

# Assessment of Three Phase Nanocomposite with Electrospun Nanofibers

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**Abstract:** *Electrospun nylon66 nanofibers of submicron diameter were used to for interlaminar reinforced in glass epoxy composite. Nylon 66 nanofibers (NFs) equivalent to 1% weight were used to improve the interphase characteristics. This paper presents the comparison of interlaminar strength using SBS ASTM (D 2344) for composite produced by Vacuum Assisted Resin Transfer Molding (VARTM) and by Hand Molding (HM) process. For both methods VARTM and HM, specimen with interlaminar NFs and without NFs was tested. It is observed that interlaminar with NFs improves in both VARTM and HM specimens. Further it was observed that better improvement in interlaminar shear strength (ILSS) is achieved in VARTM than HM.*

**Keywords:** Electrospinning, Nylon 66 nanofibers, VARTM, Short beam shear strength and Interlaminar shear strength

## 1. Introduction

Glass/Carbon Fiber reinforced polymeric composites are attractive structural materials for defence, aerospace, marine, automobile, transportation, sporting goods industries as well as in civil engineering structures due high stiffness and high strength [1]. The limitation of Glass Fiber Reinforced Polymer (GFRP) composite is their weak interlaminar strength. The three regions in a composite that have experience damage leading to the fracture are fiber, matrix and interface. Material failure occurs at i) Matrix cracking, ii) fiber breaking, iii) delamination or interface/interlaminar cracking. Most frequently encountered failure mode of fiber glass composite laminates is interlaminar delamination, which results in accidental material failure due to sub-surface hidden nature.

A number of methods has been developed to prevent delamination [2-5]. These include optimization of stacking sequence, critical ply termination, laminate stitching, edge cap reinforcement, matrix toughening, braiding and replacement of a stiff ply by one that has softer regions. Above mentioned method are either costly or weight penalties. Furthermore some material results into improve the ILSS but results into drastic degradation of inplane properties.

Nanotechnology has brought revolutionary changes in the properties of the material. As the diameter of the fibers gets reduced, aspect ratio i.e. surface area to volume ratio increases exponentially which may result in to increased binding capacity and enhancement of the mechanical properties by interleaving the nano fibers at the interface of lamina. The electrospinning process offers a potential cost effective means to reduce fiber diameters dramatically, resulting in vast improvements in fiber mechanical properties. It is anticipated that the conjugation of electrospun nanofibers with conventional microfibers in traditional composites paves way for variety of property improvements. Many researchers have worked to improve the ILSS of fiber reinforced polymer composites (FRPC) by

incorporating nanoparticles [6–13]. Tetra Ethyl Orthosilicate (TEOS) nanofibers interleaved at the interface have shown improvement in ILSS [14] 0.6 %TEOS by weight nanofiber in epoxy improved 15% ILSS [15]. Plain woven carbon with epoxy resin Epikote 828 with 0.1 wt. % Nano-Polyvinyl alcohol fibers improved Interlaminar fracture Toughness for crack initiation by 65% & crack propagation by 73%. Glass fiber Bisphenol epoxy with 0.1 wt of CNT increased interlaminar shear strength by 10% [7]. E-glass fiber with B-440 premium polyester resin with 0.1 to 0.4 wt CNF with VARTM process increased storage modulus by 92% [8]. Carbon fiber Trifunctional Epoxy (Araldite MY 510) composite with 0.1 wt. % CNF enhanced flexural modulus by 21.2% [9] Short beam shear strength increased by 20% by interleaving TEOS nanofiber[16].

## 2. Experimental Setup

Here it is proposed to produce Nylon 66 nanofibers using electrospinning process and incorporate in GFRP using Vacuum Assisted Resin Transfer Molding (VARTM) and Hand Molding (HM) process.

### 2.1 Electrospinning setup for naofiber synthesis.

There are fundamental three components associated with the electrospinning process viz. 1) A high voltage supply 2) A capillary tube with a pipette or needle of small diameter, and 3) Collector ( plate or drum) . One electrode is placed into the polymer solution/melt and the other is attached to the collector as indicated in figure. 1.a actual setup at BVUCOE, Pune is seen in figure 1.b.

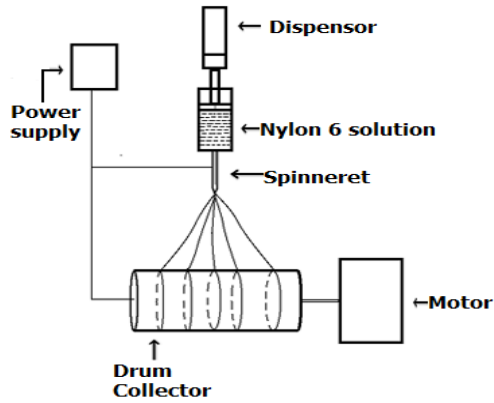


Figure 1: a) Schematic of Electrospinning Setup for Aligned Fiber Deposition [15]

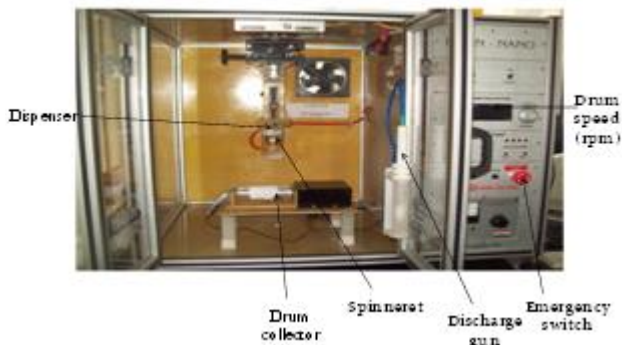


Figure 1: b) Actual Setup for Electrospinning Process at BVUCOE, Pune

The polymer, which induces a charge on the surface of the polymer. Further with increasing the electric field, a critical value is attained at which the repulsive electrostatic force overcomes the surface tension and the charged jet of the fluid is ejected in the form of polymer nanofibers. Nylon 66 nanofiber of diameter 1200nm were synthesis with the parameters distance between drum collector and syringe 10 cm, flow rate 0.5ml/hr, voltage 20KV and concentration is 18%. The quality and variety of nanofibers produced by electrospinning, cheaper than other process.. Figure 2 shows SEM image of nylon 66 which are used to interleave between laminas to reduce the delamination.

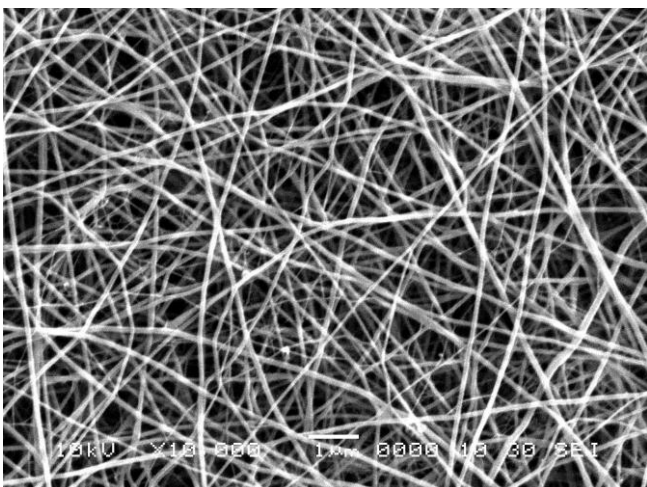


Figure 2: SEM of Nylon66 (1200 nm)

## 2.2 Vacuum Assisted Resin Transfer Molding (VARTM)

In VARTM, the upper metal tool is replaced by a vacuum bag. figure.3 and 4 shows a generalized schematic diagram and actual setup at BVUCOE,Pune of the VARTM process respectively First, peel ply in placed on the glass mold ,then electrospun nylon 66 nanofiber spun on laminas of E glass fiber 7781 purchased from Interglas as shown in figure. 6. Then this laminas are stack together as shown in the figure 7. The stack is covered with peel ply and flow distribution media mesh is placed on the peel ply for proper flow of the Epolam epoxy resin 5015 purchased from Axon France in laminate.

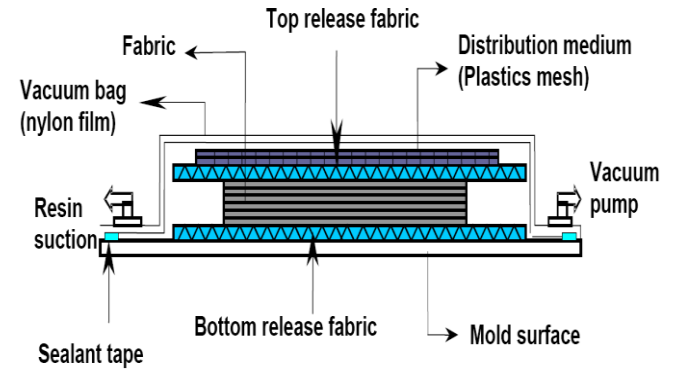


Figure 3: Schematic of VARTM

The laminas are sealed with vacuum bagging with sealant placing spiral tubing for uniform flow of resin. The vacuum pump is started on to expel air from the preform assembly. After the system has been equilibrated, the resin is allowed to flow into the preform. A pressure of 1 atm is maintained to provide driving force for the resin to impregnate the reinforcement and the compression force to compact the preform to the desired fiber volume fraction. The vacuum is left on until the resin has completely soaks in glass fiber laminate stack with NFs

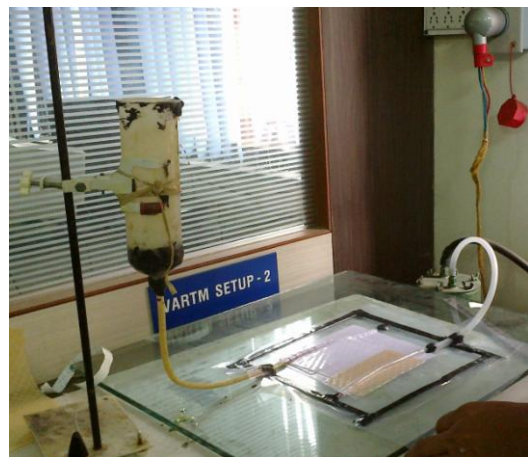


Figure 4: Actual VARTM setup at diagram BVUCOE,Pune

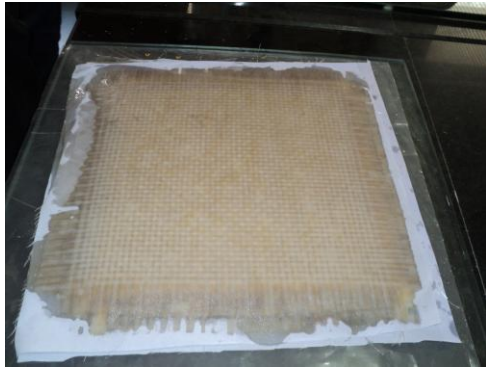


Figure 5: Hand molding setup



Figure 6: Electrospun Nylon 66 fibers on glass fiber 7781



Figure 8: Actual Setup at Praj Laboratory, Pune

### 3. Characterization

Short Beams Shear Strength ASTM D 2344. The specimen geometry and loading conditions during the SBS test using the machine are shown in figure 7. Specimen configurations for this research was a 10 layer three phase flat faced composite for which the recommended geometry was a rectangular specimen.

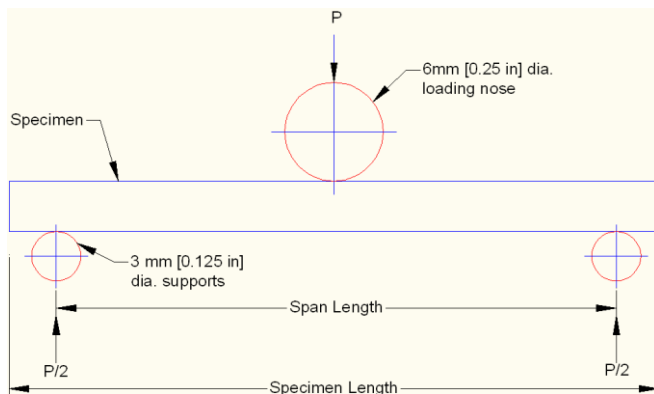


Figure 7: Specimen dimension as per ASTM D 2344

Data was recorded for load versus crosshead displacement, maximum load and final load till the failure of a specimen is reached. Finally, short beam strength was calculated as follows:

$$F_{sb} \equiv 0.75 \times \frac{P_m}{b \times h}$$

Where  $F_{sb}$  is the maximum interlaminar shear strength at failure,  $P_m$  is the maximum load,  $b$  is the specimen width and  $h$  is the specimen thickness. In this testing more than five samples were tested in each case.

### 3.1 Material System

Glass fiber 7781 by INTERGLAS Technologies AG, Benzstraße 14, D-89155 Erbach, +Epoxy(Epolam 5015 +30 % Epolam Hardener) from Axson technologies ,France +Nylon 66 nanofibers(1% by weight)

### 3.2 Testing

Testing was done in Praj testing laboratory, Pune using universal testing machine, Model-STS 248 as shown in the figure 8

### 4. Result and Discussion

Three different types of composites coupons were fabricated for unmodified and modified nanocomposites. Specimen was marked as H-SBS Neat for hand molded without fiber, H-SBS Nano for hand molded with nano fiber, V-SBS Neat for VARTM molded without fiber, V-SBS Nano for hand molded with nano fiber and V Modified –SBS Nano with nano fiber and resin added at interface before the VARTM.

Composite structures in the service cycle might encounter high stresses which lead in crack propagation through fiber matrix interfaces. Hence, stronger adhesion between fiber and matrix, higher strength, and resistance to delamination are desired in composite material design.

The short beam shear method was then used to measure interlaminar shear strength of fiber glass reinforced composites [4]. The short beam shear tests were performed on the Hand molding without nanofiber, Hand molding with nanofibers ,VARTM without nanofibers ,VARTM with nanofibers and VARTM with modified interface with rich resin zone shown in table 1 and 2.

VARTM with nanofiber is less as compared with Hand molding with nanofibers due to starvation of resin at interface or less resin at interface.

ILSS test results are presented in Table No 1 and 2. Higher ILSS was observed in Modified –SBS Nano with nano fiber compared to conventional composites probably due to the initiation of crack arrest at interface with rich resin zone.

The interlaminar shear strengths obtained in the three point bending test comparison is shown in figure 8 , shows that the ILSS of the VARTM with modified interface with rich resin zone composite is higher than that of the VARTM with nanofibers composite, VARTM without nanofibers , Hand molding without nanofiber and Hand molding with nanofibers.

Due to presence of the nylon 66 nanofibers at interface the load transfer is easily done without matrix failure.

**Table1: SBS test results for Hand Molding**

Sr. No	Sample	Thick ness (mm)	Width (mm)	Load (N)	SBS (MPa)	Avg SBS (MPa)
1	H-SBS Neat1	3.45	6.93	750	23.52	23.50
2	H-SBS Neat2	3.48	6.99	765	23.58	
3	H-SBS Neat3	3.48	6.99	771	22.77	
4	H-SBS Neat4	3.48	6.97	769	23.77	
5	H-SBS Neat5	3.47	6.99	771	23.84	
6	H-SBS Nano1 (1%)	3.81	7.65	1010	25.98	26.06
7	H-SBS Nano2 (1%)	3.85	7.74	1040	26.17	
8	H-SBS Nano3 (1%)	3.85	7.73	1033	26.03	
9	H-SBS Nano3 (1%)	3.83	7.70	1021	25.96	
10	H-SBS Nano3 (1%)	3.82	7.69	1025	26.16	

H-SBS Neat –Hand molded coupons without nanofibers

H-SBS Nano –Hand molded coupons with nanofibers

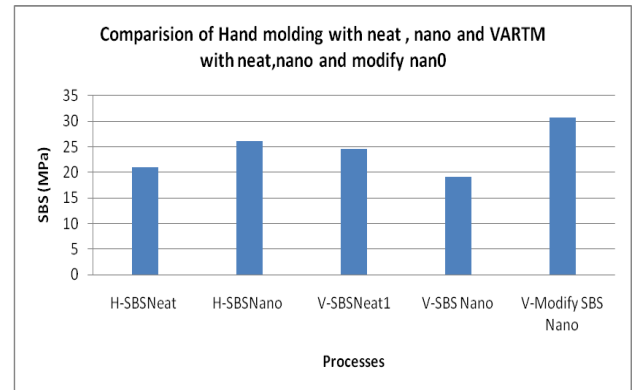
**Table 2: SBS test results for VARTM for 1200nm nanofiber**

Sr. No	Sample	Thickness (mm)	Width (mm)	Load (N)	SBS (MPa)	Avg SBS (MPa)
1	V-SBS Neat1	3.30	7.52	750.68	22.68	23.34
2	V-SBS Neat2	3.29	7.50	766.15	23.28	
3	V-SBS Neat3	3.29	7.53	780	23.61	
4	V-SBS Neat4	3.28	7.50	778	23.71	
5	V-SBS Neat5	3.27	7.49	765	23.42	
6	V-SBS Nano1(1%)	3.63	7.52	753.62	20.70	19.04
7	V-SBS Nano2(1%)	3.54	7.51	678.16	19.31	
8	V-SBS Nano3(1%)	3.50	7.50	677	19.34	
9	V-SBS Nano4(1%)	3.51	7.48	665	18.99	
10	V-SBS Nano5	3.55	7.53	600.74	16.85	
11	V-Modify SBS Nano1(1%)	3.56	7.51	950.45	26.66	27.18
12	V-Modify SBS Nano2(1%)	3.55	7.53	966.78	27.12	
13	V-Modify SBS Nano3(1%)	3.56	7.51	975	27.24	
14	V-Modify SBS Nano4(1%)	3.54	7.48	970	27.47	
15	V-Modify SBS Nano5(1%)	3.55	7.50	973	27.40	

V-SBS Neat –VARTM molded coupons without nanofibers

V-SBS Nano –VARTM molded coupons with nanofibers

V-Modify SBS Nano – Modified VARTM molded coupons with nanofibers



**Figure 9: Comparison of VARTM and hand molding results for SBS**

## 5. Conclusion

- The effect of electrospun nanofibers (ENFs) on interlaminar shear strength of plain weave woven glass fiber reinforced epoxy composites was investigated with hand molding, VARTM and Modified VARTM. Modified VARTM enhanced interlaminar shear strength of glass fiber composites.
- With introducing up to 1 %wt. of nanofibers nylon 66 at interface have shown up to 9.8% increase in the interlaminar shear strength compared to unmodified case of for hand molding.
- Enhancement of 14.12 % ILSS with modified VARTM compared to neat VARTM.

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