

Study on the repeatability of manufacturing nano-silica (SiO_2) reinforced composite laminates

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Abstract. Repeatability to manufacture nano-silica reinforced composite laminates with consistent mechanical properties is studied. In this study, composite laminates are manufactured by hand layup and there after mechanical properties of the laminates are evaluated under tensile and flexural loading conditions. Composite laminates are fabricated and tested under equivalent conditions. Plain weave E-Glass fabric and epoxy LY556 are used as reinforcement and matrix. Nano-silica of size 17nm is used as filler. To enhance the reliability of composite characterization, utmost care is taken to avoid defects like voids, surface defects and under-saturations. Homogeneous distribution of nano silica in matrix is analyzed using TEM study. Inconsistencies in mechanical properties are quantified by coefficient of variation. In this study, the coefficient of variation is estimated in terms of break load for tensile test is 4.45 and for flexural test is 2.27 and is well within the limits.

1. Introduction

The requirement of advanced materials has increased drastically in the recent decades and this lead to the development of composite materials. These novel materials offer superior properties when compared to conventional materials in terms of stiffness, strength to weight ratio and are economical. Synthetic fibers like glass fiber, carbon fiber, Kevlar, nomex etc., are few widely used materials for various engineering applications. However all materials have their own draw backs. Research is focused on resolving these drawbacks to manufacture superior composite materials. Glass fiber and Epoxy based matrix composite material is found to be a potential material to replace traditional materials. Structural modification of epoxy is one of the novel techniques to enhance the mechanical properties of the composite materials. This is achieved by reinforcing the matrix with micro and nano sized filler materials. [1] Used alumina powder as filler material in epoxy matrix and studied the effect of adding alumina powder on mechanical properties. Parameters like particle size, shape and even distribution of filler material in matrix displayed some effect on mechanical properties of the composites. Also the density of matrix used and concentration of filler in matrix are other parameters that may alter the overall performance of the composite. [2] Reinforced matrix with nano alumina as filler particles. Nano indentation technique is used to access the mechanical properties of composite laminates. Nano particles had the ability to resist the surface defects like scratches and wear. Failure mechanism of nano particles reinforced composite materials is presented in [3]. It is observed that nano particles enhance the fiber matrix bonding by impregnating the voids in the fiber. This leads to slower crack propagation as path of void growths are either hindered or diverted by the nano particles.



[4] Identified nano silica as a potential filler to reinforce epoxy matrix. Such a modified composite material displayed superior mechanical properties than the unmodified composite. It is recorded that the elastic moduli of modified composites are superior to neat composites. Likewise, a similar study is presented in [5] which discuss the effect of highly dispersed nano fillers. Significant improvement in mechanical properties of the composite materials is recorded. [6] Studied the effect of different nano sized silica fillers on mechanical properties of composite laminates. It is observed that with the reduction in the size of the nano filler, there is an increase in the mechanical properties. This is because with the reduction in the size of the nano particle, the interfacial bonding increases and the tendency to form agglomeration is reduced. Hand layup, compression molding, vacuum molding and resin transfer molding are some techniques used to manufacture composite materials. Hand layup technique is an economical method to manufacture composites. [7] Studied the repeatability in manufacturing composite laminates using various processes.

In this study, an attempt is made to manufacture nano silica reinforced composite laminates with consistent mechanical properties using hand layup technique.

2. Experimental

2.1. Materials

Plain weave E Glass fabric and epoxy LY556 along with the hardener HY951 are used as reinforcement and matrix in this study. All the materials are purchased from M/s. Sakhti fiber Glass Ltd, Chennai, Tamil Nadu, India. Nano-silica of size 17 nm is used as filler. The properties of E-glass fabric, epoxy and nano silica are listed below.

2.1.1 Properties of E-glass fabric[8]

Grade	: GSM610
Density (g/cm ³)	: 2.55
Diameter (μm)	: 15 - 25
Tensile strength (MPa)	: 2000 - 3500
Young modulus (GPa)	: 70 - 73
Elongation at break (%)	: 2.5 - 3.7

2.1.2 Properties of epoxy LY556

Density (g/cm ³)	: 1.15
Tensile strength (MPa)	: 80 - 95
Tensile modulus (GPa)	: 0.3 - 0.6

2.1.3 Properties of nano-silica

Particle size (nm)	: 17
Specific surface area (m ² /g)	: 202
Ph. value	: 4.12
Tamped density (g/Ltr.)	: 44
SiO ₂ content (%)	: 99.88
Carbon content (%)	: 0.06
Chloride content (%)	: 0.009
Al ₂ O ₃ (%)	: 0.005
TiO ₂ (%)	: 0.004
Fe ₂ O ₃ (%)	: 0.001

2.2. Fabrication Technique

Conventional hand layup technique is used to fabricate laminates having dimensions 150x300 mm. Six layers of plain weave glass fabric is used to fabricate composite laminates of 2.6 mm thick. Hand layup method is used to fabricate the composite laminate required for this study. A 12mm thick toughened glass panel is used as work table. Releasing agent like wax or poly-vinyl alcohol is applied over the glass to enable easy removal of the composite laminate. Thereafter a homogeneous mix of resin-hardener is prepared in the ratio 10:1. A thin layer of this mix is applied over the glass and a layer of glass fabric is laid over it. Excess resin is removed with the help of a roller. This process is repeated for six layers of glass fabric. Total quantity of resin required is estimated using following expression.

$$W_R = (W_F/60\%) * 40\%$$

where,

W_R is the weight of resin required (g)

W_F is the weight of the reinforcement fiber (g)

The laminate is then allowed to cure in room temperature under compression. The fiber volume fraction in the fabricated laminate is assessed using the following expression.

$$V_F \% = (M_F/M_L) * 100$$

where,

V_F is the fiber volume fraction (%)

M_F is the mass of reinforcement (g)

M_L is the mass of laminate (g)

In this study, the fiber volume fraction of fabricated composite laminates is estimated as 69.23 %. Samples for tensile and flexural tests are cut from the same composite laminates as per ASTM D638 and ASTM D790. All the specimens are cut from the same laminate as per the scheme shown in Figure 1. Five samples are cut for tensile test and five for flexural test. A CNC operated abrasive water jet cutting machine shown in Figure 2 is used to cut the samples from the laminate since delamination is a major peril in conventional cutting techniques.



Figure 1. Laminate cutting scheme



Figure 2. CNC operated AWJM

The cutting parameters are listed below:

Pressure (bar)	: 3700
Cutting Speed (mm/min)	: 1200
Abrasive flow (gram/min)	: 700
Abrasive grade	: Garnet 80 mesh
Nozzle Diameter (mm)	: 1.1
Orifice (mm)	: 0.35
Working Distance (mm)	: 3
Jet Range (mm)	: 1.5 – 2

Figure 3 and Figure 4 display the laminate samples used for tensile and flexural testing.

**Figure 3.** Tensile Test samples**Figure 4.** Flexural Test samples

2.3 Testing Methods

All the tensile tests are carried out on a same day at 20°C using a UK make Instron 3382 with 100 KN load cell according to ASTM: D638. A wedge type grip (Figure 5. b) is used to hold and prevent the specimen from slipping. Speed of upper crosshead is set to 5 mm/min for all the tests. The tensile properties such as break load, tensile strength and failure strain are determined as per the standard.

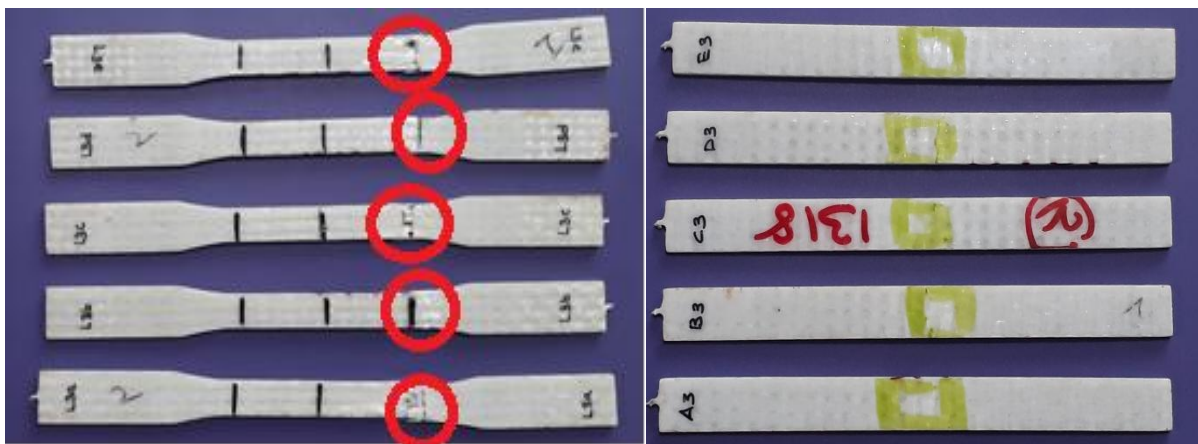
**Figure 5.a.** Instron 3382 UTM**Figure 5.b.** Wedge type grip

Flexural tests are carried out using a Shimadzu Autograph AG-IS equipment fitted with 50KN load cell and a specially designed holding module (Figure 6. b) to hold the sample. The speed of the cross head is set to 2 mm/min.

**Figure 6.a.** Autograph AG-IS**Figure 6.b.** Holding module

3. Results and discussion

The failed samples in tension and flexural loading are shown in Figure 7.a, b. It is evident from the results that failure has occurred at same region in tension and flexural for all five tests. This is due to minimum defects like under saturation, air bubbles and structural defects. Elimination of such defects has an effect on the mechanical properties of the composite laminate. Uniform thickness of the laminate also plays a vital role in estimating the repeatability of results. The composite laminates produced by the hand lay-up technique displayed good quality during visual evaluation.

**Figure 7.a.** Failed tensile samples**Figure 7.b.** Failed flexural samples

Following are the results of tensile tests.

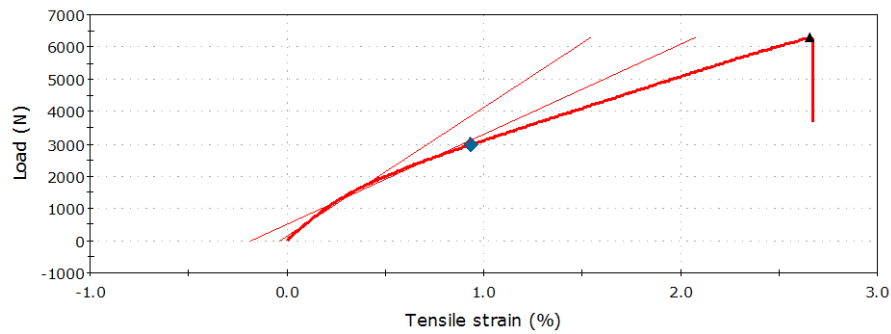


Figure 8.a. Load vs strain for sample L3a

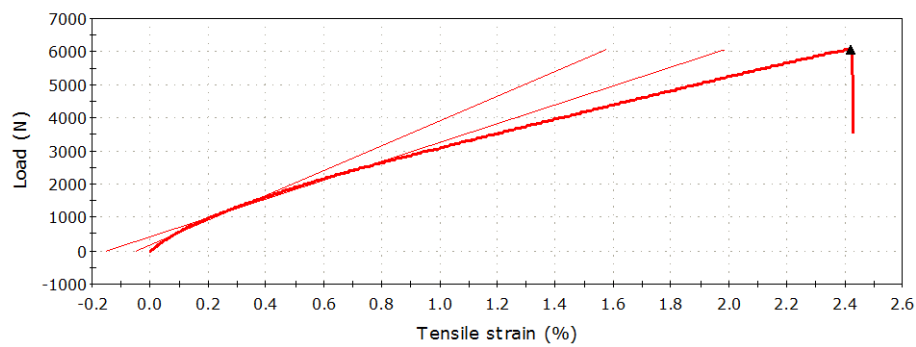


Figure 8.b. Load vs strain for sample L3b

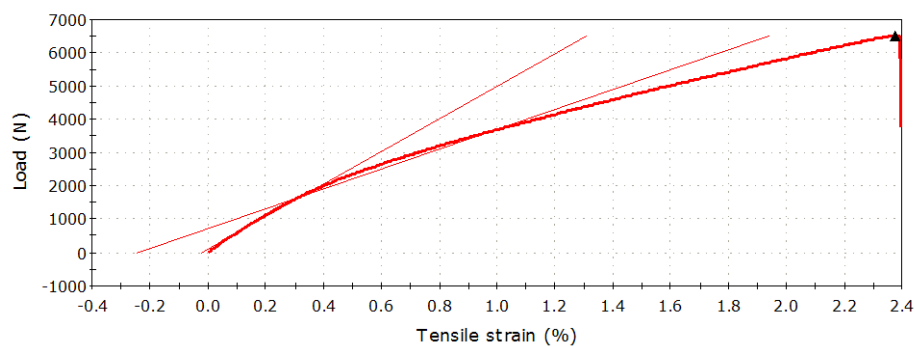


Figure 8.c. Load vs strain for sample L3c

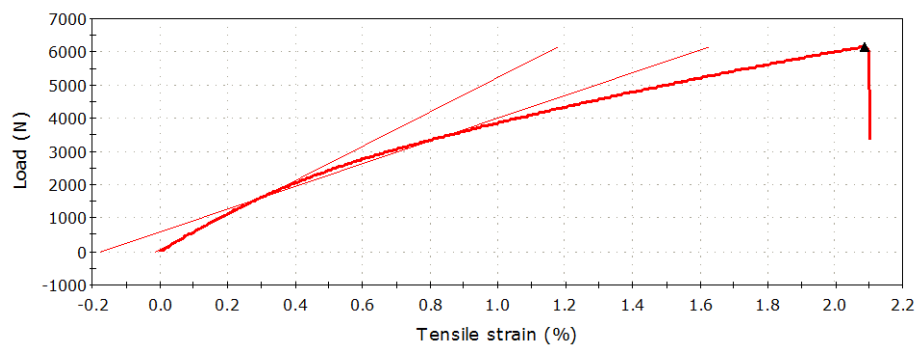


Figure 8.d. Load vs strain for sample L3d

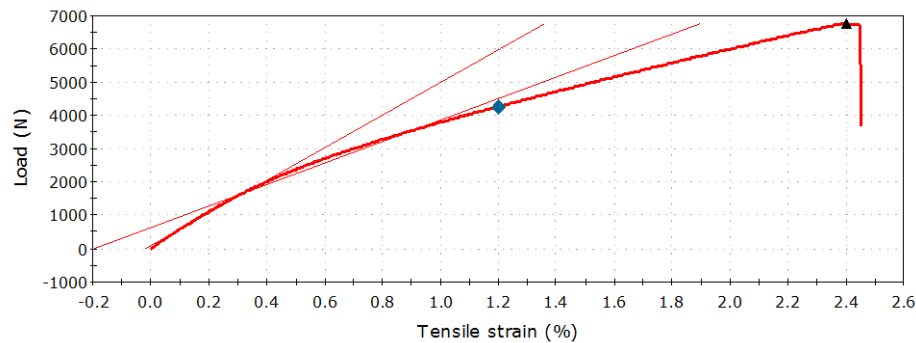


Figure 8.e. Load vs strain for sample L3e

Table 1. Tensile properties 3% nano silica-GFRP composite laminates

Sample Identification	Weight of sample (g)	Thickness (mm)	Break Load (N)	Strain (%)	Ultimate Tensile Strength (MPa)
L3a	13	2.69	6284	2.68	178
L3b	12	2.62	6065	2.42	
L3c	13	2.64	6524	2.40	
L3d	13	2.69	6148	2.11	
L3e	13	2.69	6755	2.44	

Table 2. Flexural properties 3% nano silica-GFRP composite laminates

Sample Identification	Weight of sample (g)	Thickness (mm)	Break Load (N)	Stroke (mm)	Ultimate Flexural Strength (MPa)
A3	8	2.69	331.14	3.22	281
B3	8	2.66	318.30	3.10	
C3	8	2.62	325.57	3.16	
D3	8	2.69	320.70	3.37	
E3	9	2.64	311.81	2.95	

Coefficient of variation

Coefficient of variation is a relative measure which depends on the arithmetic mean value. The coefficient of variation C_V , is assessed using the following expression.

$$C_V\% = \sigma/\sigma_x * 100$$

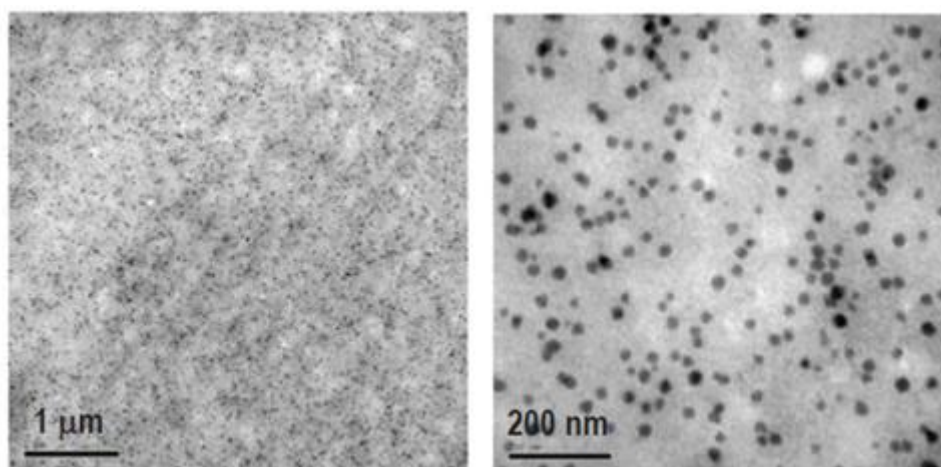
where,

σ is the standard variation of results

σ_x represents the mean value of results

In this study, the coefficient of variation is estimated in terms of break load for tensile test is 4.45 and for flexural test is 2.27 and is well within the limits.

One of the major challenges in reinforcing nano-silica in epoxy is to achieve homogeneous dispersion. Poor dispersion leads to poor mechanical properties and poor interfacial bonding of matrix and reinforcement filler. In this study, the homogeneous dispersion of nano-silica in epoxy matrix is witnessed with the help of TEM. No agglomeration of nano silica is reported for 3% concentration.

**Figure 9. a.** 22500x**Figure 9. b.** 115000x

4. Conclusion

An attempt is made to manufacture nano-silica reinforced composite laminates with consistent mechanical properties using hand layup technique. Repeatability to manufacture nano-silica reinforced composite laminates with consistent mechanical properties is studied. In this study, composite laminates are manufactured by hand layup and there after mechanical properties of the laminates are evaluated under tensile and flexural loading conditions. Results report that due to the absence of defects, consistency in mechanical properties is achieved. Also TEM study reveals that homogeneous dispersion of nano fillers is possible even in hand layup technique and there is no nano particle agglomeration in the matrix. Inconsistencies in mechanical properties are quantified by coefficient of variation. In this study, the coefficient of variation is estimated in terms of break load for tensile test is 4.45 and for flexural test is 2.27 and is well within the limits.

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