Smaller diameter with large surface area usually results into better wetting of fibers during two phase or three phase composite manufacturing process. This also helps to achieve

better fiber volume fractions, less voids during the composite manufacturing. The sintered TEOS electrospun nanofibers manufactured using the procedure discussed earlier were interleaved into S-2 glass fiber composite as discussed below.

2.3 Materials

In order to manufacture three phase composites following constituent materials were used:

- S-2 glass woven fibers BGF 240(S-2 463-AA-250) [21]
- EPON resin 862, EPICURE system curing agent W
- Tetraethylorthosilicate (TEOS) electrospun nanofibers. Glass Fibers

The details of each of the constituent materials are provided below:

S-2 glass woven fibers BGF 240(S-2 463-AA-250) The S-2 BGF 240 glass fibers are a repeating square packed array, called unidirectional fiber square packing geometry (see Figure 2.8).

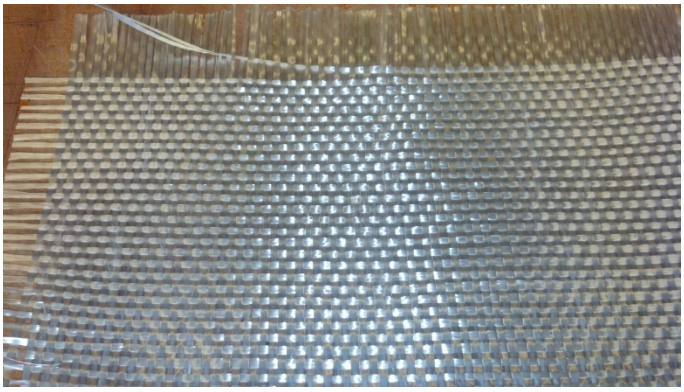

Figure 2.8. *The S-2 glass fibers BGF 240*

These glass fibers are popular for structural applications due to low cost and high strength and stiffness. Some of the applications include Many applications of the BGF 240(S-2 463-AA-250) glass fibers include sports, automotive, aerospace and energy (wind turbine blades) for the panel fabrication process with and without the TEOS electrospun nanofiber composites.

The resin system used in the present study consisted of two-part thermoset epoxy resin. These two parts included EPON resin 862 and the W curing agent EPICURE. EPON resin 862 (Diglycidyl Ether of Bisphenol F) (see Figure 2.9), is a low viscosity, liquid epoxy resin manufactured from epichlorohydrin and Bisphenol-F. When EPON Resin 862 is cross-linked with the W curing agent EPICURE (diethyl methyl benzenediamine), it results into superior mechanical, adhesive, electrical and chemical resistance properties.

Figure 2.9. *a) EPON resin 862, b) W curing agent EPICURE*

Typically for every 100 grams of EPON 862 26.4 grams of curing agent EPICURE W is added. The amount of resin required for fabrication of fiberglass panels depend upon size of the panel, number of layers (thickness of the panels). The following section presents details of composite fabrication process using heated vacuum assisted resin transfer molding process.

2.4 Panel Fabrication

The present study involved fabrication of two composite panels, first without TEOS electrospun nanofibers and the second one with TEOS electrospun nanofibers. In both cases six sheets of S2 glass fiber were cut evenly with the dimension of 14'X 9" and were stacked on each other in the same (zero degrees) direction. In the case of panels with TEOS electrospun nanofibers, the 5 layers of electrospun nanofibers were interleaved between the six layers of S2 glass fiber sheets.

The composite panels fabrication involved use of glass mold and double vacuum bag technique commonly used in vacuum assisted resin transfer molding. Two panels one with and the second one without TEOS electrospun nanofibers were manufactured using H-VARTM process [12] (see Figure 2.10). Panels fabricated using H-VARTM process were then cured as per the manufacturer recommend cycle as shown in Figure 2.11 and LR Technologies model number ST867TUL240V90KW walk in oven as shown in Figure 2.12

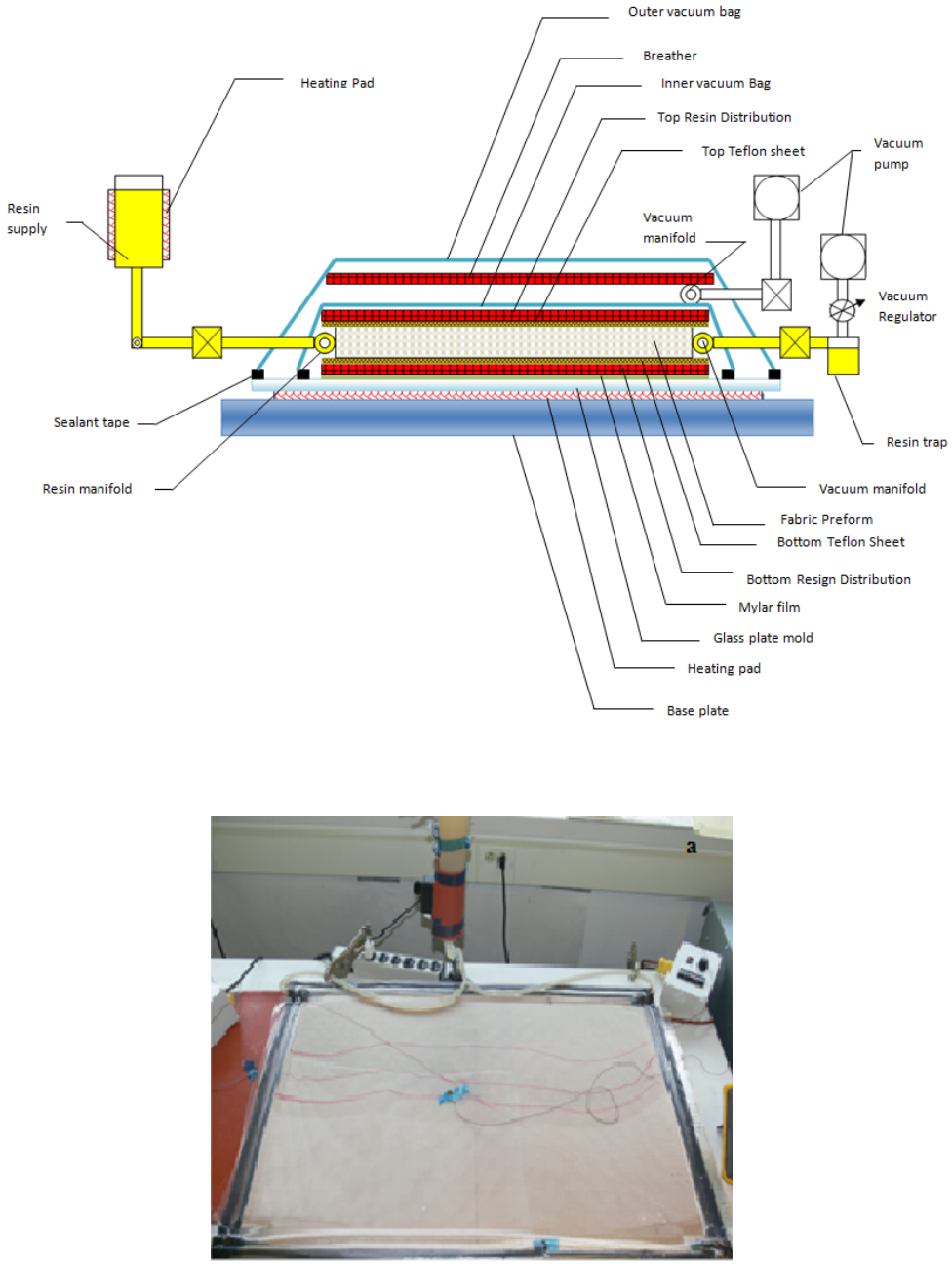

Figure 2.10. *H-VARTM process- schematic and actual set up*

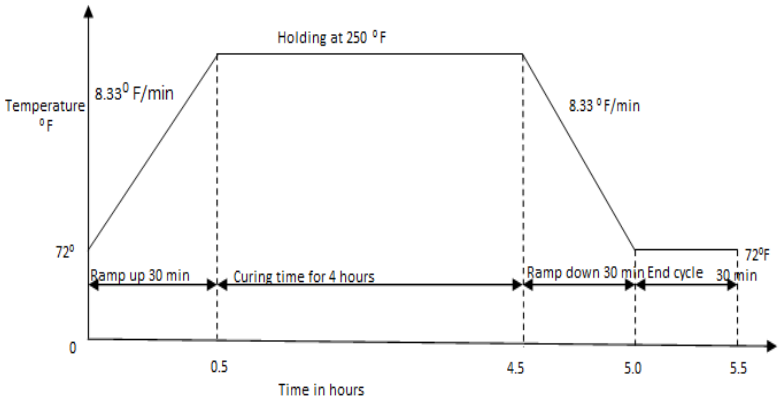

Figure 2.11. *Curing cycle*

Figure 2.12. *LR Technologies ST867TUL240V90KW walk in oven used for curing cycle of the composite fabrication*

The cured panels were then cut into flexural coupons to determine the flexural properties of fiberglass composites with and without electrospun TEOS nanofibers. The details of flexural characterization are provided in the next chapter.

CHAPTER 3

Characterization of Nanoengineered Composites

This chapter presents details of flexural testing of composite laminates which were fabricated using H-VARTM method. The first panel included six layers of S2 glass woven fibers infused with EPON resin 862 and curing agent EPICURE W and the second panel had identical constituent materials except TEOS sintered electrospun nanofibers were interleaved between the S2 fiber glass layers. These two types of fabricated panels are shown in the Figure 3.1.

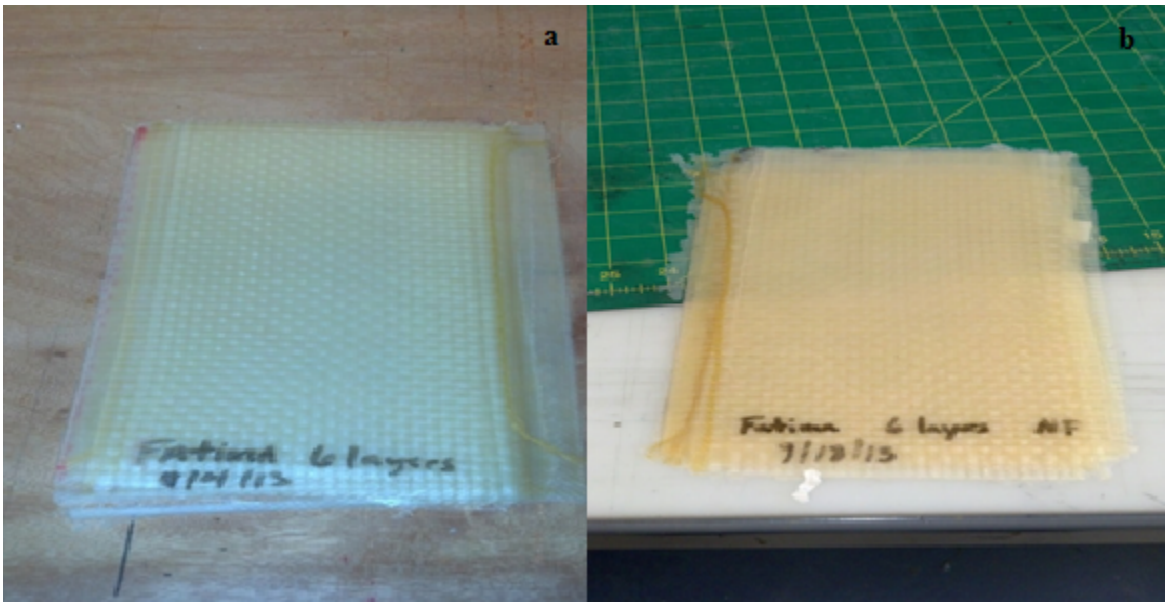

Figure 3.1. *After curing a) six layers S2 fiberglass composite panel b) six layers S2 fiberglass composite panel with TEOS sintered electrospun nanofibers*

3.1 Specimen Preparation

Before infusion of resin, weight of both S2 fiberglass layers and TEOS electrospun sintered nanofibers that were used in the fabrication of the two panels was determined. After the infusion and curing of the panels, they were weighed again. This was necessary to determine the

fiber volume fraction of each of the panel. It was observed that the panel with TEOS sintered electrospun nanofibers weighed almost 20% more than the one without the presence of electrospun nanofibers. This might be due to the fact that during resin infusion process, more resin is used in wetting electrospun nanofibers. The cured panels then were cut into flexural coupons as per the ASTM D7264 standard using the Flow International M2-1313b water jet cutting machine (see Figure 3.2). Water jet cutting machine was programmed to cut the coupons from the two panels as per ASTM D7264 standard and were 10 inches long and 0.5 inches wide as shown in Figures 3.3 and 3.4 respectively.

Figure 3.2. *Water Jet machine for cutting the flexural coupons*

These coupons were then labeled as GF1-GF6 and NF1-NF6, where GF symbol was used for the panel without presence of nanofibers and NF symbol was used for the panels with the

presence of nanofibers. All 12 coupons, GF1-GF6 and NF1-NF6 were measured for the dimensions and details are provided in Table 3.1 and Table 3.2 respectively.

Figure 3.3. *S2 fiberglass flexural coupons numbered GF1-GF6*

Table 3.1 *Thickness and width measurements for S2 fiberglass flexural coupons*

Coupon Number	**Thickness (inches)**	**Width (inches)**
GF-1	0.155	0.496
GF-2	0.151	0.502
GF-3	0.159	0.494
GF-4	0.154	0.504
GF-5	0.158	0.499
GF-6	0.163	0.498

The width and thickness was calculated using the micrometer. The coupons cut from the S-2 glass fibers plus the TEOS electrospun nanofibers were labeled NF1-NF6 and the thickness and width were taken from each coupon as well

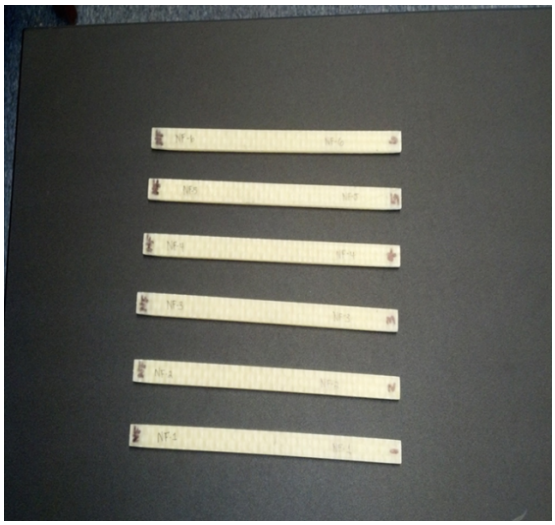

Figure 3.4. *S2 fiberglass flexural coupons with sintered TEOS electrospun nanofibers numbered*

Table 3.2 *Thickness and width measurements of the S2 fiberglass flexural coupons with sintered TEOS electrospun nanofibers NF1-NF6*

Coupon Number	Thickness (inches)	Width (inches)
NF-1	0.205	0.492
NF-2	0.216	0.501
NF-3	0.202	0.499
NF-4	0.215	0.503
NF-5	0.209	0.495
NF-6	0.213	0.500

3.2 Determination of Fiber Volume Fraction

The fiber volume fraction of the flexural coupons was determined using the ASTM D3171-11[19] standard. The ASTM D3171 test method is usually used to determine the constituent content of composite materials. This method involves physically removing the matrix by either digestion or ignition method. Once the matrix is removed, then the fiber weight/volume is measured. Do determine the fiber volume fraction for the panels with and without sintered

TEOS electrospun nanofibers, three specimens 1'X 1" squares samples were cut from each of the panels (see Figure 3.5), resulting into six specimens.

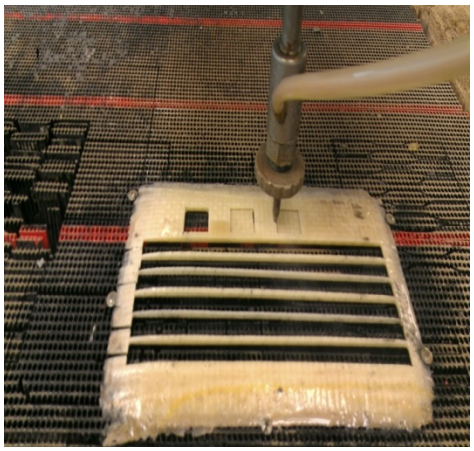

Figure 3.5. *Cutting samples for determination of the fiber volume fraction*

In order to remove the matrix from the glass fiber composites, they were placed in high temperature Furnace 6000 made by Barnstead Thermodyne Inc. At high temperature, the burn-out process removes the EPON resin 862, and the EPICURE system curing agent W from the composites leaving the glass fibers for three specimens and glass fibers containing the TEOS electrospun nanofibers fibers for the other three specimens. Once the burn-out process was complete, fiber residue for each of the specimen was weighed. The fiber volume fraction ratio was then calculated as per the procedure outlined in ASTM D3171. The results are provided in Tables 3.3 and 3.4.

Table 3.3 *Fiber volume fraction for composite specimens without TEOS nanofiber*

Average mass of composite specimens (grams)	4.238
Average mass of the glass fibers (grams)	2.905
Average mass of the matrix (grams)	1.333
Density of glass fibers (gram/cc)	2.46
Density of matrix (gram/cc)	1.2
Average volume of composite specimens (cc)	2.291
Average volume of S2 glass fibers (cc)	1.18
Average volume of matrix (cc)	1.11
Fiber volume fraction	0.515

Table 3.4 *Fiber volume fraction for composite specimens with TEOS nanofiber*

Average mass of the nanoengineered composite specimens (grams)	5.071
Average mass of the glass fibers + TEOS nanofibers (grams)	3.011
Average mass of TEOS nanofibers (grams)	0.106
Average mass of the matrix (grams)	2.06
Density of glass fibers (gram/cc)	2.46
Density of matrix (gram/cc)	1.2
Average volume of composite specimens with TEOS nanofibers (cc)	2.94
Average volume of S2 glass fibers + TEOS nanofibers (cc)	1.224
Average volume of matrix (cc)	1.716
Fiber volume fraction (including TEOS nanofibers)	0.416

Once the fiber volume fractions for the composite specimens with and without electrospun nanofibers were determined, coupons were tested under flexural loading. The details of flexural loading are provided in the next section.

3.3 Flexural Testing

The composite specimens with and without sintered TEOS electrospun nanofibers were tested using 3-point bend test fixture as outlined in ASTM D7264 [20]; standard test method for flexural properties of polymer matrix composite materials. This test method utilizes center point loading on a simply supported beam. The flexural specimen is simply supported on both end supports and is loaded at the center of the two supports as shown in Figure 3.6.

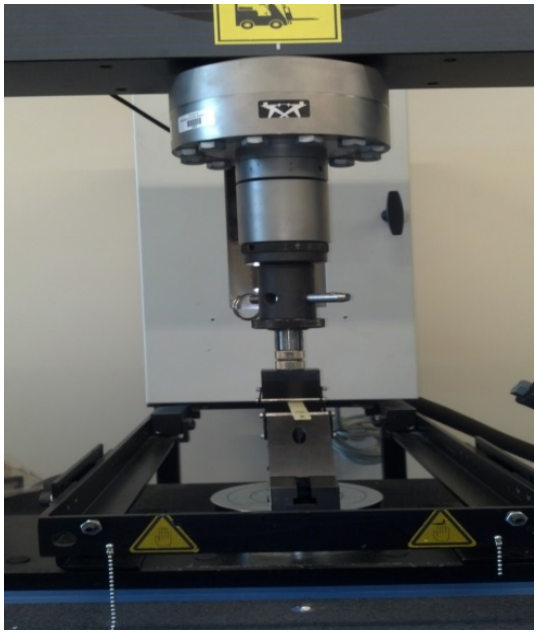

Figure 3.6. *Three point bend fixture set up on Instron 5584 machine with 150 KN load cell*

In the present case for the flexural coupons the span-to-thickness ratio was about 32:1, with specimen thickness ranging from 0.15” for S2 fiberglass composites to 0.20” for S2

fiberglass composites interleaved with sintered TEOS electrospun nanofibers and width was of 0.5". All the tests were conducted at the rate of 0.05 in. /min (see Figure 3.7). The Blue hill system on the Instron machine was used to record flexural stress and strain values and to calculate the flexural modulus. Figure 3.8 shows the comparison of flexural strength and modulus for the S2 fiberglass composites and results are shown in Tables 3.5 and 3.6. The flexural stress strain responses are presented in Figures 3.9 and 3.10.

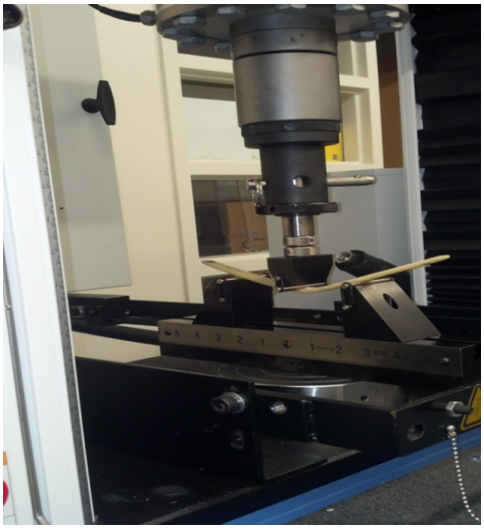

Figure 3.7. *Flexural testing of composite specimens using 150 KN Instron 5584 machine*

Table 3.5 Results of flexural testing for S2 fiberglass composites

Specimen number	Support span(inches)	Thickness (inches)	Width (inches)	Maximum strain	Maximum stress (Ksi)	Flexural modulus (Msi)
1	5	0.155	0.496	0.027	61.95	3.159
2	5	0.151	0.502	0.028	54.06	2.733
3	5	0.159	0.494	0.031	65.65	3.191
4	5	0.154	0.504	0.027	67.37	3.357
5	5	0.158	0.499	0.019	53.89	3.256
6	5	0.163	0.498	0.028	67	3.217
Mean	5	0.159	0.499	0.027	61.95	3.152
Standard deviation	0	0.004	0.004	0.004	6.250	0.216

Table 3.6 *Results of flexural testing for S2 fiberglass composites with sintered TEOS electrospun nanofibers.*

Specimen number	Support span(inches)	Thickness (inches)	Width (inches)	Maximum strain	Maximum stress (Ksi)	Flexural modulus (Msi)
1	5	0.205	0.492	0.03	50.16	2.54
2	5	0.216	0.501	0.029	46.69	2.622
3	5	0.202	0.499	0.029	47	2.39
4	5	0.215	0.503	0.03	45.81	2.63
5	5	0.209	0.495	0.032	42.46	2.47
6	5	0.213	0.5	0.028	50.47	2.39
Mean	5	0.210	0.498	0.030	47.098	2.507
Standard deviation	0	0.005	0.004	0.001	2.711	0.098

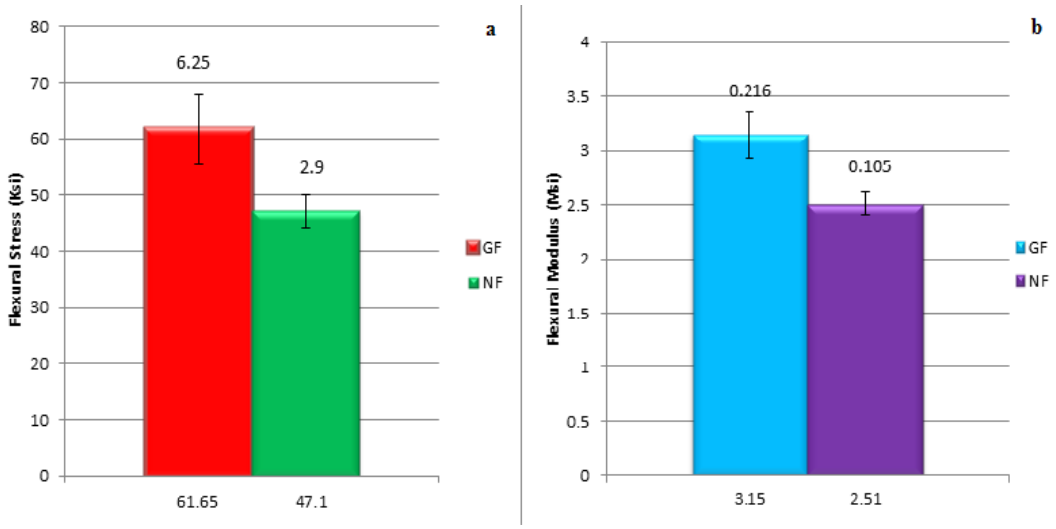

Figure 3.8. *a) Maximum flexural stress b) Flexural modulus of elasticity (Chord Modulus)*

Typically under flexural loading the specimen can fail in various modes and these modes are generally classified into three parts (see Table 3.7). The first part involves initiation of the failure, second part is the progressive failure or intermittent failure and the third part is the final failure mode. The failure methods for the beginning, intermittent or progressive failure and the final failure are recorded and are assigned three characters.

In the present study, all the tested specimens exhibited CAT (compressive, at loading nose, top surface) failure modes. In general failure mode was due to compression and interlaminar shear. The cross sections of the failed specimens were examined using scanning electron microscope to study the failure mechanisms. The micrographs of failed S2 fiberglass composite specimen and S2 fiberglass specimens with sintered TEOS electrospun nanofibers are shown in Figure 3.11.

Table 3.7 *Three-part Identification codes of failure*

First Character	
Failure Mode	Code
Tension	T
Compression	C
Buckling	B
Interlaminar Shear	S
Multi-mode	M(xyz)
Other	O

Second Character	
Failure Mode	Code
At loading nose	A
Between loading noses	B
at Support nose	S
between Load and support nose	L
Unknown	U

Third Character	
Failure Mode	Code
Top	T
Bottom	B
Left	L
Right	R
Middle	M
Various	V
Unknown	U

3.4 Results and Discussion

The present study involved flexural testing of the S2 fiberglass composites and S2 fiberglass composites interleaved with sintered TEOS electrospun nanofibers. Flexural coupons for both types of composites were obtained using water jet cutting of the composite panels. These panels were fabricated using H-VARTM method. Study indicates that the thickness of sintered TEOS nanoengineered composite was approximately 33% higher as compared to S2 fiberglass composite. The flexural stiffness of sintered TEOS nanoengineered composite was 21% lower and flexural strength was 23% lower as compared to S2 fiberglass composite. Study also revealed that the failure strains for sintered TEOS nanoengineered composites was 11% higher compared to the S2 fiberglass composites (see Figures 3.9 and 3.10). The reduction in the stiffness and strength can be attributed to the fact that the sintered TEOS electrospun nanofibers

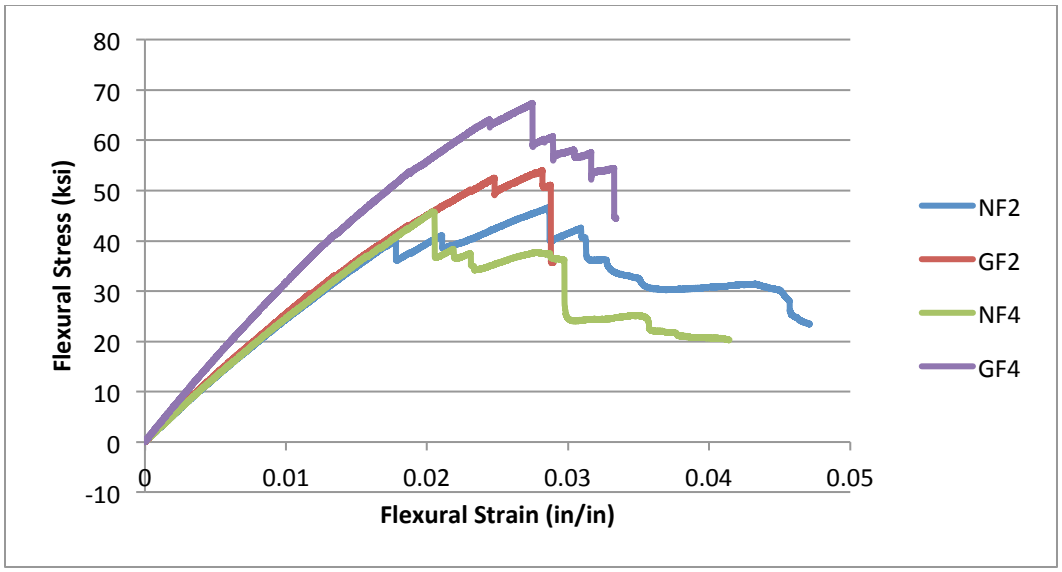

Figure 3.9. *Flexural stress vs. Flexural strain (Samples 2 and 4)*

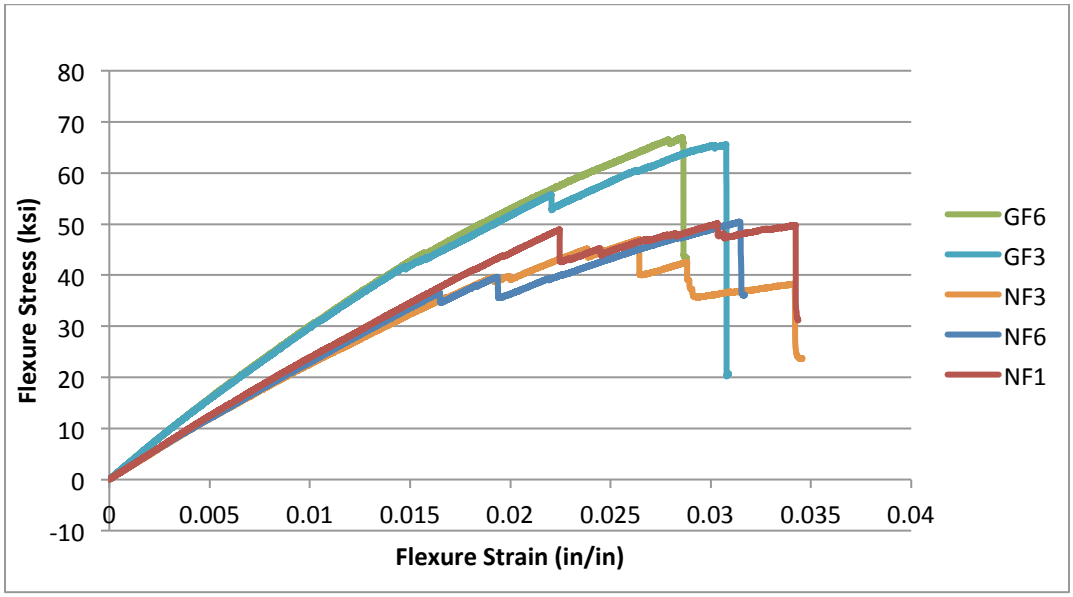

Figure 3.10. *Flexural stress vs. Flexural strain (Samples 3 and 6)*

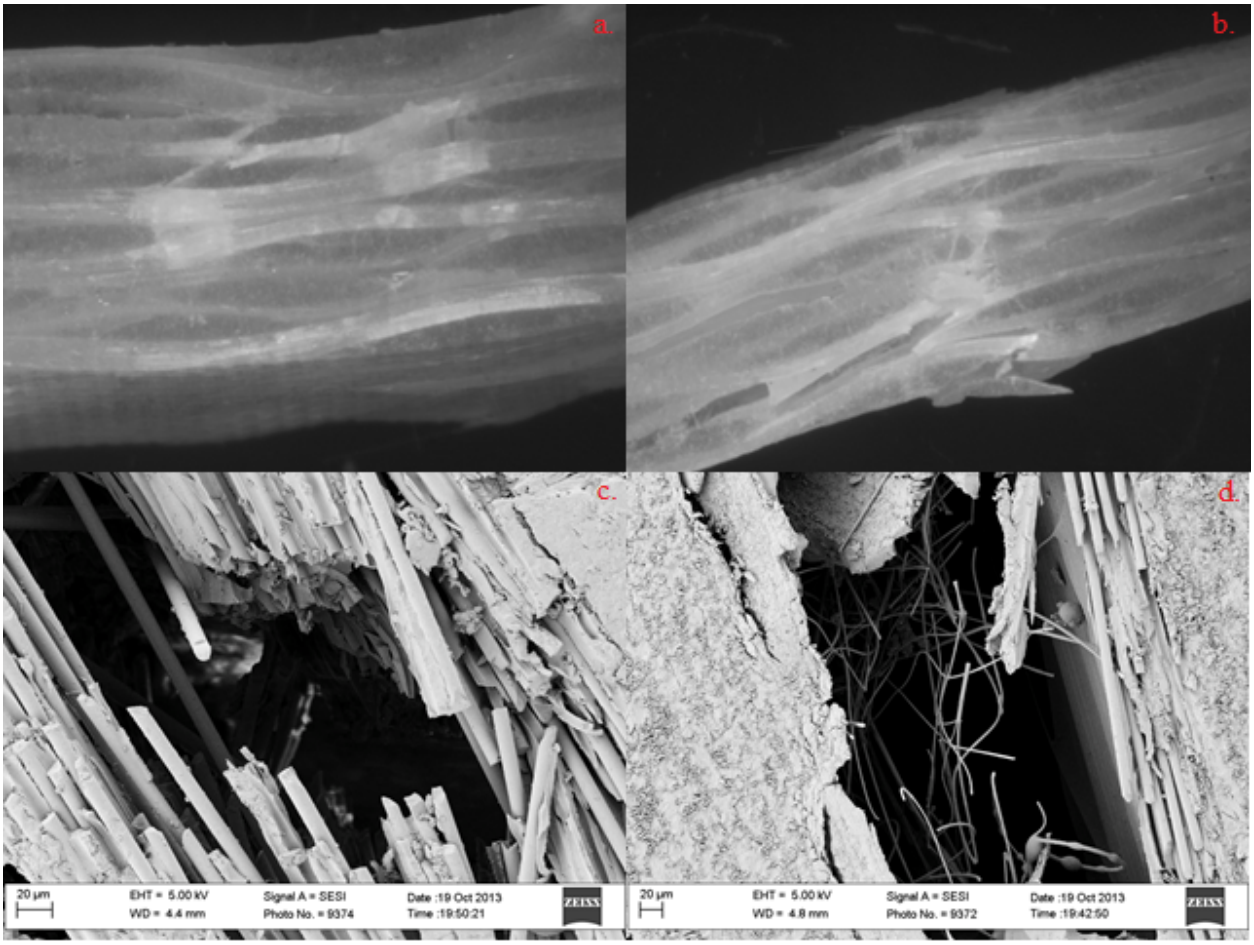

Figure 3.11. *a) Microscopic image of the failed S2 fiberglass composite b) Microscopic image of the S2 fiberglass composites interleaved with sintered TEOS electrospun nanofiber c) SEM Image of the failed S2 fiberglass composite d) SEM Image of the S2 fiberglass composites interleaved with sintered TEOS electrospun nanofiber*

used in the present study were not aligned along the S2 fiberglass direction, but were randomly oriented. Furthermore the sintered TEOS electrospun nanofibers were not functionalized. Silane functionalization might improve the load transfer between nanofibers and epoxy resin. Fractographic examination of the failed specimens (see Figure 3.11) revealed that sintered TEOS nanoengineered specimens exhibited different interlaminar failure mechanisms as compared to S2 fiberglass composites. Interleaved TEOS electrospun nanofibers served as interlaminar crack arrester and provided delamination resistance as could be seen from the flexural stress-strain response. Also study indicates that the sintered TEOS electrospun nanofibers would help to improve the toughness of the composites but the nanoengineered composites would exhibit lower flexural stiffness and strength as compared to S2 fiberglass composites.

CHAPTER 4

Summary and Conclusions

In the present study S2 fiberglass composite panels were fabricated using the H-VARTM method. The first panel was fabricated using six layers of woven S2 fiberglass sheets interleaved with sintered TEOS electrospun nanofibers and infused with EPON resin 862 and the W curing agent EPIKURE. The second panel consisted of six layers of S2 fiberglass sheets and was fabricated using identical constituent materials as the first panel without the presence of TEOS electrospun nanofibers. TEOS nanofibers used in the fabrication of nanoengineered composite panels were manufactured using electrospinning of the TEOS sol-gel solution. Both types of panels exhibited good quality with fiber volume fractions in the range of 40% to 50% and void contents of less than 1%. The panels were cut into flexural coupons and were tested using three point bend fixture to determine the flexural modulus and strength. The study indicates that nanoengineered S-2 glass fiber composites containing the TEOS electrospun nanofibers exhibited significantly higher strains to fracture and absorbed more energy than the S-2 glass fiber composites without the electrospun nanofibers. The study also showed both reductions in flexural stiffness and strength for nanoengineered composites as compared to S2 fiberglass composites. The study indicates that the nanoengineered composites comprising of TEOS electrospun nanofibers interleaved into the S-2 glass fibers and EPON 862-W resin has improved toughness as compared to the conventional S-2 glass fiber composites and would be suitable for applications involving out of plane flexural loadings.

Some of the future directions that would help to improve the stiffness and strength of the nanoengineered composites include: (a) varying sintering temperature of nanofibers (b) varying

percentage of interleaved TEOS nanofibers (c) studying the effects of functionalization on the stiffness and strength of the nanoengineered composites (d) to study the effect of alignment of sintered TEOS nanofibers.

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