# Physical, Mechanical and Thermal Characterization of Glass Micro-sphere filled Epoxy Composites

**Gaurav Gupta<sup>a,\*</sup>, Alok Satapathy<sup>b</sup>** <sup>a</sup>**Dept. of Mechanical Engg., National Institute of Technology**, Rourkela, India E-mail address: gauravgupta198606@gmail.com

<sup>b</sup>Dept. of Mechanical Engg., National Institute of Technology, Rourkela, India E-mail address: satapathy.alok@gmail.com

#### ABSTRACT

In spite of significant research done in the field of particulate filled polymeric composites, reports on polymers filled with glass micro-spheres have been extremely rare. In view of this, the present work includes the processing and characterization of a new class of epoxy composites filled with different proportions of borosilicate glass micro-spheres (BGM). Hand lay-up technique is used for making these composites in a laboratory scale. Various physical and mechanical properties are evaluated under controlled laboratory conditions. It is found that while the tensile and flexural strength are marginally influenced, the impact strength is improved quite significantly. Besides, multifold enhancement in composite micro-hardness is also noticed. The effective thermal conductivity is measured using Unitherm<sup>™</sup> Model 2022 following ASTM-1530 test standards. It is observed that with increase in the filler content there is appreciable drop in the conductivity of the composites leading to an improvement in their heat conduction capability.

Keywords: Polymer Composite, BGM, Characterization.

# 1. INTRODUCTION

In recent years, polymers and their composites are fast replacing the conventional materials in many engineering and structural applications. They have been used in various engineering fields starting from space craft applications to industrial usages due to high specific strength, high modulus, low density and better wear resistance (Hutchings, 1992). Being light weight, while they are the most suitable materials for weight sensitive applications, their high cost sometimes restricts their use in general applications. Use of low cost, easily available fillers is therefore useful to improve the properties and to lower the overall cost of components (Kranthi & Satapathy, 2010; Unal et al., 2003). Hard particulate fillers consisting of ceramic or metal particles are being used these days to improve wear and thermal resistance of the polymer (Gregory et al., 2003). The inclusion of such particulate fillers into polymers for commercial applications is primarily focused at the cost reduction and stiffness improvement (Rothon, 1999). Various researchers (Suresha et al., 2010; Mohan et al., 2011; Schwartz & Bahadur, 2001; Nayak et al., 2010) have reported that the wear and thermal resistance of polymers is improved by the addition of fillers.

In past two decades, ceramic filled polymer composites have emerged as a subject of extensive research. The present research work has been undertaken, with an objective to explore the potential of boro-silicate glass microspheres (BGM) as a filler material in

polymer composites and to investigate its effect on the physical, mechanical and thermal characteristics of the resulting composites. This work is an attempt to find a possible use of BGM which might gainfully be employed as particulate filler in polymers for developing high strength and thermal resistant composites.

Boro-silicate glass microspheres consist of outer stiff glass which results in some unique properties, such as light weight, high strength, low thermal conductivity. Based on these properties, BGMs have been used in the fabrication of polymer composites for different applications (Liang, 2002; Liang, 2005; Zhao, 2007; Kim & Khamis, 2001). These have multifunctional properties including high specific compressive strength, low moisture absorption and higher thermal stability which make them more suitable for aeronautical and marine applications (Kim & Khamis, 2001; Kim & Plubrai, 2004; Gupta & Nagorny, 2006; Wouterson et al., 2004).

Boro-silicate glass has excellent thermal properties with its low coefficient of expansion and high softening point, it also offers a high level of resistance to attack from water, acids, salt solutions, organic solvents and halogens. Therefore, the objective of the present work is to fabricate a new class of epoxy based composites reinforced with borosilicate glass micro-sphere particles in different weight proportions (0, 10, 20 and 30 wt%) and to study the physical, mechanical and thermal characteristics of these composites.

# 2. EXPEREMENTAL DETAILS

#### **2.1 Materials**

In the present work, epoxy resin (LY 556) is used as the matrix material. Its common name is bisphenol-A-diglycidyl-ether and it chemically belongs to the 'epoxide' family. The epoxy resin and the corresponding hardener (HY 951) are supplied by Ciba Geigy India Ltd. Epoxy is chosen primarily because of its excellent dimensional/thermal stability and good corrosion resistance (Suresha et al., 2010). Micro-sized borosilicate glass microspheres of average size 100  $\mu$ m procured from NICE Ltd. are used as the filler material. These microspheres are normally obtained by heating tiny droplets of dissolved sodium meta-silicate (Na<sub>2</sub>SiO<sub>3</sub>, commonly referred to as water glass or liquid glass) during ultrasonic spray pyrolysis process.

#### **2.2 Composite Fabrication**

Low temperature curing epoxy resin (LY 556) and corresponding hardener (HY951) are mixed in a ratio of 10:1 by weight as recommended. Borosilicate glass micro-spheres are reinforced in the resin to prepare the composites in different proportions according to the experimental requirement. The uniformly mixed dough (epoxy filled with BGM) is then slowly decanted into the glass molds, coated beforehand with wax and a uniform thin film of silicone-releasing agent. The castings are left to cure at room temperature for about 24 hours after which the glass molds are broken and samples are released. The composites are cast in these molds so as to get rectangular slabs of dimension 200 x 200 x 4 mm. From these slabs circular disc and dog-bone shaped specimens are cut for different characterization tests.

### **2.3 Density and Void Fraction**

The theoretical density of composite materials in terms of weight fraction can easily be obtained as for the following equations given by

$$\rho_{ct} = \frac{1}{\left(W_p / \rho_p\right) + \left(W_m / \rho_m\right)} \tag{1}$$

Where, W and  $\rho$  represent the weight fraction and density respectively. The suffix p and m represent particulate filler material and matrix material respectively.

The actual density ( $\rho_{ce}$ ) of the composite, however, can be determined experimentally by simple water immersion technique. The volume fraction of voids ( $V_v$ ) in the composites is calculated using the following equation:

$$V_{v} = \frac{\rho_{ct} - \rho_{ce}}{\rho_{ct}}$$
(2)

### 2.4 Micro-hardness Measurement

Micro-hardness measurement is done using a Leitz micro-hardness tester. A diamond indenter, in the form of a right pyramid with a square base and an angle  $136^{0}$  between opposite faces, is forced into the material under a load F. Lengths of the diagonals X and Y of the indentation left on the surface of the material after removal of the load are measured and their arithmetic mean L is calculated. In the present study, the load considered is F = 0.5 N and Vickers hardness number is calculated using the following equation (William, 2005):

$$H_{v} = 0.1889 \frac{F}{L^{2}}$$

$$\text{and } L = \frac{X+Y}{2}$$
(3)

where, F is the applied load (N), L is the diagonal of square impression (mm), X is the horizontal length (mm) and Y is the vertical length (mm). The hardness values are then expressed in GPa.

### 2.5 Tensile Strength

The tensile strength is generally performed on the flat specimens. The dog-bone type specimen with end tabs is commonly used for tensile test. ASTM-D3039-76 standard test method is employed for tensile test of composite specimens. The test is performed in the universal testing machine (UTM) Instron 1195 at across head speed of 10 mm per minute. Three numbers of specimens with same composition are used to get the mean value of the tensile strength.

### 2.6 Flexural Strength

The short beam shear (SBS) tests are performed on the composite samples at room temperature to evaluate the value of flexural strength. It is a 3-point bend test, which generally promotes failure by inter-laminar shear. The SBS test is conducted as per standard ASTM: D5379/D5379M using the same UTM. The flexural strength (*FS*) of any composite specimen is determined using the following equation:

$$F \cdot S = \frac{3Pl}{2bt^2} \tag{4}$$

Where, l is the span length of the sample. P is the load applied; b and t are the width and thickness of the specimen respectively.

### 2.7 Impact Strength

The pendulum impact testing machine confirming to ASTM: D256 ascertains the notch impact strength of the material by shattering the specimen with a pendulum hammer, measuring the spent energy and relating it to the cross section of the specimen. The machine is adjusted such that the blade on the free-hanging pendulum just barely contracts the specimen (zero position). The specimens are clamped in a square support and are struck at their central point by a hemispherical bolt of diameter 5 mm.

# 3. RESULTS AND DISCUSSION

## **3.1 Density and Void Fraction**

The measured densities and volume fraction of voids (porosities) of all BGM polymer matrix composites are presented in Table 1. It is observed that, by the addition of BGM particles, the density of the composites gradually increases. With the addition of filler, more voids are found in the composites. As the filler content increases from 0 wt. % to 30 wt. %, the volume fraction of voids is also found to increase proportionately. Similar observations have been reported earlier by previous researchers (Biswas & Satapathy, 2009; Biswas & Satapathy, 2010; Patnaik et al., 2008; Srivastava & Pawar, 2006).

	Composition	Density and Void fraction		
S. No.		Theoretical	Experimental	Void (%)
1	Ep+0 wt% BGM	1.100	1.098	0.18
2	Ep+10 wt% BGM	1.159	1.151	0.69
3	Ep+20 wt% BGM	1.224	1.212	0.98
4	Ep+30 wt% BGM	1.297	1.279	1.39

Table 1: Density and void fraction of the composites

### 3.2 Micro-hardness

The measured hardness values of all the four composites are presented in Table 2. It can be seen that the hardness is affected significantly with addition of BGM particles. It is observed that the micro-hardness, which is considered to be one of the important factors in composites for determination of wear rate, increases with increase in filler content. With the increase in BGM content from 0 wt.% to 30 wt.% the hardness is found to increase from 0.085 GPa to 0.586 GPa. This implies an increment of about 6 times in hardness of the BGM filled composites compared to the neat resin.

**Micro-hardness** Tensile Flexural Impact Thermal S. Composition (GPa) Strength Strength Strength Conductivity No. (MPa) (MPa)  $(KJ/m^2)$ (W/mK) Ep+0 wt% BGM 28.76 1 0.085 58 18.5 0.363 2 Ep+10 wt% BGM 0.229 57.54 27.03 21.7 0.235 3 Ep+20 wt% BGM 0.448 56.79 26.22 26.3 0.201 28.9 4 Ep+30 wt% BGM 0.587 55.29 25.87 0.197

Table 2: Mechanical and thermal properties of the composites

### **3.3 Tensile Strength**

The tensile strengths of BGM filled epoxy composites are shown in Table 2. It is found that, there is a gradual drop in tensile strength with increase in filler content. The unfilled epoxy has strength of 58 MPa in tension, and this value drops to 57.4 MPa, 56.79 MPa and 55.29 MPa with BGM addition of 10 wt.%, 20 wt.% and 30 wt.% respectively.

#### **3.4 Flexural Strength**

Composite materials used in structures are prone to fail in bending and therefore the development of new composites with improved flexural characteristics is essential. In the present work, the variations of flexural strength of the BGM filled polymer matrix composites are presented in Table 2. Marginal decrement in flexural strength is recorded for all the composite samples with the incorporation of BGM particles.

#### 3.5 Impact Strength

The impact strength of a material is its capacity to absorb and dissipate energies under impact or shock loading. The suitability of a composite for certain applications is determined not only by usual design parameters, but also by its impact or energy absorbing properties. Table 2 presents the measured impact energy values of the BGM filled polymer matrix composites. It is seen from the table that the impact energies of the composites increase gradually with filler content increasing from 0 to 30 wt%.

### **3.6 Thermal Conductivity**

Unitherm<sup>TM</sup> Model 2022 is used to measure thermal conductivity of the composites fabricated for this investigation. This is an instrument based on guarded heat flow method and is used for a variety of materials. The test is carried out in accordance with ASTM E-1530 standard. Table 2 presents the variation of effective thermal conductivity ( $K_{eff}$ ) of composites with different glass micro-sphere content. It is encouraging to note that the incorporation of BGM results in significant drop in thermal conductivity of epoxy resin and thereby increases its thermal insulation capability. With addition of 30 wt. % of BGM, the thermal conductivity decreases by about 84% as compared with neat epoxy resin.

## 4. CONCLUSIONS

• Boro-silicate glass microspheres can be used as a potential filler material for epoxy based composites.

• With improved hardness, these composites can have potential applications in tribological situations.

• It can be very useful for thermal resistance applications since the thermal conductivity is substantially decreasing by addition of BGM in different wt%.

# REFERENCES

[1] Hutchings, I.M. (1992). Tribology: friction and wear of engineering materials. CRC Press.

[2] Kranthi, G. & Satapathy, A. (2010). Evaluation and prediction of wear response of pine wood dust filled epoxy composites using neural computation. *Computational Materials Science*, 49, 609–614.

doi: 10.1016/j.commatsci.2010.06.001

[3] Unal, H., Findik, F. & Mimaroglu, A. (2003). Mechanical behavior of nylon composites

containing talc and kaolin. *Journal of Applied Polymer Science*, 88, 1694–1697. doi: 10.1002/app.11927

[4] Gregory, S.W., Freudenberg, K.D., Bhimaraj, P. & Schadler, L.S. (2003). A study on the friction and wear behavior of PTFE filled with alumina nanoparticles. *Wear*, 254(5–6), 573–580.

doi: 10.1016/S0043-1648(03)00252-7

- [5] Rothon, R.N. (1999). Mineral fillers in thermoplastics: filler manufacture and characterization. *Advances in Polymer Science*, 139, 67–107. doi: 10.1007/3-540-69220-7\_2
- [6] Suresha, B., Ramesh, B.N., Subbaya, K.M., Ravi Kumar, B.N. & Chandramohan, G. (2010). Influence of graphite filler on two-body abrasive wear behavior of carbon fabric reinforced epoxy composites. *Materials and Design*, 31, 1833–1841. doi: 10.1016/j.matdes.2009.11.006
- [7] Mohan, N., Natarajan, S. & KumareshBabu, S.P. (2011). Abrasive wear behavior of hard powders filled glass fabric epoxy hybrid composites. *Materials and Design*, 32, 1704–1709.

doi: 10.1016/j.matdes.2010.08.050

- [8] Schwartz, C.J. & Bahadur, S. (2001). The role of filler deformability, filler–polymer bending, and counterface material on the tribological behavior of polyphenylene sulfide. *Wear*, *251*, 1532–1540.
- [9] Nayak, R., Dora, P.T. & Satapathy, A. (2010). A computational and experimental investigation on thermal conductivity of particle reinforced epoxy composites. *Computational Materials Science*, 48, 576-581. doi: 10.1016/j.commatsci.2010.02.025
- [10] Liang, J.Z. (2002). Tensile and impact properties of hollow glass bead-filled PVC composites. *Macromolecular Materials and Engineering*, 287(9), 588–591.
   doi: 10.1002/1439-2054(20020901)287:9<588::AID-MAME588>3.0.CO;2-6
- [11] Liang, J.Z. (2005). Tensile and flexural properties of hollow glass bead-filled ABS composites. *Journal of Elastomers and Plastics*, 37(4), 361–370. doi: 10.1177/0095244305054674
- [12] Zhao, H.G., Liu, Y.Z., Wen, J.H., Yu, D.L. & Wen, X.S. (2007). Dynamics and sound attenuation inviscoelastic polymer containing hollow glass microspheres. *Journal of Applied Physics*, 101(12), 123518.
- [13] Kim, H.S. & Khamis, M.A. (2001). Fracture and impact behaviours of hollow micro-sphere/epoxy resin composites. *Composites Part A*, 32(9), 1311-1317. doi: 10.1016/S1359-835X(01)00098-7
- [14] Kim, H.S. & Plubrai, P. (2004). Manufacturing and failure mechanisms of syntactic foam under compression. *Composites Part A*, 35(9), 1009–1015. doi: 10.1016/j.compositesa.2004.03.013
- [15] Gupta, N. & Nagorny, R. (2006). Tensile properties of glass microballoon-epoxy resin syntactic foams. *Journal of Applied Polymer Science*, 102(2), 1254–1261. doi: 10.1002/app.23548
- [16] Wouterson, E.M., Boey, F.Y.C., Hu, X. & Wong, S.C. (2005). Specific properties and fracture toughness of syntactic foam: Effect of foam microstructures. *Composites Science* and Technology, 65(11–12), 1840–1850. doi: 10.1016/j.compositesh.2005.03.012

doi: 10.1016/j.compscitech.2005.03.012

- [17] William, F.H. (2005). Mechanical behavior of materials. UK, Cambridge University Press.
- [18] Biswas, S. & Satapathy, A. (2009). Tribo-performance analysis of red mud filled glass epoxy composites using Taguchi experimental design. *Materials and Design, 30*,

2841-2853.

doi: 10.1016/j.matdes.2009.01.018

[19] Biswas, S. & Satapathy, A. (2010). A study on tribological behavior of alumina-filled glass-epoxy composites using Taguchi experimental design. *Tribology Transactions*, 53, 520–532.

doi: 10.1080/10402000903491309

- [20] Patnaik, A., Satapathy, A., Mahapatra, S.S. & Dash, R.R. (2008). Implementation of Taguchi design for erosion of fiber reinforced polyester composite systems with SiC filler. *Journal of Reinforced Plastics and Composites*, 27(10), 1093–1111. doi: 10.1177/0731684407087688
- [21] Srivastava, V.K. & Pawar A.G. (2006). Solid particle erosion of glass fiber reinforced flyash filled epoxy resin composites. *Composites Science and Technology*, 66, 3021–3028.

doi: 10.1016/j.compscitech.2006.02.004