



Fabrication and Characterization of Nano-filled Polymer Composites

F.Shehata , A.Fathy, M. Megahed* , D. Morsy

Department of Mechanical Design and Production Engineering, Faculty of Engineering, Zagazig University, P.O. Box 44519, Egypt.

ARTICLE INFO

Article history:

Received 20 November 2019
Received in revised form
30 November 2019
Accepted 04 December 2019
Available online 10
December 2019

Keywords:

Nano-filled composites
Micro-filled composites
Mechanical properties
Glass fiber/epoxy composite
Agglomeration

ABSTRACT

Polymers are essential matrix materials for fabricating advanced composites. The reinforcing materials take many forms. In addition to continuous fibers, filler reinforced was used. In this work, the influence of incorporation nano-metal particles to glass fiber/epoxy composites was investigated. Aluminum was used as metal nanoparticles. Nano and Micro fillers was sonicated with epoxy resin by ultrasonic processor to ensure good distribution of fillers. The composite laminate was fabricated by hand lay-up technique. Woven glass fiber was used as fiber reinforcement. The volume fraction of glass fiber was 35%. The aluminium fillers were added with weight percentage of 0.2 wt%, 2-wt% and 4-wt%. The results show that the fabricated composites have shown an improvement in tensile, flexural, and hardness strengths as compared to neat glass fiber/epoxy composites. The failure strain increased in both the tensile and flexural tests. Increasing the weight content of nano aluminium particles to 4-wt % decrease the tensile strengths. However, increasing the aluminium micro-particles to 4 wt% has shown enhancement in mechanical properties. Micro aluminum fillers indicated a maximum of 195 MPa at 4wt% Al. A higher weight fraction of nano Al in the epoxy resin had showed little agglomerations. The water absorption values observed were lower for composite filled with nanoparticle. The flexural strength increased by 52.21%, flexural strain by 21.4 %, and flexural modulus by 76.57% compared to GFRE.

1. Introduction

Polymeric composite materials replace conventional materials because of their high specific strength and stiffness. Polymer composites reinforced with fiber reinforcement are commonly used in different applications as in marine, automotive, aerospace, and construction industries [1], [2]. Epoxy is a popular choice of thermoset polymers because of their good adhesion to fillers and fibers giving good stiffness and dimensional stability [3],[4]. Some studies have attempted to improve the properties of such polymers by introducing fillers to enhance toughening mechanisms [1], [5], [6], [7], [8].

Recently, polymer matrices filled with micro and nanofiller composites have gained attention due to their high strength-to-weight and stiffness-to-weight ratios. These particulate composites give high durability with lightweight and design flexibility. Reinforced polymer composites are considered attractive materials used in several industrial and technological applications [8]. Nanotechnology now is considered as one of the most public areas for research in all technical disciplines [9]. Due to the improvement of several properties as mechanical and physical properties, nanocomposites become a new alternative to polymers [2, 3, 4] as they can improved properties as compared to their macro and micro

* Corresponding author. Tel.: +201222857519; Fax: +20552304987
E-mail address: monawafa6@gmail.com

composites [10], [9]. Dispersing of nanofillers into polymer matrix influence the mechanical and physical properties of nanocomposites [11], [12], [13]. However, nanofillers tend to agglomerate because of their high surface energy. Enhanced properties are mostly relying on the good dispersion of nanofiller within the polymeric matrix, as agglomerated nanofillers act as defects [14], [15]. The main aim of this paper is to differentiate between the mechanical properties of glass fiber/epoxy filled with micro and nano aluminum fillers.

2. Experimental Work

2.1. Materials

The micro/nanocomposites were fabricated from an epoxy type (Kemapoxy 150 RGL). The epoxy was supplied from CMB Group, Egypt. The fiber reinforcement used is woven E-glass fiber of diameter 16 μm . This glass fiber was used with fiber volume fraction of 35%. The aluminium fillers were added with weight percentage of 0.2 wt%, 2-wt% and 4-wt%. These percentages were chosen to present low, moderate and high weight percentages. The other reinforcement filler is aluminum that is added in micro or nano-sized. The size of micro-filler particle is 60 μm in diameter; however, aluminum (Al) nanoparticles are 70 nm in diameter. Aluminum nanofillers were supplied by US Research Nanomaterials, Inc with purity of 99.9%. Metal fillers were used to toughen epoxy thus improve their mechanical and physical properties [28]. Metal nanoparticles as Al greatly improved tensile strength, maybe due to the presence of the metal nanoparticles that could fill in the surface cracks of the reinforced fibers and increase the radius of the crack tip in order to avoid the stress concentration [39]. Aluminum filler is easy availability and their low cost.

2.2. Sonication of nano and micro Al in epoxy resin

Micro and nano-filler Al was dispersed in epoxy via sonication. Mixing was carried out with Hielscher ultrasonic processor UP 200 S as shown in Fig. 1. Sonication was carried out at 0.5on/0.5off cycles per second with amplitude 70% for 3 hrs. To prevent epoxy resin degradation, the mixture was cooled by setting on a beaker with water bath before sonication is occurred [16], [6]. After that hardener was carefully added to the blend with a ratio of 1:2 by weight of kemaepoxy. A single mesh of glass fiber is placed over a glass slab covered with a layer of

epoxy matrix. Then epoxy matrix mixture was poured onto the glass fiber mesh and was equally spread over the glass fiber laminar mesh using a roller brush. A slight load was applied while rolling to prevent the formation of air gaps. On top of the previous layer, another glass fiber laminate layer was placed and epoxy was poured and distributed along the surface of the glass fiber as shown in Fig. 2. This process was carried out until all seven layers of glass fibers were placed on top of each other. Another glass slab was placed on top of this glass epoxy layers to complete the curing under load of 35 Kg. The fabricated micro/nano composite was left at room temperature for curing for about 24 hrs. A plate of 4mm thickness plate was produced.



Fig. 1. Mixing Al particles with Ultrasonic processor



Fig. 2. Spread the micro/nanophased epoxy on the glass fiber layers

2.3 Materials Characterization

2.3.1 tensile tests

Tensile tests were carried out on micro/nano composites according to ASTM D3039. The tensile tests were conducted on a computerized universal Jinan Test Machine WDW 100 kN. Crosshead speeds

were set at 2mm/min. Tensile tests were carried at room temperature. Five specimens were tested for each composite composition and the average value was considered to present the obtained value from tensile test.

2.3.2 Flexural tests

The flexural properties were investigated using a three-point flexural test and was carried out by using the same universal testing machine according to ASTM standard D-790.

2.3.3 Hardness

Hardness was measured by PCE-1000. Hardness was measured randomly at 10 different randomly points for each composite sample, the maximum and minimum results were disregarded. The mean value of hardness was calculated using the remaining eight values.

2.3.4 Water Absorption Test

Water absorption test was performed to determine the absorptivity of the material. Unreinforced and reinforced glass fiber/epoxy specimens were prepared according to ASTM D 570 standards. The specimens were immersed in distilled water (i.e. for 110 days) till the curves reach the saturation point at ambient temperature. Water absorption tests were performed according to ASTM D 570 specifications in which the water absorption content M_t at time t for all specimens was calculated as follows [17]:

$$M(t) = \frac{w_t - w_o}{w_o} \times 100 \quad (1)$$

where W_0 and W_t are the initial mass and the mass at time t of the sample, respectively.

3. Results and Discussions

Figures 3 to 5 show the tensile strength, elongation and Young's modulus, of Aluminum filled composites against Aluminum particles contents in both micro and nano sized particles respectively.

Maximum strength value of 200 MPa was achieved in a sample containing 2wt% Al nanoparticles. Whilst samples with micro aluminum fillers indicated a maximum of 195 MPa at 4wt% Aluminum.

It was found that increasing of the Aluminum nanoparticles to 4 wt%, the tensile strength was decreased to 191.5 MPa. This decrease in the

strength may be attributed to the agglomeration of nanoparticles at high concentrations.

The addition of nanoparticles usually provided very large surface area available to be mixed with epoxy matrix and improved the strength of composite matrix. At high concentrations the nano-filler particles were joined together forming large agglomerated particles that adverse the matrix properties.

Figures 6 and 7 show the flexural strength and flexural strain of filled composites versus Aluminum particles contents in both micro and nano sized particles, respectively. These curves showed that the different Aluminum filler contents leads to different flexural behaviors. It is noted that composite filled with nano Al particles showed higher values than corresponding micro particles.

The highest flexural property was obtained by the addition of 4-wt% nanosized Aluminum filler particles. The enhancements in flexural properties have the same general trend of tensile behaviour. This is probably due to compressing effect tension of outer layers in bending. The compression in inner layers in bending can close and delay propagations of fine cracks. During the bending process, compression is attained in the composite material on the inside of the neutral plane of the bending specimen, while the material on the outside of the neutral plane is under tension.

Figure 8 shows the hardness of neat, glassfiber epoxy composites with and without the addition of nano or micrometer sized Aluminum filler. These figure showed that the different filler contents gave different hardness values. It can also be seen from; that the highest hardness value was obtained by addition of 2 -wt.% Aluminum nanoparticles where hardness had value doubled the hardness of neat composite without any filler. The improvements in hardness had been shown in both nano and micro particles of aluminum. However, nanoparticle fillers was more effective than micro particles. It may be attributed to great surface area of nanoparticles compared to micro ones. It should also note that the hardness at 4-wt% Aluminum nanoparticles decreased compared to 2 wt. %. This could be due to agglomeration caused in the nanometer sized particles at greater contents.

Fig. 9 shows the water Absorption of glass fiber/epoxy reinforced with micro and nano Al particles. The results revealed that addition of 4wt% Al has reduced water diffusion by 9% as compared to composite without any filler.

Water absorption is generally affected by presence

of cracks, holes. Voids or gabs between epoxy and glassfiber. Strong adhesion implies fewer locations that could store water. The nano particles would fill voids, gabs or fine cracks that may be present leading to strong composites with lower places to store the water. Therefore composites with larger nanoparticles showed lower water absorption.

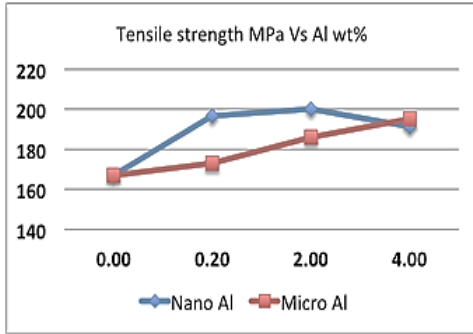


Fig. 3. Tensile strength of glass fiber/epoxy reinforced with micro and nano Al particles

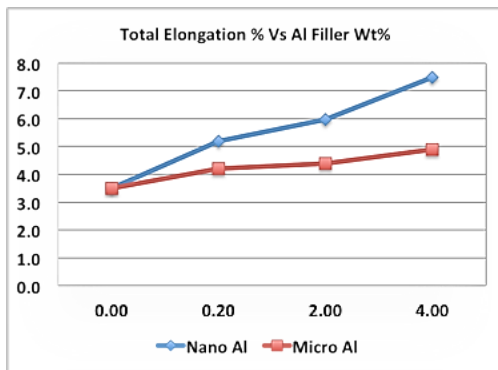


Fig. 4. Elongation at break of glass fiber/epoxy reinforced with micro and nano Al particles

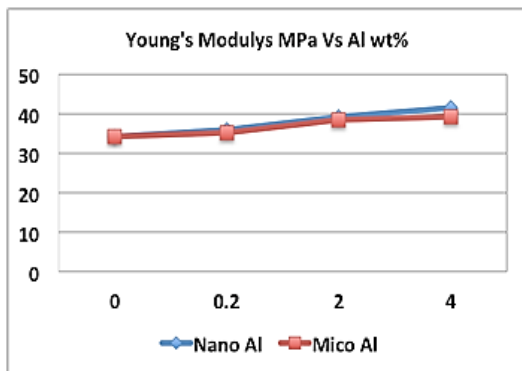


Fig. 5. Tensile modulus of glass fiber/epoxy reinforced with micro and nano Al particles

3.1 Flexural tests

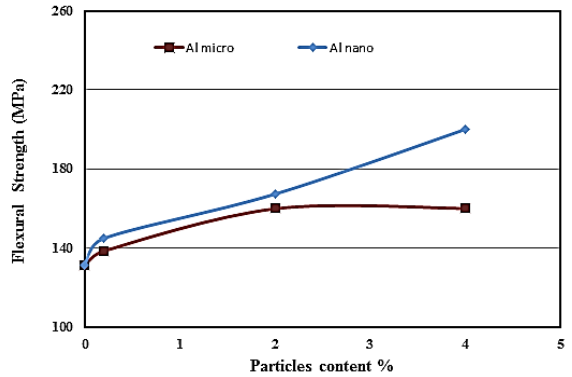


Fig. 6. Flexural strength of glass fiber/epoxy reinforced with micro and nano Al particles

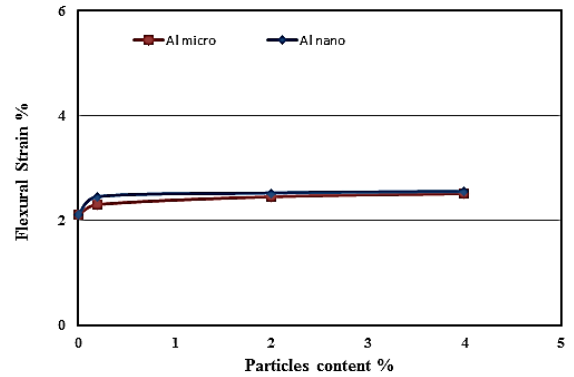


Fig. 7. Flexural strain of glass fiber/epoxy reinforced with micro and nano Al particles

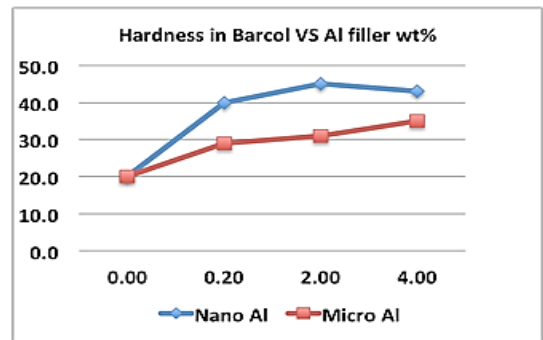


Fig. 8 Hardness in Barcol, for neat GFRE Glassfiber composite with and without aluminum fillers.

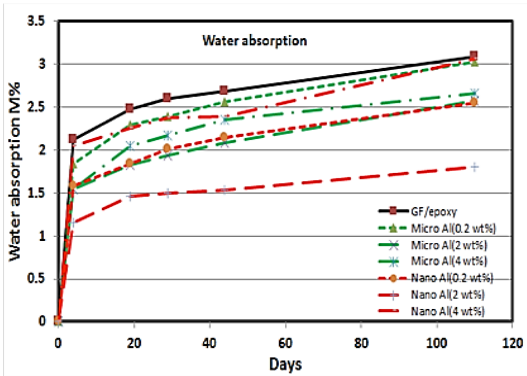


Fig. 9 Water Absorption of glass fiber/epoxy reinforced with micro and nano Al particles

Increasing the weight content of nano-aluminum particles to 4-wt % decrease the tensile strengths. A higher weight fraction of nano Al in the epoxy resin had showed little agglomerations as shown in Fig. 10.

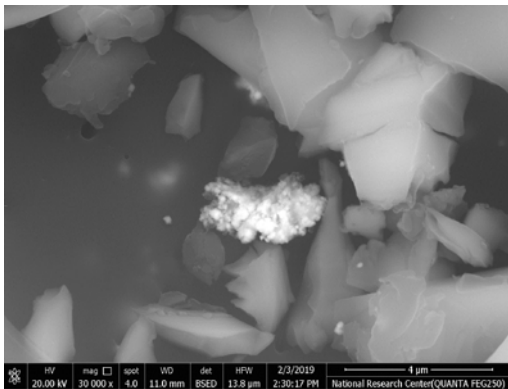


Fig. 10. SEM of glass fiber/epoxy reinforced with 4 wt% nano Al particles

4. Conclusions

In this work, the effects of using nanometer- and micrometer-sized aluminum particles into glass fiber-reinforced epoxy composites have been characterized. Based on the experiments, the following results can be drawn;

1. The glass fiber/epoxy composite filled with Aluminum nanoparticles gave maximum tensile strength of 200 MP which is 1.28 times larger than glass fiber/epoxy composite without any filler.
2. The glass fiber/epoxy composite with Aluminum micro particles gave maximum

tensile strength of 1955 MP which is 1.255 times larger than glass fiber/epoxy composite without any filler.

3. The glass fiber/epoxy composite with 4-wt% Al nanoparticles showed a slight decrease in tensile strength compared to Al filler of 2-wt%. This is attributed to nanoparticle agglomerations at high concentrations.
4. The highest flexural property was obtained by addition of 4-wt% nano sized aluminum particles. The flexural strength improved by 52.21%, flexural strain by 21.4 %, and flexural modulus by 76.57%.
5. The highest hardness value was obtained by addition of 2-wt% nanometer sized aluminum particles where hardness has value more than doubled (2.1 times) the hardness of neat glass fiber/epoxy.

References

- [1] H. J. Kim, D. H. Jung, I. H. Jung, J. I. Cifuentes, K. Y. Rhee, and D. Hui, "Enhancement of mechanical properties of aluminium / epoxy composites with silane functionalization of aluminium powder," *Compos. Part B*, vol. 43, no. 4, pp. 1743–1748, 2012.
- [2] A. Fathy, A. Shaker, M. A. Hamid, and A. A. Megahed, "The effects of nano-silica / nano-alumina on fatigue behavior of glass fiber-reinforced epoxy composites," *J. Compos. Mater.*, vol. 51, no. 12, pp. 1667–1679, 2016.
- [3] P. Sarkar, N. Modak, and P. Sahoo, "Effect of Aluminum Filler on Friction and Wear Characteristics of Glass Epoxy Composites," *Silicon*, pp. 1–9, 2017.
- [4] Z. Wang *et al.*, "Effect of micro-Al₂O₃ contents on mechanical property of carbon fiber reinforced epoxy matrix composites," *Compos. Part B*, vol. 91, pp. 392–398, 2016.
- [5] S. C. Zunjarrao and R. P. Singh, "Characterization of the fracture behavior of epoxy reinforced with nanometer and micrometer sized aluminum particles," *Compos. Sci. Technol.*, vol. 66, no. 13, pp. 2296–2305, 2006.
- [6] A. A. Megahed and M. Megahed, "Fabrication and characterization of functionally graded nanoclay/glass fiber/epoxy hybrid nanocomposite laminates," *Iran. Polym. J.*, vol. 26, no. 9, pp. 673–680, 2017.
- [7] M. Megahed, A. A. Megahed, and M. A. Agwa, "Mechanical properties of on/off-axis loading for hybrid glass fiber reinforced epoxy filled with silica and carbon black nanoparticles," *Mater. Technol.*, vol. 33, no. 6, pp.

- 398–405, 2018.
- [8] M. Manjunath, N. M. Renukappa, and B. Suresha, “Influence of micro and nanofillers on mechanical properties of pultruded unidirectional glass fiber reinforced epoxy composite systems,” *J. Compos. Mater.*, vol. 50, no. 8, pp. 1109–1121, 2016.
- [9] D. R. Paul and L. M. Robeson, “Polymer nanotechnology : Nanocomposites,” *Polymer (Guildf)*., vol. 49, pp. 3187–3204, 2008.
- [10] V. S. Nguyen, D. Rouxel, R. Hadji, B. Vincent, and Y. Fort, “Effect of ultrasonication and dispersion stability on the cluster size of alumina nanoscale particles in aqueous solutions,” *Ultrason. Sonochem.*, vol. 18, no. 1, pp. 382–388, 2011.
- [11] E. T. Thostenson, C. Li, and T. Chou, “Nanocomposites in context,” *Compos. Sci. Technol.*, vol. 65, pp. 491–516, 2005.
- [12] M. Megahed, A. A. Megahed, and M. A. Agwa, “Mechanical properties of on / off-axis loading for hybrid glass fiber reinforced epoxy filled with silica and carbon black nanoparticles,” *Mater. Technol.*, vol. 33, no. 6, pp. 398–405, 2018.
- [13] M. E. Kabir, M. C. Saha, and S. Jeelani, “Effect of ultrasound sonication in carbon nanofibers/polyurethane foam composite,” *Mater. Sci. Eng. A*, vol. 459, no. 1–2, pp. 111–116, 2007.
- [14] J. C. Santos, L. M. G. Vieira, T. H. Panzera, M. A. Schiavon, A. L. Christoforo, and F. Scarpa, “Hybrid glass fibre reinforced composites with micro and polydiallyldimethylammonium chloride (PDDA) functionalized nano silica inclusions,” *Mater. Des.*, vol. 65, pp. 543–549, 2015.
- [15] A. I. Alateyah *et al.*, “Processing, Properties, and Applications of Polymer Nanocomposites Based on Layer Silicates : A Review,” *Adv. Polym. Technol.*, vol. 32, no. 3, pp. 1–49, 2013.
- [16] M. Megahed, A. A. Megahed, and M. A. Agwa, “The influence of incorporation of silica and carbon nanoparticles on the mechanical properties of hybrid glass fiber reinforced epoxy,” *J. Ind. Text.*, vol. 49, no. 2, pp. 181–199, 2019.
- [17] A. M. M. Abdelhaleem, M. Megahed, and D. Saber, “Fatigue behavior of pure polypropylene and recycled polypropylene reinforced with short glass fiber,” *J. Compos. Mater.*, vol. 52, no. 12, pp. 1633–1640, 2018.