



Water absorption behavior, mechanical and thermal properties of nano TiO₂ enhanced glass fiber reinforced polymer composites



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ABSTRACT

Nano TiO₂ particle is one of the promising inorganic nano fillers used in polymer matrix composites to enhance the mechanical properties. However, reliability of this type of nano composites is yet to be ensured in hydrothermal environment. The present work investigates the addition of nano TiO₂ filler on water sorption, residual strength and thermal properties of glass fiber reinforced polymer (GFRP) composites. The results revealed that addition of 0.1 wt% TiO₂ has reduced water diffusion coefficient by 9%, improved residual flexural strength by 19% and residual interlaminar shear strength by 18% among all the nano TiO₂ modified composites. The improvement of mechanical properties in hydrothermal environment creates opportunity and reliability to be used in different engineering applications. Weibull design parameters are evaluated and found a good agreement between Weibull stress-strain curves and experimental one. Fractographic analysis confirmed the various failures and strengthening mechanisms of nano composites in dry and hydrothermal environment.

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1. Introduction

Fiber reinforced polymer composites (FRP) are used in different sectors like automotive, railway, aerospace, aircraft, marine, wind, and etc. due to its desirable physical and mechanical properties over traditional metallic and non metallic materials. However, these materials are facing challenges and threats at different environments like high and low temperature, water, hydrothermal, alkaline, corrosive, and UV light exposure. Generally, polymer matrix composites absorb moisture in hydrothermal environment. The absorbed moisture/water molecules in the polymer composites are two types: bound and free water. Bound water usually bonded with the hydroxyl group of epoxy chemically and free water is clustered in the free volume/voids present inside the epoxy or at the matrix fiber interface [1,2]. Moisture absorbed composites deteriorate its physical, thermal, electrical and mechanical properties. Physical change of epoxy is basically due to plasticization and swelling and chemical change of epoxy is due to chain scission and hydrolysis [3,4]. Glass transition temperature (T_g) is affected by physical change (plasticization) of epoxy and hydrolysis of polymer cross linking [5,6]. Swelling reduces the interface bond strength, resulting in reduction in mechanical

properties especially interlaminar shear strength, flexural strength and modulus. Hence, mechanical properties are affected by physical, chemical structure change of epoxy. Overall, moisture absorption leads to change in thermo-physical, mechanical and chemical characteristics of FRP composites [7]. Therefore, retention of the mechanical and thermal properties of hydrothermally conditioned composites is a challenge for materials engineers and researchers.

Gautier et al. [8] observed that matrix microcrack formed due to interfacial debonding, differential swelling and osmotic cracking at the interphase in hydrothermal environment. Similar observation also cited by Hodzic et al. [9]. Ellyin and Maser [10] observed that water temperature enhances the damage in matrix and fiber-matrix interface. This might be possibly due to either leaching out of the glass fiber interface layer or matrix plasticization and hydrolysis of polymer. Huang and Sun [11] observed that rate of water absorption increases with prolonged immersion time because of capillary action and absorption of hydrophilic group in the glass fiber and unsaturated polyester. Therefore, the overall mechanism for deterioration of mechanical properties of FRP composites in hydrothermal environment are matrix swelling, interphase debonding, and hydrolysis of epoxy.

Fan et al. [12] observed that the one of the probable solution to close the pores/voids and improvement of interface/interphase strength is by adding nano fillers in GFRP composites. Among the different nano fillers, inorganic nano fillers are the most promising

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because of their availability, low fabrication cost and readily optimization of mechanical and thermal properties at the design stage [13,14]. Inorganic fillers are mixed with polymer matrix either in disperse form or mechanically contacted or chemically bonded or combination of two or all mechanism as shown in Fig. 1 [15]. Among the investigated inorganic nano fillers, nano TiO₂ is the most promising fillers because of its unique properties like corrosion stability, improvement of mechanical properties, thermal stability, non toxicity and good compatibility with other materials [16–19]. Shi et al. [20] found that nano SiO₂ and TiO₂ improves the corrosion resistance.

The effect of nano TiO₂ filler on water absorption kinetics, mechanical and thermal properties of glass fiber reinforced polymer composites used in structural application have not been reported based on the existing literature so far. Therefore, an attempt has been made to investigate the effect of nano TiO₂ enhanced GFRP composites on moisture diffusion behavior, flexural strength, interlaminar shear strength and glass transition temperature. Furthermore, post failure analysis of fractured surfaces of ILSS and flexural samples are analyzed using scanning electron microscopy (SEM).

2. Materials and method

2.1. Material

Glass fiber (GF) reinforced polymer composites (GFRP) is fabricated using Diglycidyl ether of Bisphenol A (DGEBA) type of epoxy, Triethylene tetra amine (TETA) as hardener supplied by Atul Industries, India and woven roving E-glass fiber procured from Owens Corning, India. Nano GF composites are made of nano TiO₂ fillers, epoxy, hardener and woven fabric glass fiber. The TiO₂ nano fillers is supplied by SRL Industries limited, India. Some of the important properties of epoxy, E-glass fiber and nano particle are reported in Table 1. It is observed that nano TiO₂ particle has superior mechanical properties compared to neat epoxy and it is expected that the nano filler may enhance mechanical/thermal properties of the epoxy matrix and matrix/fiber interface.

2.2. Fabrication of nano composites

Nano TiO₂ is dried at 100 °C before it mixed with epoxy to remove moisture in it. Control GF composites are fabricated without nano fillers and nano GF composites are fabricated with different wt% of nano TiO₂ fillers. The fraction of fiber and epoxy is maintained at 60:40 ratios by weight for both control GF and nano GF composites during fabrication. As per the suppliers instruction 10% of epoxy of hardener is used for curing process. Researchers have reported that mechanical stirring followed by sonication is a good method to disperse nano fillers in polymer matrix [23–

Table 1
Properties of raw materials [21,22].

Properties	Epoxy	TiO ₂ (Rutile)	Glass fiber
Density (g/cm ³)	1.15	4.00	2.58
Tensile strength (MPa)	70	51.6	3800
Tensile modulus (GPa)	3.6	228	78
Poisson's ratio	0.30	0.27	0.20

[25]. In this work instead of mechanical stirring high speed magnetic stirring is adopted and followed by sonication. This is because, in mechanical stirring the probability of formation of bubbles is more and increases the void content. It is expected that magnetic stirring may reduce bubbles formation and reduce the void content. The epoxy, nano TiO₂ at different wt% is stirred by magnetic stirrer for one hour followed by sonication at 60 °C for another 45 min to disperse nano particles in the epoxy matrix. It is expected that the shear force developed between the nano particles helps the process of de-agglomeration of nano particles. Composite laminates are fabricated with 16 layers of woven fabric glass fiber by hand lay-up techniques followed by temperature assisted compression molding (pressure 10 kg/cm²) and at a temperature of 60 °C for 20 min. Further curing of composites is done at 140 °C for 6 h before characterization. Samples of different sizes are cut as per ASTM standard using diamond coated tipped cutter for further characterization.

3. Results and discussions

3.1. Morphology of TiO₂ nano particles

The shape and size of nano TiO₂ particles has been examined by field emission scanning electron microscope (FESEM). Fig. 2(a and b) shows the nano TiO₂ particles shape and XRD plot respectively. It is observed that the shape of the nano TiO₂ particles is nearly spherical and the purity level of nano particle is good. Nano particles are mixed with epoxy through magnetic stirring and sonication. Distribution of nano particles in epoxy matrix is observed through FESEM and found reasonable uniform distribution of nano particle in the epoxy matrix of the composites having 0.7 wt% of nano TiO₂ shown in Fig. 2c & d.

3.2. Void content

Void content is nothing but closed pores present in the composites, which plays the initial absorption of water into the composites and resulting reduction in mechanical properties. Void content of the GFRP composites are determined by resin burn off test. The fiber weight fraction and void content of each laminate is determined as per ASTM D 3171-99. As per the standard, there are six

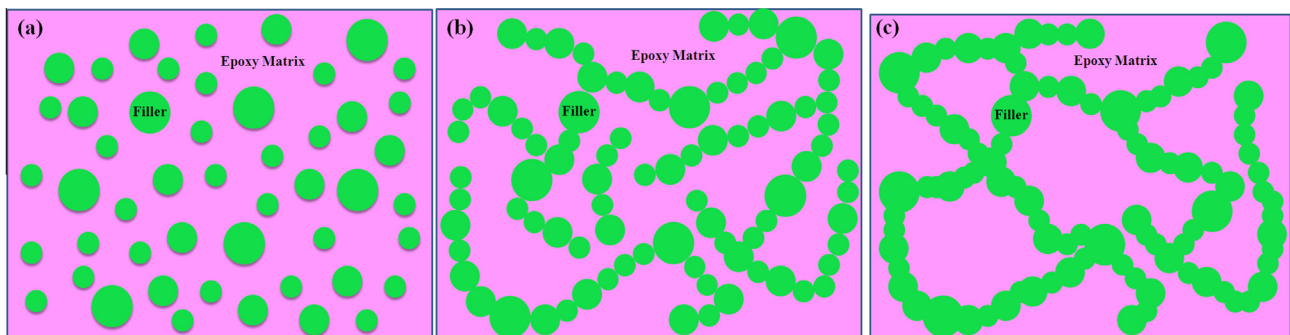


Fig. 1. Nano inorganic fillers interaction with epoxy matrix (a) dispersed, (b) mechanically entangled, and (c) chemically bonded [15]. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

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