To study dispersion pattern of nano filler in polymer composite for Radiation Hardened Package

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Nanocomposite technology is a young material science and plastic nanocomposites are among its first commercial applications. Nanocomposite products improve various properties of many plastics. They are not only used to improve existing products, but also are extending their reach into areas formerly dominated by metal, glass, ceramic and wood.

Main focus of this study is the dispersion of nano filler in epoxy resin to produce radiation hardened plastic packaging which can be used as a shielding against a high frequency radiation attack mainly gamma radiation. The study is carried out as part of research project for a development of Radhard Plastic Package for VLSI. It is mainly focused on dispersion pattern of regular sized fillers against nanofillers and combination of both in sample.

Good nanofillers dispersion in a polymer resin is crucial for achieving performance. Microscope images are also an important part of the quality control function. Few experiments are carried out and microscopic images are also taken to study dispersion in resin.

Small batches using gravity casting method at laboratory with different sizes of same fillers varying from microns to nano and combination of both using compatibilisers to carry out experiments and an exhaustive literature review is also been done to understand the effect on radiation resistance.

Keywords: Nanofillers, Radiation hardened, Epoxy Resin, Plastic package, dispersion

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1 Objective

Main focus of this study is the dispersion of possible nano filler in epoxy resin to produce radiation hardened plastic packaging which may in turn can be used as a shielding against a very high frequency radiation attack mainly gamma radiation. The study is carried out as part of research project for a development of Radhard Plastic Package for VLSI application. It is mainly focused on dispersion pattern of regular sized (microns) fillers against nanofillers and combination of both makes changes in properties of end products.

2 Introduction

Nanocomposite technology is a young material science and plastic nanocomposites are among its first commercial applications. Nanocomposite products improve barrier, flame resistance, thermal and structural properties of many plastics. They are not only used to improve existing products, but also are extending their reach into areas formerly dominated by metal, glass, ceramic and wood [1].

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 composites are made up of just two materials. One material (the matrix or binder) surrounds and binds together a cluster of fibres 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 [2].

Based on the form of reinforcement, common composite materials can be classified as follows [3-5]: 1.Fibre (short and long) reinforced composites; 2.Particle (flakes, beads, spheres, needles, 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.

Precious property of composites is possibility of planning structure in purpose to obtain established characteristics of these materials. 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 their nature, so they are applied as electrically insulating materials. Polymers contain a very low concentration of free charge carriers, and thus they are non-conductive 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.

Graphite is pure carbon in a crystal form much like that of mica–sheets of strongly linked atoms, with very weak bonds between the sheets. This structure makes graphite an excellent dry lubricant wherever temperatures do not get too high. Pencils make use of graphite for the same reason, as graphite rubs off on paper so easily. Graphite is very soft, measuring 1 or 1.5 on the Mohs scale of mineral hardness [6]. Graphite fibers reinforced composites have exceptional mechanical properties which are unequalled by other materials.

The material is strong, stiff, and lightweight. Polymeric graphite fibers composite is the material of choice for applications where lightweight & superior performance is paramount, such as components for spacecrafts, fighter aircrafts, and race cars. Composite materials are extremely versatile. The engineer can choose from a wide variety of fibers and resins to obtain the desired material properties. Also the material thickness and fibre orientations can be optimized for each application. The influence of graphite additions on the friction and wear behavior of moulded epoxy resin has been evaluated using a pin-on-disc wear unit under dry sliding conditions. Graphite addition reduces the friction coefficient. Further, wear loss drops significantly when graphite is present in small amounts in the resin. The tests conducted on the composites containing 3wt% or more of graphite yielded extremely small amounts of wear [7].

The main problem during the production of the composite, that changes properties continuously, is suitably choice of technological parameters. These materials have application in aircraft, automotive industry, space equipment, marine equipment and many others [8-9].

For epoxy, the properties which can be improved by addition of nano fillers are; Higher Tg, Stiffness, Solvent / chemical resistance, Flame resistance, Rheology control, Scratch and marks, Anti-bloom, radiation resistance depending upon type of filler[10].

Gravity casting method is one of the methods relying on gravity without applying pressure. It is possible to produce spatially distribution of component materials by casting step-by-step two or more melts. First of all one of melts is poured and when is partially solidified or crosslinked then the second melt is poured [11].

Good nanofillers dispersion in a polymer resin is crucial to achieving performance. Optical and electronic microscopy technologies are the most common tools. Optical microscopes and proprietary techniques may be utilized to investigate dispersion in various forms of samples. Microscope images are also an important part of the quality control function.

During sedimentation in a column differences in the particle velocity caused by different density or size of the powder particles lead to de-mixing 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 de-mixing and the top of the sediment contains only the powder fraction with the lowest sedimentation velocity [12].

Few experiments are carried out and microscopic images are taken to study dispersion in resin.

3 Experimental

Sedimentation of graphite during gravitational casting produced composites with throughout filler content in specimens. On the basis of research results the specimen thickness was determined i.e. 3 mm. Performed research had a preliminary character because only one polymeric and only one graphite type were applied. It is planned to continue research for other polymeric systems and other fillers.

Very small batches using gravity casting method at our laboratory with different sizes of same fillers (graphite) varying from microns to nano and combination of both using compatibilisers to carry out experiments and an exhaustive literature review is also been done to understand the effect especially on radiation resistance of a sample.

When nanofillers 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 filers 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 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 stabilisers are also used which can absorb the UV, X rays and Y rays and save the material from early degradation. How ever particular fillers are also required which 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 and graphite nanopowder is selected for the purpose but a nano clay is also used to study the dispersion pattern because of its light colour the microscopic view was more favorable. The size of clay and graphite are almost same.

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 was used as filler. The density of the graphite was 0.45 to 0.50 g/Cm^3. Basic characteristics of component materials are shown in tables 1-3 as per suppliers' data.

Table 1. Physical and chemical properties of epoxy resin grade 230.

Form	:	clear low-viscosity liquid
Odour	:	Weak
Epoxide equivalent weight	:	230-250
Viscosity in poises at 25°C	:	15-25
Density g/CM³ at 25°C	:	1.12-1.13
Solubility	:	Aromatic hydrocarbons, alcohols, ketones, esters and glycol ethers

Table 2. Physical and chemical properties of Hardener grade.

Form	:	Brown High - viscosity liquid
Odour	:	Weak
Epoxide equivalent weight	:	230-250
Viscosity in poises at 25°C	:	15-25
Density g/CM³ at 25°C	:	1.12-1.13
Solubility	:	Aromatic hydrocarbons, alcohols, ketones, esters and glycol ethers

Table 3. Physical and chemical properties of Graphite powder.

Form	:	Powder
Oil Absorption	:	100 to 120 % wt/wt
Particle size	:	Average particle size is ~15 microns and Maximum size is 35 microns.
Bulk density	:	0.45 to 0.50 g/cc.
Epoxide equivalent weight	:	230-250

Table 4. Physical and chemical properties of Graphite nanopowder.

Chemical constitution	:	C
Molecular Weight	:	12.01 g/mol
Melting point/freezing point	:	Melting point/range: 3.652 - 3.697 °C
Size	:	< 45 μm
Relative density	:	1,900 g/cm3

3.2 Samples preparation

The specimens were obtained using gravitational casting methods which is one of the technologies relying on gravity without applying pressure. With this method it is possible 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 – 1feet, width – 1 feet depth – 3 mm (Fig. 1) [14].

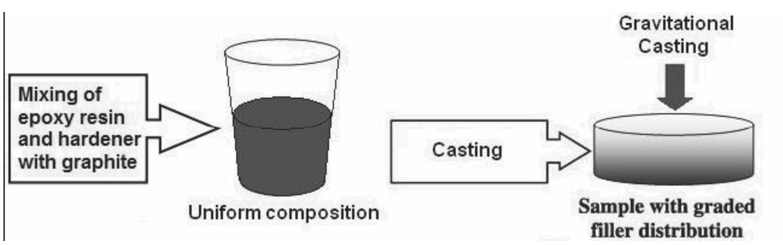


Fig. 1 – Method of Gravity casting for sample preparation

3.3 Procedure:

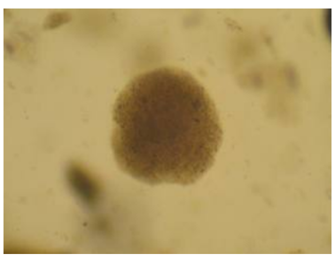
1) Took 400 ml of Epoxy resin in beaker.
2) Stirred with very low speed.
3) Diluted stabilisers in suitable solvent and added into resin with very low speed.
4) Stabilisers were taken 0.3 wt% each.
5) Stirred the mixture very slowly so that bubbles do not create.
6) Added 2.5 wt% dry powders (Filler) to Resin under mild stirring conditions, sufficient to “wet out” the surface and create a homogeneous mixture.
7) Temperature should be room temperature.
8) Continuous stirring is not necessary but it is advisable if practical.
9) Add hardener into the mixture and again stir it for 10 min. to create homogeneous mixture.
10) The curing time depends upon the amount of hardener added.
11) Here 200 ml of hardener is added into the mixture.
12) Now the mixture is poured very slowly in to the frame fabricated to get sheet of 300 mm X 300 mm X 3 mm
13) Covered with toughened glass by applying releasing agent on it.
14) Allowed to be cured for 6 hrs.
15) The cured sheet is kept at room temperature for 8 hrs and then kept into the hot air oven for 24 hrs for post curing.
16) The samples from different regions are cut using jig saw and grinder and observed in microscope by using high brightness (adjustable) illumination with halogen lamp 6V/30W.

3.4 Control of Viscosity:

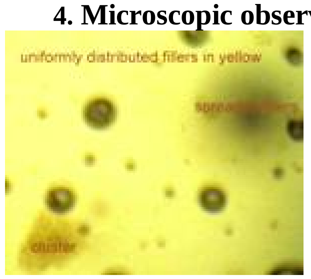
Dispersed nanoclays/ nanofillers have high surface area. The viscosity of the mixture will increase. Nanoclay loadings of 2.5% wt/wt can usually be achieved without exceeding most system capabilities. If viscosity creates a problem there are two options for controlling it:

1) Increased Agitation. Nanoclay/resin mixtures are thixotropic. If shear is increased the viscosity will decrease.
2) resin/solvent. Additional solvent can be added with no affect on dispersability. This option is contingent upon having downstream capability to remove excess solvent under controlled conditions.

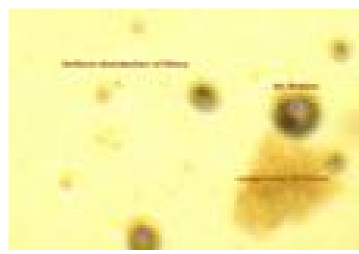
4. Microscopic observations:



Pic:1 of sample having only micro filler



Pic: 2 of sample having only nanofillers



Pic: 3 of sample having combination of micro and nanofillers

Microscopic view of sample with combination of Micro and Nanofillers.

Microscope of model CS – U158 trinocular is used with 100X magnification having high brightness (adjustable) illumination with halogen lamp 6V/30W.

This study was carried out in order to study the dispersion pattern of the gradient composites. On the basis of microscopic pictures the following conclusions were drawn:

1. Very less bubbles are found in micro fillers sample. (Pic.1)
2. The coagulation of powder is found more in micro fillers sample. (Pic.1)
3. More bubbles are found in nanofillers sample. (Pic.2)
4. The distribution of powder was more uniform in nanofillers sample. (Pic.2)
5. Very less bubbles with more uniform distribution was found in combination of 2% of micro fillers and 1% of nanofillers. (pic.3)
6. However few bubbles and very few clusters of powder were also seen in combination of fillers. (Pic.3)

5. Conclusions

1 Addition of graphite filler should be limited to 2 to 3% as on increasing the % of fillers, exfoliation and brittleness of sheet was observed.

2. Combination of Micro and nano fillers can give best results in comparison to only micro fillers and only nano fillers.

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