Available online at www.sciencedirect.com

ScienceDirect

Procedia Materials Science 6 (2014) 1359 – 1364

Procedia Materials Science

www.elsevier.com/locate/procedia

3rd International Conference on Materials Processing and Characterization (ICMPC 2014)

# Effect of epoxy modifiers ($Al_2O_3/SiO_2/TiO_2$) on mechanical performance of epoxy/glass fiber hybrid composites

Ramesh K. Nayak [a*], Alina Dash[a] and B.C.Ray [b]

[a]*School of Mechanical Engineering, KIIT University, Bhubaneswar, 751024, India*

[b]*Department of Metallurgical and Materials Engineering, National Institute of Technology, Rourkela, 769008, India*

**Abstract**

Fiber reinforced polymer composite is an important material for structural application. The diversified application of FRP composite has taken centre of attraction for interdisciplinary research. However, improvements on mechanical properties of this class of materials are still under research for different applications. In this paper we have modified the epoxy matrix by $Al_2O_3$, $SiO_2$ and $TiO_2$ micro particles in glass fiber/epoxy composite to improve the mechanical properties. The composites are fabricated by hand lay-up method. It is observed that mechanical properties like flexural strength, flexural modulus and ILSS are more in case of SiO2 modified epoxy composite compare to other micro modifiers. This may be because of smaller particle size of silica compare to others. Alumina modified epoxy composite increases the hardness and impact energy compare to other modifiers. Agglomeration of Al2O3 micro particles in the matrix is observed in SEM. This may be because of bigger particle size of Alumina. SEM analysis clearly indicates the mode of failure is the combination of crack in matrix, matrix/fiber debonding and fiber pull out for all types of composites.

© 2014 Elsevier Ltd. This is an open access article under the CC BY-NC-ND license
(http://creativecommons.org/licenses/by-nc-nd/3.0/).
Selection and peer review under responsibility of the Gokaraju Rangaraju Institute of Engineering and Technology (GRIET)

*Key words:* Polymer Matrix Composite; Glass-fiber; Flexural strength; Micro fillers ($Al_2O_3$, SiO2, TiO2)

## 1. Introduction

Development of new composite materials or modification of existing composite material is the real challenge for most of the materials engineers. Epoxy base matrix composite has tremendous potential to substitute the traditional metallic materials. Polymer matrix modification is one of the approaches to develop new class of polymer structural

* Corresponding author. Tel.: +91 8763724080.
*E-mail addresses*: rameshkumarnayak@gmail.com (Ramesh K. Nayak)

2211-8128 © 2014 Elsevier Ltd. This is an open access article under the CC BY-NC-ND license
(http://creativecommons.org/licenses/by-nc-nd/3.0/).
Selection and peer review under responsibility of the Gokaraju Rangaraju Institute of Engineering and Technology (GRIET)
doi:10.1016/j.mspro.2014.07.115

materials. This modification can be done by addition of different ceramic powders of different sizes to achieve the required mechanical properties. McGrath et al. (2008) studied the effect of alumina powder in epoxy on mechanical properties. They found that there is a little effect on change in particle size, shape and size distribution on final properties. However the resin crosslink density and filler loading were the most critical variable, which may change all properties. Kardar et al. (2008) studied the effect of nano alumina particles on physical–mechanical properties of UV cured epoxy acrylate via nano-indentation. In their study it is observed that the scratch resistance and self-healing of the film improve in the presence of nano-particles. Hongxia Zhao and Robert K.Y.Li (2008) studied the effect of water absorption on the mechanical and dielectric properties of nano-alumina filled epoxy nanocomposites. They found that mechanical properties like stiffness of the matrix increases with nano alumina incorporation and also improve the dielectric properties because of increase in total interfacial area. Zhao et al. (2008) studied the mechanism leading to improved mechanical performance in nanoscale alumina filled epoxy. In their observation it is found that the improvement of the tensile strength is because of stronger interface lead to crack deflection and micro cracking. Rongguo and Wenbo Luo (2008) studied the effect of nano silica filers on mechanical properties of epoxy composite. They found that elastic moduli of the nano-$SiO_2$/epoxy composite are more than those of the neat epoxy resins. However, the elongation of the composites decreases with increasing $SiO_2$ mass fraction. Chen et al. (2008) studied effect of highly dispersed nano silica-epoxy resins with enhanced mechanical properties. It is seen that there is substantial improvement on mechanical properties of the composite with nano $SiO_2$ fillers. Ahmad et al. (2008) studied the effect of $SiO_2$ particle shape on mechanical properties of $SiO_2$/epoxy composite. They observed that the elongated shape of silica mineral shows the highest mechanical properties compare to other shapes and also the mechanical properties increases with increase in filler percentage. This is because of filler agglomeration, filler-matrix compatibility, bonding at interface and aspect ratio of the fused silica in the epoxy system. Johnsen et al. (2007) studied the toughening mechanisms of silica nano particle-modified epoxy polymers. They found that the glass transition temperature ($T_g$) was unchanged by the addition of the nano particles. However at the same time the modulus and toughness were increased with nano $SiO_2$ fillers. A. Mirmohseni and S. Zavareh (2010) studied the improvement of toughness by addition of nano fillers like $TiO_2$. They observed that impact and tensile strength increases with nano fillers compare to neat epoxy. Amit Chatterjee and Muhammad S. Islam (2008) have studied the effect of nano $TiO_2$ fillers on mechanical properties of $TiO_2$-epoxy nano composite. It is concluded that nano filler infusion improves the mechanical, thermal and viscoelastic properties of the epoxy resin. They also indicated that there is a improvement on storage modulus, tensile modulus, $T_g$, Flexural modulus and short beam shear strength from neat epoxy resin. Zhou et al. (2010) studied the effect of $TiO_2$ particle size and weight fraction on the flexural strength and failure mode of reinforced epoxy. They observed that micro sized particle has little effect on flexural strength at low weight fraction compare to nano particles. However, more than 1% of nano $TiO_2$ reduces the mechanical properties because of stress concentration caused by agglomeration of nano particles. Hamming et al. (2009) quantified the effect of dispersion and interfacial modification on macro scale properties of $TiO_2$ polymer-Matrix nanocomposites. It is observed that there is a decrease in $T_g$ as weight percent of the unmodified nanoparticles increases. In their study it is indicated that the $T_g$ is highly sensitive to both the quality of the interfacial interaction and quality of dispersion of the nanoparticles. Siddhartha et al. (2011) observed that tensile strength, flexural strength, tensile modulus, flexural modulus and impact strength increase with increase in filler percentage up to 20 wt.%.

It is observed from literature that there is a significant improvement on mechanical properties with epoxy matrix modification. However, comparative study on mechanical properties in different micro modifiers like $Al_2O_3/SiO_2/TiO_2$ with equal wt. % has not been studied for glass fiber/epoxy hybrid composite. Therefore in this paper the mechanical properties with 10 wt. % different micro fillers filled glass fiber/epoxy hybrid composites have been studied. It is observed that there is an improvement of mechanical properties in epoxy modified composite. It is observed that alumina modified epoxy composite increases the hardness and impact energy compare to other modifiers. Agglomeration of $Al_2O_3$ micro particles is observed in SEM analysis. From SEM analysis it is also observed that the mode of failure is the combination of crack in matrix, matrix/fiber debonding and fiber pull out irrespective of micro fillers.

## 2. **Experimental details**

### *2.1 Materials*

In our study, commercially available $Al_2O_3$ (<200 micron), $SiO_2$ (< 10 micron), $TiO_2$ (< 300 micron) particles are used to modify the epoxy matrix. Micro fillers of $Al_2O_3$, $SiO_2$ and $TiO_2$ were purchased from SRL Chemicals, Alfa Aesar and Finar Chemicals, India respectively. Commercially available woven roving fabric E-glass fiber with silane-coupling sizing system (Saint-Gobian Vetrotex) with fiber thickness of 8 micron is used as re-enforcement. The epoxy which is used was Araldite( LY-556 ) an unmodified epoxy resin based on bisphenol-A-diglycidyl-ether and chemically belongs to 'epoxide' family and hardener (HY -951),aliphatic 951, aliphatic primary amine are supplied by Ciba-Geigy ,India .

### *2.2 Fabrication of hybrid FRP composite*

There are three types of hybrid FRP composite with different fillers are fabricated using hand lay-up method. The weight percentage of epoxy, fiber, filler and hardener are fixed and designations of the composites are reported in Table -1. Where A, S and T indicate $Al_2O_3$, $SiO_2$ and $TiO_2$ modified FRP composite respectively. Initially the micro alumina/silica/titania powders are dried at 60 $^oC$ for 2hrs before mixing with epoxy. The fillers are mixed with neat epoxy and stirred manually using glass rod for a time period of 30 minutes before hardener addition. In each layer of the composite, mild steel (MS) roller is used to remove entrapped air and maintain uniform thickness of epoxy. Laminates are cured at room temperature for at least 72 hours before characterization.

Table -1 Designation and composition of hybrid composite

| **Designation of composites** | **Composition** |
|---|---|
| A | Epoxy + 60 Wt% Glass fiber + 10 Wt % $Al_2O_3$ |
| S | Epoxy + 60 Wt% Glass fiber + 10 Wt % $SiO_2$ |
| T | Epoxy + 60 Wt% Glass fiber + 10 Wt % $TiO_2$ |

## 3. Results and Discussions

### *3.1 DSC analysis*

The effects of micro particles on glass transition temperature of the hybrid composite are analyzed for A, S and T types of composite. The glass transition temperature with different modifiers is reported in Table 2. It is observed that there is no significant change in glass transition temperature for different modifiers. Therefore modification of matrix using different micro ceramic particle does not have much effect of glass transition temperature.

Table 2 Glass transition temperature and micro-hardness of epoxy modified composites

| **Name of the composite** | **Glass Transition Temperature ($^oC$)** | **Micro-hardness ( Hv)** |
|---|---|---|
| A | 55.62 | 21.6 |
| S | 53.57 | 20.7 |
| T | 54.81 | 19.4 |

### *3.2 Micro-hardness*

Digital Leco micro-hardness tester is used to measure the micro-hardness of the composite. A diamond indenter is forced into the composite specimen under a load F = 3 N for a time period of 10 seconds to get the impression on the surface. The average Vickers hardness has been reported in the Table 2. It is observed that the hardness of 'A' type hybrid composite is more compare to 'S' and 'T' type. This is quite obvious that the hardness of Alumina particles is more comparing to Silica and Titania. However the hardness values indicate that there is no significant difference in hardness between A, S & T type of composite.

*3.3 Flexural and Interlaminar Shear Strength*

The inter-laminar shear strength (ILSS) and Flexural strength ( FS) are calculated by performing short beam shear (SBS) test. The test is conducted as per the ASTM- D2344/D2344M-00. The dimension of the specimen for the test is 28 mm X 11 mm X 5.5 mm and span length of 22 mm. Universal testing machine Instron 5967 is used to conduct the test. The cross head speed is maintained at 1 mm/min and the test is repeated for four samples of same type and average values are reported. The ILSS and flexural strength (FS) are calculated as per the following equations:

$$ILSS = \frac{3P}{4bt} \quad (1)$$

$$FS = \frac{3PL}{2bt^2} \quad (2)$$

Where, P is the maximum load applied (N), t is the thickness (mm) of the sample and b is the width (mm) and L is the length of the sample. Figure 1(a) and (b) show comparison of flexural strength and flexural modulus with different fillers respectively. It is observed that both flexural strength and modulus of silica modified epoxy is more comparing to alumina and Titania. This may be because of finer particle size (<10 micron) of silica compare to Alumina and Titania (200- 300 micron). As decrease the particle size, increases the surface area and better adhesive bond between matrix and filler, therefore $SiO_2$ particles gives better adhesive strength and improve the mechanical properties. Interlaminar shear strength also shows similar trend like flexural modulus. However it is observed from figure 2 that the impact energy maximum for alumina modified epoxy compares to others. It may be because of more hardness of alumina compare to silica and Titania.

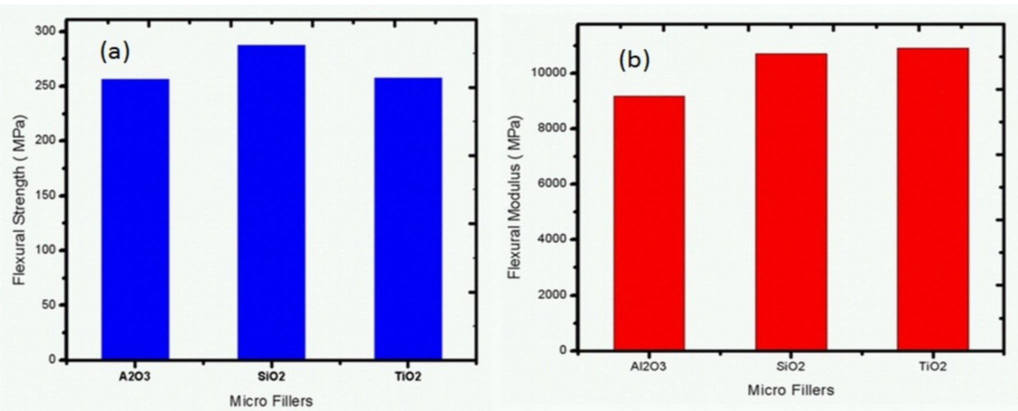


Figure 1: (a) comparison of Flexural Strength Vs Micro Fillers; (b) comparison of Flexural Modulus Vs Micro Fillers

*3.4 SEM analysis*

Fracture surface of SBS tested samples have been investigated using Scanning Electron Microscope (SEM). Figure 3 (a) shows the SEM micrograph of fractured samples of $Al_2O_3$ epoxy modified composite. It is clearly observed that there is an agglomeration of alumina particles during mixing of particles with epoxy by hand stirring method. We also observed there are pot holes, crack in the matrix and delamination fibers from matrix are predominate failure mechanism of this composite. Figure 3(b) shows the fracture surface of $SiO_2$ modified epoxy composite. It is observed that there are pot holes in the matrix because of hand layup fabrication technique. It is also observed that the predominant mechanism for fracture of the composite is matrix/fiber interface debonding. Because of smaller particle size compare to alumina and titanium, the interface bond between filler and matrix is better compare to alumina and titanium matrix modified composite. Figure 4 shows the fracture surface of $TiO_2$ modified epoxy composite. It is observed that the predominant mechanism for fracture of the composite is matrix/fiber interface debonding, brittle fracture of fibers and fiber pull out. This may be because of weak bond between matrix and fibers.

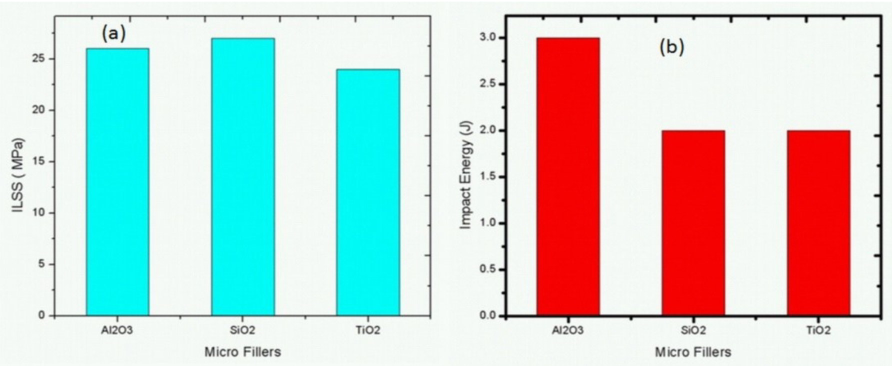


Figure 2: (a) comparison of Interlaminar Shear Strength Vs Micro Fillers; (b) comparison of Impact Energy Vs Micro Fillers

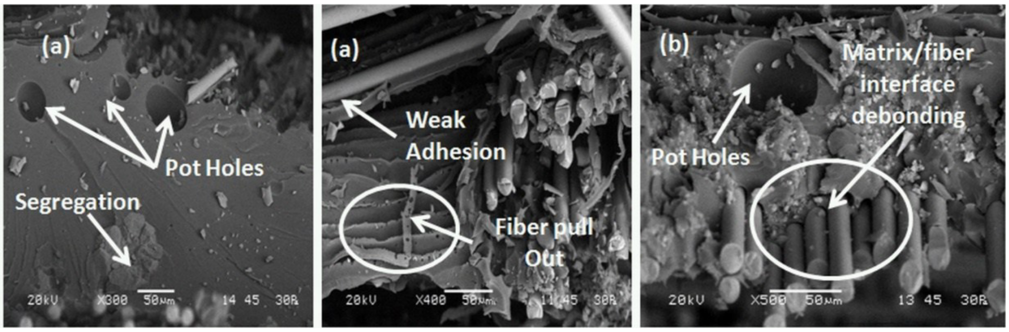


Figure 3: (a) SEM micrograph for $Al_2O_3$ Modifier (b) SEM micrograph for SiO2 particle modifier

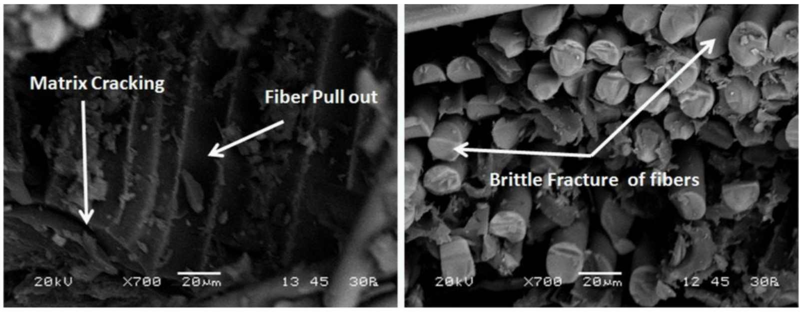


Figure 4: SEM micrograph for $TiO_2$ particle modified epoxy composite

## 4. Conclusions

The following conclusion can be made from our experimental results.

- Mechanical properties like ILSS, flexural strength and flexural modulus are more in case of $SiO_2$ modified epoxy composite compare to other fillers.
- Improvement of mechanical properties increases with decrease in ceramic particle size.
- Alumina modified epoxy composite increases the hardness and impact energy compare to other modifiers.
- Agglomeration of $Al_2O_3$ micro particles in the matrix is observed.

- SEM analysis clearly indicates the mode of fracture is the combination of matrix crack, matrix/fiber debonding and fiber pull out for all types of composite.

## References

Ahmad Farrah Noor, Mariatti Jaafar , Samayamutthirian Palaniandy, Khairun Azizi and Mohd Azizli, 2008. Effect of particle shape of silica mineral on the properties of epoxy composites. Composites Science and Technology 68, 346–353.

Chatterjee Amit and Muhammad S. Islam, 2008. Fabrication and characterization of TiO2–epoxy nanocomposites, Materials Science and Engineering A 487, 574–585.

Chen Chenggang, Ryan S. Justice, Dale W. Schaefer, Jeffery W. Baur, 2008. Highly dispersed nanosilica–epoxy resins with enhanced mechanical properties, Polymer 49, 3805–3815.

Johnsen B.B., A.J. Kinloch, R.D. Mohammed, A.C. Taylor, S. Springer, 2007. Toughening mechanisms of nano particle-modified epoxy polymers, Polymer 48,530-541.

Kardar P. , M. Ebrahimi , S. Bastani, 2008. Study the effect of nano-alumina particles on physical–mechanical properties of UV cured epoxy acrylate via nano-indentation, Progress in Organic Coatings 62, 321–325.

Lesley M. Hamming, Rui Qiao , Phillip B. Messersmith, L. Catherine Brinson, 2009. Effects of dispersion and interfacial modification on the macro scale properties of TiO2 polymer–matrix nanocomposites, Composites Science and Technology 69,1880–1886.

McGrath Laura M. , Richard S. Parnas , Saskia H. King , John L. Schroeder , Daniel A. Fischer , Joseph L. Lenhart, 2008. Investigation of the thermal, mechanical, and fracture properties of alumina epoxy composites, Polymer 49, 999-1014.

Mirmohseni A. and S. Zavareh, 2010. Preparation and characterization of an epoxy nanocomposites toughened by a combination of thermoplastic, layered and particulate nano-fillers, Materials and Design 31, 2699–2706.

Siddhartha, Amar Patnaik , Amba D. Bhatt,2011. Mechanical and dry sliding wear characterization of epoxy–TiO2 particulate filled functionally graded composites materials using Taguchi design of experiment, Materials and Design 32, 615–627.

Zhao Hongxia, Robert K.Y. Li, 2008. Effect of water absorption on the mechanical and dielectric properties of nano-alumina filled epoxy nanocomposites. Composites Part A 39, 602–611.

Zhao Rongguo, Wenbo Luo, 2008. Fracture surface analysis on nano-SiO2/epoxy composite. Materials Science and Engineering A 483–484, 313–315.

Zhao Su, Linda S. Schadler, Renee Duncan , Henrik Hillborg , Tommaso Auletta,2008. Mechanisms leading to improved mechanical performance in nanoscale alumina filled epoxy, Composites Science and Technology 68, 2965–2975.

Zhou Yuanxin , Evert White, Mahesh Hosur, Shaik Jeelani,2010. Effect of particle size and weight fraction on the flexural strength and failure mode of $TiO_2$ particles reinforced epoxy, Materials Letters 64, 806–809.