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Study the Effect of Dispersion of Filler in Polymer Composite for Radiation Shielding

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Good filler dispersion in a polymer resin is crucial for achieving performance. Main focus of this study is the effect of dispersion of various fillers in epoxy resin to produce radiation hardened plastic packaging which can be used as a shielding against a high frequency radiation attack. High-frequency rays have tendency to intrude in the resin and polymer especially are quite vulnerable to be penetrated by high-frequency rays which may create an early degradation of product. Hence, stabilizers are also used which can absorb the high-frequency radiation and save the material from early degradation. However, particular fillers are also required that can uniformly disperse in the resin and create a film which can provide extra protection against high-frequency radiation attack. Three types of compositions are prepared using epoxy/graphite, epoxy/lead, and epoxy/boron nitride nanopowder. Scanning Electron Microscope (SEM) images are used to study dispersion in resin. Small batches using gravity casting method at laboratory using compatibilizers are prepared to carry out experiments. Results of linear absorption coefficient carried out using Am–Be neutron source are also discussed in the paper. POLYM. COMPOS., 00:000–000, 2013. © 2013 Society of Plastics Engineers

INTRODUCTION

Composites are engineering materials made from two or more constituents with significantly different physical or chemical properties which remain separate and distinct on a macroscopic level within the finished structure. Most of the composites are made up of just two materials. One material (the matrix or binder) surrounds and binds together a cluster of fibers or fragments of a much stronger material (the reinforcement). For the matrix, many modern composites use thermosetting or thermoplastic polymers (also called resins). The plastics hold the reinforcement together and help to determine the physical properties of the end product [1].

Based on the form of reinforcement, common composite materials can be classified as follows [2–4]: (1) Fiber (short and long) reinforced composites; (2) particle (flakes, beads, spheres, needles, and irregular) reinforced composites; (3) gas reinforced composites (foams). Although glass fibers are by far the most common reinforcement, many advanced composites now use fine fibers of pure carbon or graphite.

An important property of composites is the possibility of planning structure in purpose to obtain established characteristics. In effect, composite materials are commonly applied in modern technology. One of the largest areas of application for polymers, polymer blends, and composites on their basis are electronic and electrical industries. Pure polymers are generally electrical insulators in nature; so they are applied as electrically insulating materials. Polymers contain a very low concentration of free charge carriers, and thus they are nonconductive and transparent to electromagnetic radiation. For that reason, they are not capable for being used as enclosures for electronic equipment as they cannot shield it from outside radiation or prevent the escape of radiation from the component. They also cannot provide protection against electrostatic discharge in handling sensitive electronic devices. These drawbacks have led to this research.

In the present work, authors have selected epoxy resin in view of its good mechanical thermal and corrosion resistance properties [5] and prepared its three different composites using three different fillers; graphite [6], lead [7], and boron nitride nanopowder [8] separately.

These materials have application in aircraft, automotive industry, space equipment, marine equipment, and many others [9, 10].

Gravity casting method is one of the methods relying on gravity without applying pressure. It is possible to

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produce spatial distribution of component materials by casting step-by-step [11].

Good fillers dispersion in a polymer resin is crucial to achieving performance. Optical and scanning electronic microscopy technologies are the most common tools to investigate dispersion in various forms of samples.

During sedimentation in a column, differences in the particle velocity caused by different density or size of the powder particles lead to demixing of the different particle types. If sedimentation occurs in a liquid column free of particles, a gradient with a continuous increase or decrease of the concentration of one particle type will be formed. If the sediment is directly formed from the suspension, a complicated transition function is retained. The bottom layer will still have the average composition of the suspension followed by an increase of demixing, and the top of the sediment contains only the powder fraction with the lowest sedimentation velocity [12].

Few experiments are carried out and SEM images are taken to study dispersion in resin. Then, its effect on absorption coefficient is studied.

EXPERIMENTAL

Sample Preparation

Methodology. Very small batches using gravity casting method at our laboratory are prepared with different sizes of fillers using compatibilizers to carry out experiments, and an exhaustive literature review is also been done to understand the effect especially on radiation resistance of a sample. On the basis of research results, the specimen thickness was determined (3 mm).

When fillers are added into the resin, viscosity and rheology properties of epoxy also play a vital role in dispersion of fillers. There are chances of uneven distribution of fillers into the resin and which may create clusters of regions, where few clusters are dense and few are not and some regions may keep vacant space of fillers.

High-frequency rays have the tendency to intrude in the resin, and polymers especially are quite vulnerable to be penetrated by high-frequency rays which may create an early degradation of product. These phenomena can be inhibited or slowed down by modifying polymers using suitable stabilizers, to save the material particularly in high frequencies of radiation from early degradation [13]. However, particular fillers are also required that can uniformly disperse in the resin and create a film which can provide extra protection against high-frequency radiation attack.

In this research work, graphite powder, lead powder, and boron nitride nanopowder are used.

The thermosetting matrix used in this study was Bisphenol A based unmodified epoxy resin cured at room temperature with 50% by weight of hardener. The density of the epoxy resin was 1.20 g/cm^3. Graphite powder

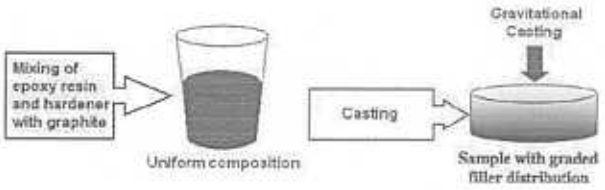

FIG. 1. Experimental setup.

was used as the filler. The density of graphite was 0.50 g/cm^3, lead 11.34 g/cm^3, and boron nitride nanopowder 2.1 gm/cm^3.

Sample Fabrication. The specimens were obtained using gravity casting methods, which is one of the technologies relying on gravity without applying pressure. It is possible with this method to produce one-dimensional gradient of component materials content in liquid matrix. The gradient is retained after matrix solidification. Due to sedimentation process, the highest filler content is expected in the lowest sample layer. The procedure of samples preparation is shown schematically in Fig. 1. Specimens were cast into our own designed steel frame. Cavity of the mould has the following dimensions: length – 300 mm, width – 300 mm, and depth – 3 mm (Fig. 1) [14].

Procedure. Epoxy resin, fillers, and stabilizers were commercial grade and used without any modifications. Epoxy resin was taken in beaker and stirred with very low speed (approx. 30 to 40 rpm) to avoid bubble creation. Diluted stabilizers were taken in suitable solvent and added into resin. Then, 3 wt% dry powders were added (filler) to resin under mild stirring conditions, sufficient to "wet out" the surface and create a homogeneous mixture at room temperature. Continuous stirring is advisable. Then, hardener (50 wt%) is added into the mixture and again stirred for 10 min to create homogeneous mixture. The curing time depends upon the amount of hardener added. Now the mixture is poured very slowly into the frame fabricated to get sheet. The sheet is postcured at 50°C in the oven for 24 hours. The fillers (3%), primary

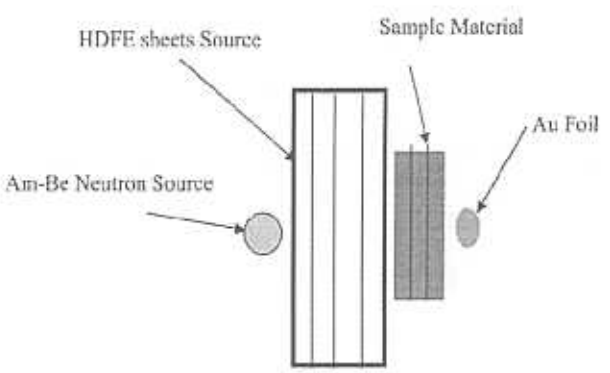

FIG. 2. Setup for linear absorption coefficient. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

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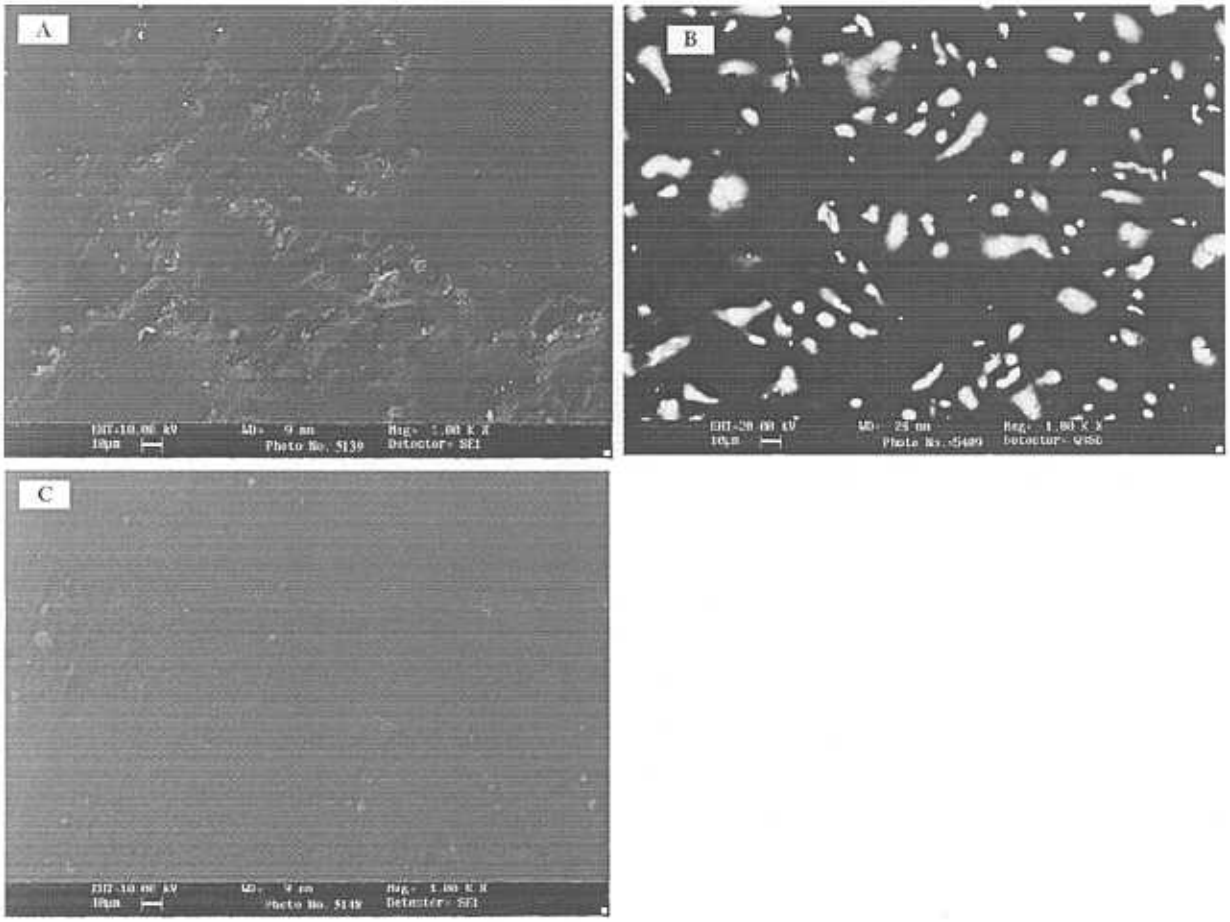

FIG. 3. SEM images for (A) epoxy + graphite, (B) epoxy + lead, and (C) epoxy + boron nitride nanopowder.

TABLE 1. Linear absorption coefficient of developed composites.

Material	μ/cm
Epoxy/lead	0.307
Epoxy/BNNP	0.357
Epoxy/graphite	0.559

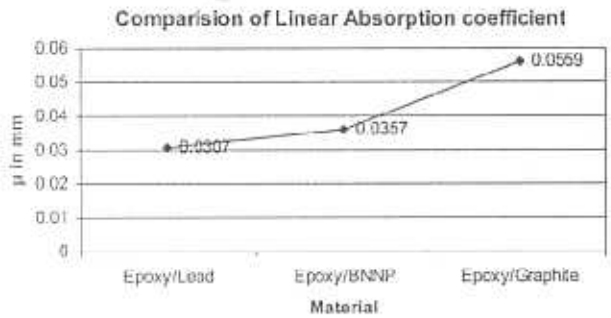

FIG. 4. Comparison of linear absorption coefficient.

stabilizer (0.3%), and secondary stabilizer (0.15%) were taken by weight. Care was taken for uniform dispersion of fillers into resin.

Control of Viscosity. Dispersed fillers have high surface area, and hence the viscosity of the mixture will increase. Filler loading of 3% wt/wt can usually be achieved without exceeding most system capabilities. If viscosity creates a problem, there are two options for controlling it:

1. Increase agitation. Filler/resin mixtures are thixotropic. If shear is increased, the viscosity will decrease.
2. Additional solvent can be added to bring down viscosity, with no effect on dispersability.

Experiment for Linear *Absorption Coefficient*

To find linear absorption coefficient for neutrons, experiment was carried using Am–Be neutron source of strength ~3 × 107 n/s. Neutrons were thermalized by 3 cm thick High Density Polyethylene (HDPE) sheet kept in between source and given sample material as shown in Fig. 2.

Transmitted neutrons were detected by Gold (Au) foils using neutron activation technique. In this technique, Au foil is activated by $Au^{197}(n,\gamma)Au^{198}$ reaction due to neutron interaction. Activated foil is counted for induced 412 keV gammas using well-calibrated high purity germanium (HPGe) detector. The foil activity production of Au198 was estimated by *Eq. 1*.

$$N_{Au198} = \frac{C}{I\gamma \varepsilon\gamma e^{(-\lambda t_c)}\left(1-e^{(-\lambda t_m)}\right)} \quad (1)$$

where C is net counts in photopeak, λ is decay constant of daughter nuclide, ε_γ is photopeak efficiency of gamma ray, I_γ is gamma abundance, t_c is cooling time, and t_m is measurement time.

Thicknesses of materials were 3, 6, 9, 12, and 15 mm. For each thickness, Au^{198} production was estimated. Au^{198} production without sample material was also estimated. For each shot, foils were irradiated and counted for 900 seconds.

Linear absorption coefficient was found by *Eq. 2*

$$\ln [I_0/I] = \mu X \quad (2)$$

where I_0 is Au^{198} production without absorber material, I is Au^{198} production with absorber material of thickness X, and μ is linear absorption coefficient for the absorbing material.

RESULTS AND DISCUSSION

Microscopic Observations (SEM)

SEM images were acquired on a Model LEO 440i at a chamber pressure of 50 Pa. Electron beam of 20 kV was used for scanning to minimize any possible charging effect. The regions of low pixel intensity are attributed to regions composed of polymer resin.

In first composition, the morphology of the sample shows the granular nature of the sample. As graphite powder is black in color, the dispersion is not so visible although the reflections of light on particles show the uniform distribution of fillers (see Fig. 3A).

In the second composition, morphology of the sample shows a very clear dispersion of particles; as lead is a metal, light illuminates the particles and shows uniformity in dispersion (see Fig. 3B).

In third composition, the morphology of the sample shows flat topography and the best uniformity amongst all the three samples. As boron nitride is the nanopowder, the dispersion pattern is the best (see Fig. 3C).

Linear *Absorption Coe*fficient

The points are plotted between $\ln(I_0/I)$ and thickness X (refer Table 1), and it is a straight line; slope of this graph gives the linear absorption coefficient (See Fig. 4)

Looking to the results, it is observed that the absorption coefficient of lead is less than the other two fillers and graphite has the maximum absorption coefficient amongst all the three fillers.

CONCLUSIONS

Best dispersion is observed in the Graphite, Boron and Lead sample respectively. Over all dispersion in all samples are uniform which is necessary to restrict radiation to be passed through. Results of Linear absorption co efficient is as per shown in the graph 1 (see Figure 4). Finally we may conclude that, Graphite/Epoxy has the best Linear Absorption co efficient.

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