Production of Nano-Antimony Oxide-filled Polymer-based Glass Fiber Composites with Enhanced Interlaminar Shear Strength (ILSS)

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Abstract: Interlaminar shear strength (ILSS) of fiber-reinforced polymer (FRP) composites is one of the most important mechanical properties desired by various structural applications. This work has developed a three-phase (consisting of glass fibers, a polymer matrix, and antimony oxide nanoparticles as additional reinforcements) composite material with superior interlaminar shear strength (ILSS) suitable for structural applications. Initially, antimony oxide nanoparticles are synthesised by the solution combustion method using antimony nitrate as fuel and parthenium as an oxidizer. The synthesised antimony oxide nanoparticles were characterised using XRD, SEM, and EDX. Further, the nanoparticles are uniformly dispersed in the diglyceryl ether of bisphenol(DGEBA).A diglycidyl ether of bisphenoland triethylenetetramine (TETA) systems, and the resulting mixture is used along with the glass fibers to develop composite laminates by vacuum bagging. The specimens were cut from the developed composite laminates, and the interlaminar shear (ILSS) tests were conducted. Test specimens cut from unmodified matrix-based laminates are also tested to obtain baseline data. The results showed a significant enhancement in the ILSS of the nano-modified FRP composite due to the addition of antimony oxide nanoparticles in the matrix.The fractured surfaces of the fiber-reinforced composites are also examined by the scanning electron microscope to understand the failure mechanisms.

Keywords: Nano-Antimony Oxide, Glass Fiber, Polymer Matrix,Interlaminar Shear Strength.

1 Introduction

Polymer-based fibre Reinforced composite materials are highly desired materials for various engineering applications, particularly as structural materials in aircraft, marine vessels, and automobiles. Thus, the enhancement of their mechanical properties is a key issue that must be addressed by the researchers working in the field of polymer science and engineering [1,2].Nanomaterials are highly regarded as potential filler materials for the polymer due to their high specific surface areas (SSA). Nano-polymer composites have superior mechanical and physical properties over host polymers, partially due to the large interfacial area between polymers and nano-

fillers [3-5]. Much research in polymer sciences and engineering has been inspired by the SSA of several nano-sized filler components. Nano-polymer composites have been studied for decades to understand the modifying effects of various nanomaterials on the mechanical and physical properties of polymer nanocomposites [6-9].

Few authors have demonstrated the potential improvement in the properties and performances of polymer-based composites in which nanomaterials are used as reinforcements with various polymer matrices. Furthermore, due to their high SSA, nanomaterials such as CNTs, nanoparticles, and nanoplatelets have been found to be highly efficient as nanoscale reinforcements in polymeric systems [10-14].

However, due to the very high superior properties of conventional fibers, it is considered impractical to replace conventional fibers with nano-sized fillers in polymer-based composites [15]. Hence, the production of conventional fiber-reinforced polymer-based composites in combination with nano-sized fillers has led to the development of a new class of material suitable for multi-functional applications. FRP-nanocomposites showed enhanced mechanical properties even at very low loadings of nanofillers. However, efficient FRP nanocomposites can only be created by achieving a high degree of nanoscale material dispersion in the polymer. The enormous specific surface area (SSA) of the nano-fillers has made them a superior and unique material that can act as an interface material between the matrix and the fiber for stress transfer [16,17].

The nanofillers used in the development of FRP-nanocomposites are of various types such as nanofibers, nanoplatelets, nanoclays and nanoparticles with the dimensions ranging between 1–100 nm. Several methods and techniques are developed by the researchers for processing and characterization of FRP-nanocomposites. A very few authors have demonstrated the potential improvement in the properties and performances of polymer-based fiber reinforced composites by the inclusion of nanomaterials as additional reinforcements [18-21].

This paper describes the use of antimony oxide nanoparticles as reinforcing fillers in the manufacture of traditional glass-fibre reinforced polymer hybrid composites. In this work, two sets of composite laminates are fabricated: a) neat FRP composites and b) nano-modified FRP composites, and their interlaminar shear strengths are evaluated and compared. The inter-laminar shear strength (ILSS) of nano-modified FRP composite is found to be superior to the inter-laminar shear strength (ILSS) of nano-modified FRP composite. This work demonstrates an example of a nano-modified FRP composite

laminate in the dimension of a structural element. The production of nano-modified FRP composite laminate via vacuum bagging and the investigation of the resulting interlaminar shear strength properties are discussed.

2 Experimentation

2.1 Materials:

A mixture of ten parts by weight of diglycidyl ether of bisphenol (DGEBA) and one part by weight of triethylenetetramine (TETA) is used as matrix material. Unidirectional (UD) glass fibers in the form of fabric sheets of 220 GSM are used as conventional reinforcements.

Antimony oxide nanoparticles are synthesised by the solution combustion method. The stochiometric and homogeneous mixture of antimony nitrate and parthenium plant extract is dissolved in distilled water and kept in the muffle furnace maintained at 400ºC. After 4-5 minutes of combustion, Sb_2O_3 nanoparticles were formed.

The solution initially boils and undergoes dehydration, followed by decomposition and the liberation of gases. The entire smouldering type of the combustion process takes place in 4-5 minutes, and Sb_2O_3 nanoparticles were obtained.

2.2 Production of Composite laminates:

Antimony oxide nanoparticles (1.0 wt.%) were manually dispersed in the base resin and stirred strongly using a mechanical stirrer with a speed of 300 rpm for about 300 seconds. The solution was also subjected to ultrasonication for 180 seconds at 5 different locations to break up the agglomerations of the dispersed nanoparticles. The entrapped air is removed by degassing for 300 seconds. The hardener, triethylenetetramine, was then mixed in the nano-modified base resin and stirred uniformly.

Both a) neat FRP composite and b) nano-modified FRP composite are produced by hand layup. In the process, the glass-fibre sheets are placed in the mould by hand, and a neat (unmodified) epoxy matrix is applied with a brush. The sheets are laid up one above the other to obtain the required thickness. The wet laminate is covered with a vacuum bag, and the vacuum is pulled over it to remove the air bubbles. Similarly, nano-modified FRP composite laminate is also produced using an antimony oxide nanoparticle-filled epoxy matrix. The wet laminates were cured at the ambient temperature for 24 hours.

Test specimens were prepared by machining the laminates to the required dimensions. Care is taken to avoid delamination of fibers from the test samples. The specimens are sanded using 150-grit sandpaper

to obtain a fine finish. Table 1 gives details of two sets of composite laminates fabricated: a) neat FRP composites and b) nano-modified FRP composites. The ILSS of two sets of composite laminates, a) neat FRP composite and b) nano-modified FRP, is measured and recorded using the 3-point short beam test according to the guidelines of the ASTM D-2344 standard. .

Table 1 gives details of two sets of composite laminates fabricated:

Specimen no.	Specimen Type	Glass fibre (wt. %)	Epoxy (wt. %)	Antimony oxide nanoparticles (wt. %)
1	Neat FRP Composite	48	52	0.0
2	Nano-modified FRP composite	48	51	1.0

3 Results and Discussion

In order to understand the crystal structure and phase of the synthesised antimony oxide nanoparticles, X-ray diffraction analysis was performed. The obtained XRD spectrum is shown in Figure 1, and the diffraction peak was observed at 10.4°, 26.3°, 30.1°, 50.1°, and 60°, indicating the characteristic peaks of Sb_2O_3. Also, no other phases were seen in the spectrum.

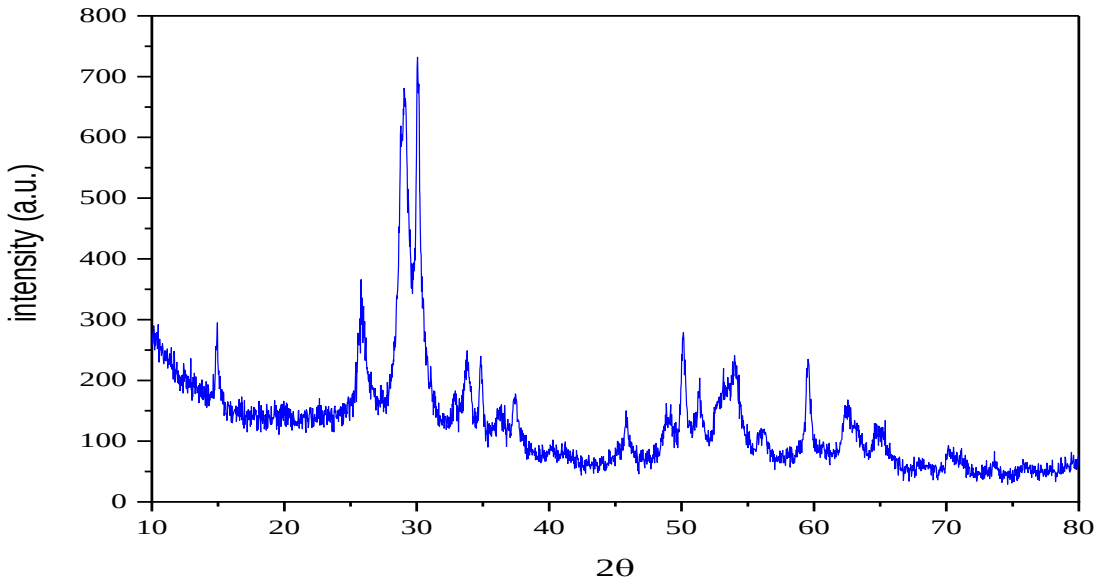


Fig.1 XRD spectrum of synthesised antimony oxide nanoparticles

Figure 2 shows the SEM micrographs of the synthesised antimony oxide nanoparticles. The SEM image clearly shows that the compound is highly porous and gives out a large surface area, due to

which it can be efficiently used for mechanical applications. Nanoparticles have the shape of rice flakes, and each nanoparticle's size ranges from 40 nm to 77 nm.

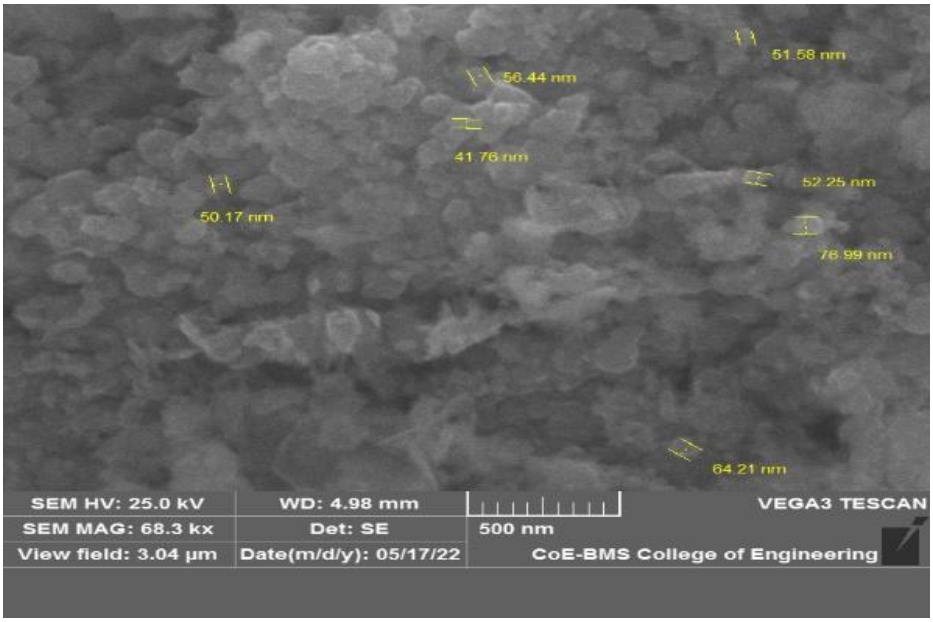
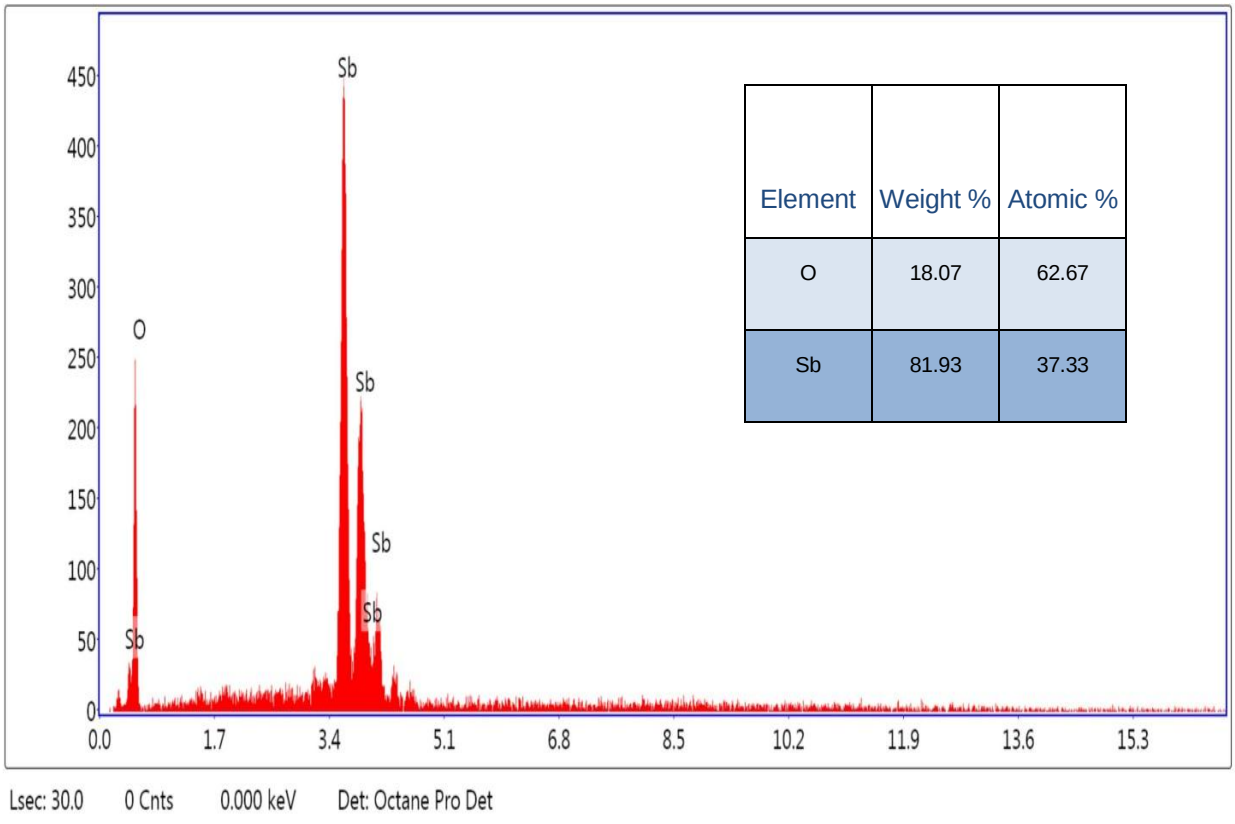


Fig.2SEM image of synthesised antimony oxide nanoparticles

The results of the EDX analysis are shown in Figure 3. EDAS analysis shows that the synthesised material is pure and no other element is found except Sb and O. The inside table shows the elemental percentages of Sb and O. According to the study, the weight percents of the elements O and Sb are 18.07 and 81.93, respectively, and the atomic percents are 62.67 and 37.33.

Fig. 3 EDX analysis of synthesised antimony oxide nanoparticles



Element	Weight %	Atomic %
O	18.07	62.67
Sb	81.93	37.33

antimony oxide nanoparticle-filled epoxy resin is used to develop the laminates by hand-layup method, which are then subjected to vacuum bagging. A small amount of increment is observed in the viscosity of the nano-modified resin. The composite laminate was successfully developed using nano-modified (containing 1.0 wt. % antimony oxide nanoparticles) epoxy resin with glass fibers. To generate baseline data, a composite panel was also developed using neat epoxy resin and glass fibers.

The ILSS of two sets of composite laminates, a) neat FRP composite and b) nano-modified FRP composite, is measured by the 3-point short beam test. The ILSS of a) neat FRP composite and b) nano-modified FRP are found to be 43.12 ± 0.13 MPa and 48.46 ± 0.14 MPa, respectively. In relation to the Neat FRP Composite, the nano-modified FRP exhibits an improved ILSS of 12.38% for 1.0 wt% antimony oxide nanoparticle content. Figure 4 shows SEM micrographs of the fractured specimens of (a) neat FRP composite and (b) nano-modified FRP composite.

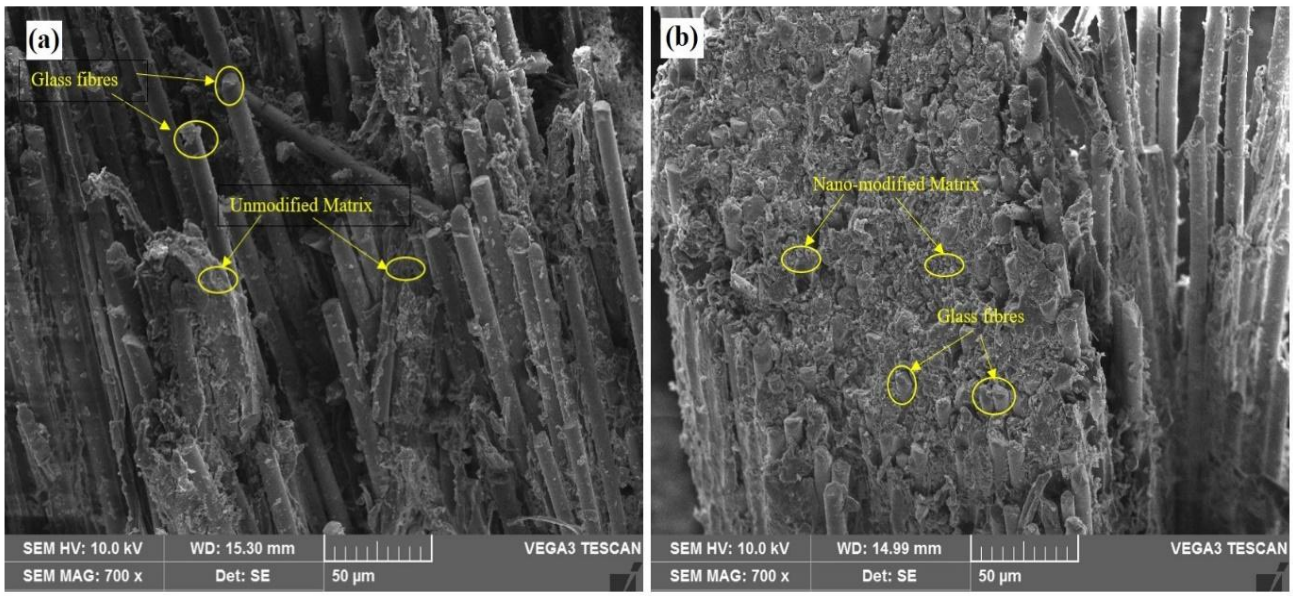


Fig. 4 shows SEM micrographs of the fractured specimens of (a) neat FRP composite and (b) nano-modified FRP composite.

A SEM micrograph of the fractured surface of the nano-modified FRP composite sample (Fig. 4.b) shows good fiber-matrix bonding compared with the neat FRP composite (Fig. 4.a) sample. The presence of antimony oxide nanoparticles in the matrix seems to have improved the bonding ability of the matrix with the fibers. The improved ILSS of the composite is due to two factors: the increased load-bearing capacity of antimony oxide nanoparticles and the strong bonding between the fibers and matrix. SEM micrographs of the fractured specimen of nano-modified FRP composite appear to be

rougher, showing a higher energy requirement for the propagation of the crack. Because of weaker fiber and matrix bonding, samples prepared from unmodified/neat matrix showed extensive de-bonding between fibers and matrix. A SEM micrograph of the fractured surface of a nano-modified FRP composite sample (Fig. 4.b) shows less matrix cracking and fiber pull-out. In a nano-modified FRP composite sample, the huge specific surface areas (SSA) of the antimony oxide nanoparticles seem to have arrested the crack propagation. The enormous specific surface area (SSA) of the antimony oxide nanoparticles seems to have acted as a strong interface material between the matrix and the fiber for stress transfer, resulting in the enhanced ILSS of the composite.

4 Conclusion

SEM micrographs of the synthesised antimony oxide nanoparticles clearly showed that the compound is highly porous and gives out a larger surface area, due to which it can be efficiently used for mechanical applications. From SEM micrographs, the particle size of the antimony oxide was also seen to be between 40 nm to 77 nm, depending on the flake shape. EDAS analysis showed that the synthesised material is pure and no other elements are found except Sb and O.

The synthesised antimony oxide nanoparticles are uniformly dispersed in the diglyceryl ether of bisphenol A (DGEBA) / triethylenetetramine (TETA) systems, and the mixture is used along with the glass fibers to develop composite laminates by vacuum bagging. A small amount of increment is observed in the viscosity of the nano-modified resin. The composite laminate was successfully developed using nano-modified (containing 1.0 wt.% antimony oxide nanoparticles) epoxy resin with glass fibers. To generate baseline data, a composite panel was also developed using neat epoxy resin and glass fibers.Thenano-modified FRP composite showed enhanced ILSS due to the inclusion of 1 wt.% of antimony oxide nanoparticles in the epoxy resin.

The fractured test specimens were studied by the scanning electron microscope (SEM) to understand the failure mechanisms. A SEM micrograph of the fractured surface of a nano-modified FRP composite sample shows good fiber-matrix bonding compared with a neat FRP composite sample. The presence of antimony oxide nanoparticles in the matrix seems to have improved the bonding ability of the matrix with the fibers. A SEM micrograph of the fractured surface of a nano-modified FRP composite sample shows less matrix cracking and fiber pull-out. In a nano-modified FRP composite sample, the huge specific surface areas (SSA) of the antimony oxide nanoparticles seem to have arrested the crack propagation, resulting in the enhanced ILSS of the composite.

Conflict of Interest

The authors declare that there is no conflict of interests regarding the publication of this paper.

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