

## RESEARCH ARTICLE

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# Thermal Properties of Graphene based Polymer Nanocomposites

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## Abstract

**Objective:** Epoxy is a commonly used material for electronic components packaging, yet its inherent thermal resistance can't meet rising demands. To improve the thermal performance of Epoxy material, the high thermal conductivity of Graphene nanoparticles interspersed into the epoxy matrix. This paper focuses on experimental results on the thermal properties of Graphene-based epoxy composite. **Methods:** Scanning Electron Microscopy and Energy Dispersive X-Ray Spectroscopy used for elemental analysis. Guarded Comparative Longitudinal Heat Flow Technique, thermogravimetric analysis, digital scanning calorimeter, and thermogravimetric analysis used to determine thermal conductivity, Coefficient of thermal expansion, specific heat capacity and thermal stability as per ASTM standard respectively. **Findings:** Thermal decomposition temperature enhanced from 170 °C of pure epoxy to 329 °C for epoxy with 0.5 wt% of Graphene nanofiller, and it increased with an increase in filler loading, hence proving the thermal stability of composite improved with the addition of graphene nanoparticles. Maximum thermal conductivity of 0.43 W/mK and thermal diffusivity of 0.686 x10<sup>-6</sup> m<sup>2</sup>/s had recorded for 2 wt% of Graphene additives. Linear thermal expansion coefficient and specific heat reduce for the composite material with an increase in filler. Therefore, this indicates heat resistivity improved with minimum heat storage. An increase in stability, conductivity, and diffusivity with the decrease in expansion coefficient and specific heat capacity due to heat proves a favourable factor for thermally conductive material. **Novelty:** The easy method employed for material preparation, also the use of ultra-low filler content tends to reduce composite cost. Graphene-based Epoxy composites with modified properties represent a conceivable packaging material.

**Keywords:** Thermal Properties; Epoxy composites; Specific heat; Thermal conductivity; Thermal diffusivity

## 1 Introduction

Due to rapid growth in the communication industry, the electronic packaging industry is also experiencing fast upgradation. In addition to bringing exceptional comfort

to consumers, the miniaturization of electronic devices also places a high demand for rapid computing and processing, multifunctioning, and advanced designs. As a result, formulations of complicated circuits result in heat build-up on operating devices, which affects the durability, performance and lifetime of these devices<sup>(1)</sup>. Transporting and dispersing heat is a critical step toward preventing these risks. A good thermally conductive material is essential for the progressive development of electronic devices<sup>(2)</sup>. Electronic applications are most often made from polymers as they offer compatibility, easy manufacturing, inexpensive, lightweight, and commercially viable material. Apart from many applications, thermal management of high-density gadgets prepared from polymer requires the removal of internal heat generation effectively<sup>(3)</sup>. Packing materials can be fractured, delaminated, melted, crept, electromigrated, or even burned when the device temperature exceeds a certain threshold. If the polymers are used as a packaging material, the generated heat is not dissipated in time due to its low inherent thermal conductivity (TC), which can cause localized overheating. Polymers have lower thermal conductivity due to a number of factors, such as disordered phonon transmission, complicated structural characteristics, chemical bonds are weak, the density of atoms is low, and vibrations of molecules being irregular<sup>(4)</sup>. As a result, polymer packed electronic circuits can't ensure the regular operation of circuits, limiting its use in electronic packaging. The good mechanical and adhesive properties, with chemical stability, make epoxy an excellent insulating material for many electronic applications. Although epoxy has a high surface area, that limits its use in many applications due to low thermal conductivity. It is, therefore, imperative that epoxy-based composite materials with higher thermal conductivity are crucial for the development of the electronic sector<sup>(2)</sup>. Epoxy with inherent thermal conductivity for commercial use is challenging and expensive to prepare. This can be achieved by arranging the molecules of epoxy in a specific direction and exerting molecular rigidity. Ensuring phonons are transmitted at a longer mean free path, scattering less, and so thermal resistance is reduced<sup>(5)</sup>. The simplest method is adding high thermal conductivity fillers in the epoxy matrix is the least expensive method to enhance the thermal conductivity of epoxy composites instead of the costly and sophisticated mesogenic elements arrangement in an orderly manner<sup>(6,7)</sup>. The shape, filler quantity, TC, and size of additives are necessary to prepare composite material. Evenly dispersal of fillers in matrix must be achieved by proper technique. composite is allowed to cure under suitable conditions after coating, casting, and removal of impurities. Since Graphene has an incredibly high thermal conductivity made up of a single carbon atom with a 2D structure, making it is easy to form a path for heat to conduct through the matrix<sup>(8)</sup>.

Ganiu et al.<sup>(9)</sup> experimented using DGEBA resin with ultra-low reduced graphene oxide (rGO) nanofiller prepared by In-suit polymerization showing an excellent homogeneous dispersion of filler by stirring. Unmodified epoxy shows TC of 0.19 W/mK whereas maximum loading of filler 0.06 wt% of rGO considered for study report 0.24 W/mK using Laser flash method at atmospheric temperature. High aspect ratio untreated surface Graphene nano particle GNP-5 compared with GNP-C750 of 2 and 5 wt% was dispersed in epoxy by mechanical stirrer with 100:14.5 ratio cured by heating at 125 °C. High TC of 0.37 W/mK and 0.47 W/mK had been reported for 2 and 5 wt% respectively for GNP-5 filler by Laser flash method<sup>(10)</sup>. Full-field image rebuilt to simulate the validation of a polymer with graphene composite heat diffusivity. Thermal diffusivity of  $5.1 \times 10^{-7} \text{ m}^2/\text{s}$  with the maximum conductivity of 0.64 W/mK for 10.0 vol% of Graphene respectively with a deviation of 5% from experimental values<sup>(11)</sup>. Further, heat conduction of graphene with polypropylene, High-density polyethylene, and Polyamide 6 (PA6) had prepared with the heat blend method analyzed by the MTPS device. The research found an increase in TC with the increase in filler content by 306% for the mass fraction of 10% graphene with PA6, also simulation results with the same trend line<sup>(12)</sup>. A hot Disk analyzer had used to determine the TC and thermal diffusivity of the Epoxy with rGO and alumina hybrid composites. TC of 0.3037 W/mK enhanced up to 53%, and diffusivity improved to 60% compared to unfilled epoxy for the 10:0 (rGO-alumina) hybrid ratio<sup>(13)</sup>. 11:9(GNP-SiC) hybrid with bisphenol A epoxy shows a 42% increase in TC compared with 20% GNP alone with epoxy due to a better synergistic effect developed for hybrid composite<sup>(14)</sup>. Most studies examine one or two thermal properties for the prepared composite of different compositions with surface-treated graphene, indicating insufficient factors of composite material to be considered for a particular application where thermal management is of utmost importance. Further, homogeneous dispersibility had achieved at low loading with better chemical stability. This work aims to determine the effect of the low loading of graphene in an epoxy matrix on the thermal properties of composites. Thermal conductivity (TC), coefficient of thermal expansion (CTE), thermal diffusivity(*a*), thermal decomposition temperature, and specific heat (Cp) of prepared materials is determined using the ASTM standard for Ep/G composites.

## 2 Methodology

### 2.1 Materials

An epoxy resin based on Bisphenol A was used, with Araldite HY 951 in 5:1 ratio to form an epoxy matrix, supplied by Ciba-Geigy Company. Thermal contact resistance between the matrix and filler material had reduced by curing the resin with polyether amine<sup>(15,16)</sup>. A 5-10  $\mu\text{m}$  nanofiller graphene had procured by Ultrananotech Private Limited.

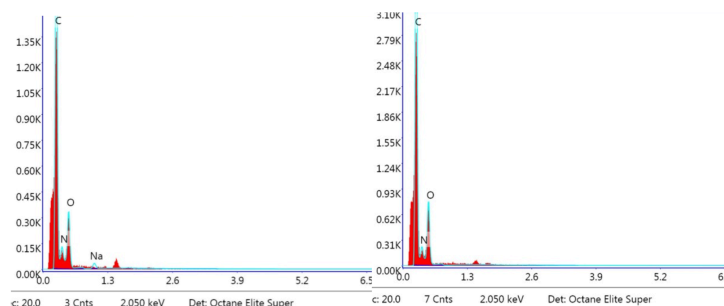
## 2.2 Preparation of Epoxy/Graphene composites

Initially, the measured mass percentage of G is added to acetone and mixed thoroughly by magnetic stirrers for about 20 min to achieve better chemical bonding at atmospheric temperatures. Acetone/G mixture had added to measure wt% of epoxy resin and stirred by magnetic stirrer at 650 revolutions per min for about 90 min, aiding in dispersing the filler mixture evenly in the matrix. To obtain homogeneous dispersal of additive and to distribute it freely and uniformly into a highly viscous epoxy resin, acetone had poured at the desired frequency into a resin. Araldite HY951 is added and stirred for proper mixing for about 5 min, vaporized, and filled into the prepared mould. Recent findings prove that harder used can cure resin at atmospheric temperature without the application of heat in 3 hours<sup>(16)</sup>. Allowed it to cure for 24 hrs in mould at atmospheric temperature further, heated at 60 °C to remove impurities. A magnetic stirrer had adopted for filler dispersion instead of sonication<sup>(17,18)</sup>. Graphene nanoparticles were added into resin without surface treatment thereby minimizing cost and preparation time<sup>(10)</sup>. Epoxy with 0.5wt%, 1wt%, 1.5wt%, and 2wt% of G compositions of the sample had been prepared and renamed as EG1, EG2, EG3, and EG4 respectively.

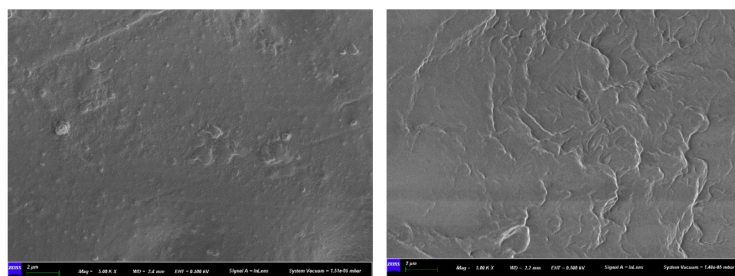
### 3 Results and Discussion

### 3.1 Energy Dispersive X-Ray Spectroscopy (EDS) and Scanning Electron Microscopy (SEM) Analysis

Elemental composition or chemical description in the surfaces of composites is examined in detail with Scanning Electron Microscopy (SEM) using Energy Dispersive X-Ray Spectroscopy (EDS). An electron beam striking the specimen generates X-ray signals measured using EDS by determining their energy and intensity distribution. Each element has its distinguished atomic structure, which permits its electromagnetic spectrum to have a specific set of peaks which is the fundamental principle of SEM<sup>(19)</sup>. Chemical composition can be detected with high precision at a point, line, or area. Figure 1 consists of C, O, and N with 66.93, 22.17, and 10.9 weight% with an atomic % of 71.57 of C, 18.39 of O, and 10.04 of N. The weight percentage of C in EG1 increased compared to neat epoxy, which shows the existence of Graphene in the composite. Graphene is composed of a single carbon atom arranged in a hexagonal pattern. The presence of Graphene in the matrix results in the formation of new materials with revised properties.



**Fig 1.** (a) EDS Analysis of (a) neat epoxy, (b) Epoxy with Graphene (EG1)



**Fig 2.** SEM Image of (a) neat epoxy, (b) Epoxy with Graphene (EG1)

The sample was examined for the existence of G in composite by SEM images. The microstructure formation of fabricated composite material affects the mechanical and heat characteristics of the material, ultimately defining how they are adopted in industrial practices. Microstructure examined with Scanning Electron Microscope XL30 ESEM. Unlike polyurethanes, epoxy exhibits no phase transformations and is homogeneous with a good network. Figure 2 (b) displays light specks in large quantities not visible in Figure 2 (a). These shiny particles are carbon revealing the existence of graphene. The image depicts interconnected filler with resin resulting in uneven dispersal and a homogeneous structure with no cluster.

### 3.2 Coefficient of Linear Thermal Expansion

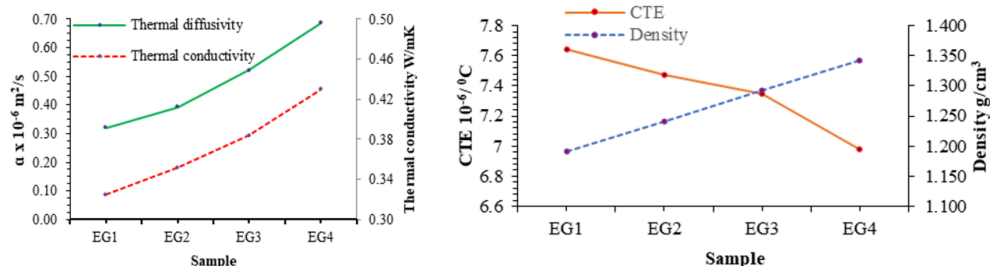


Fig 3. a) TC and TD of EG composites, b) CTE of EG composites

Almost all materials expand when subjected to heat, but the extent of expansion varies with material type. Dimensional changes occur in all directions. Linear thermal expansion refers to an increase in length due to heat. The composite coatings on surfaces of buildings, aerospace craft, aircraft, and submarines became necessary since exposure to changes in surrounding temperature occurs naturally. A composite used as coating should have high thermal stability and a low coefficient of thermal expansion. Deterioration of nanocomposite properties may occur due to self-heating as thermal strain generation at the contact point because of the difference in expansion value between surrounding materials and filler. Composites to be protected effectively against thermal strain caused by thermal expansion differences. The samples had used as per ASTM E228 to determine the CTE of EG. Figure 3(b) shows the CTE of Epoxy-graphene with decreasing trends due to an increase in G content at  $60^\circ\text{C}$ . The lowest value of CTE determined for EG3 with 2 wt% of G as  $6.98 \times 10^{-6}/^\circ\text{C}$ . As G has a high TC, it dissipates produced heat of the nanocomposite, keeping the surrounding temperature equal and decreasing CTE. Recently much simulation work on CTE of EG composite had conducted, an experimental work reports higher value for Epoxy with 1 wt% and 5 wt% GO as  $7.2 \pm 0.6$  and  $5.6 \pm 0.7 \times 10^{-5}/^\circ\text{C}$  below glass transition temperature using a complex composite synthesis process with sonication<sup>(20)</sup>.

### 3.3 Thermal Conductivity (TC)

An important physical property of a material is its thermal conductivity used to assess the thermal performance of construction materials. Thermal conductivity of EG composite is highly affected by their dispersion degree, particle shape, waver, alignment, particle content, and contact resistance due to heat between the fillers and epoxy matrix. Further, a stable enhancement in TC of composite is achieved mainly by the propagation of heat in G due to the diffusion of phonons. Therefore, a well-developed network of graphene particles in the matrix, uniform distribution of filler, and aspect ratio of the particles affect phonon transport. Thermal conductivity enhancement is lower for networks formed with smaller particles because contact resistance is higher, and multiple interfaces cause phonon scattering. ASTM C518 was used to measure thermal conductivity with a sample of  $\phi$  50 mm and 3 mm thick. Figure 3(a) shows the TC of EG with different compositions. The higher value of 0.43 W/mK TC achieved for 2 wt% of G, increase in graphene content increases the heat propagation in graphene particles, leading to better phonon transport and lowering thermal contact resistance. The obtained TC value for EG4 proved enhanced from 0.37 W/mK for 2 wt% GnP filler in epoxy with different epoxy hardener ratios and cured by heating<sup>(10)</sup>. Bisphenol A epoxy with 2 wt% GnP report 0.21 W/mK TC using three roll milling to disperse filler by Laser Flash method<sup>(21)</sup>. An improvement of 225% of TC was achieved from the TC of neat epoxy 0.191 W/mK. Additionally, the literature reports improved performance when G content increased by incorporating it into an epoxy matrix.

### 3.4 Specific Heat of EG composites

#### 3.4.1 DSC and TGA curve Steady

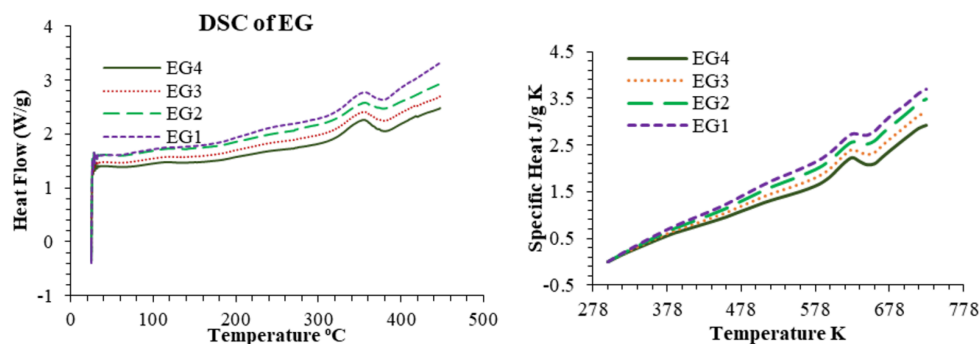


Fig 4. a) DSC of EG composites, b) Cp of EG composites

At a lower heating rate of 20 °C/min, a scan was performed from 0 °C to 400 °C to study in detail that small changes occur in the composite due to heat absorption. DSC and TGA show similar trendlines as in Figure 4a & b. Similarity occurs due to the distribution of filler and the removal of impurities during the material preparation. From TGA curve neat epoxy decomposes at 170 °C, the temperature used for thermal treatment of EG is between 0 °C to 300 °C as no mass loss occurs. The thermal oxidation of epoxy and decomposition of EG occurs between 300 to 800 °C leading to the formation of char. From Figure 5, the maximum thermal decomposition of 414.26, 421.96, 423.46, and 439.84 °C for EG1, EG2, EG3, and EG4, respectively. The thermal stability of EG4 is maximum compared with other compositions indicating better chemical stability and a high-density network obtained between G and matrix at low loading. The  $T_{5\%}$  for epoxy with graphene nanoplatelets powder coating prepared by sonication reports 328 °C for 2 wt%<sup>(22)</sup>. The onset thermal degradation temperature for 0.06 wt% of rGO is 207.1 °C<sup>(9)</sup>. An increase in Graphene content in the matrix increases its resistance.

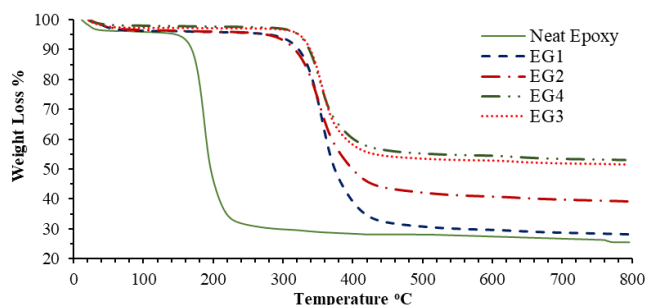


Fig 5. Thermal stability of EG composites

Higher specific heat developed for lower graphene content in the epoxy matrix due to heat dissipation occurs with the presence of G in a composite. Therefore, heat capacity of lower content graphene EG1 is greater than EG4 as in Figure 5, and higher heat energy storage capability occurs with higher specific heat material<sup>(17)</sup>.

### 3.5 Thermal diffusivity (TD) of EG composite

The EG composite diffusivity  $\alpha$  is calculated by :  $\alpha = \frac{K}{\rho C_p}$  where  $\rho$ ,  $C_p$ ,  $K$  and  $\alpha$  are the density, specific heat, thermal conductivity, and thermal diffusivity of EG composite material respectively. The thermal diffusivity of EG measured as 0.321, 0.393, 0.521 and 0.686  $\times 10^{-6}$  m<sup>2</sup>/s as in Figure 3(a). Thermal diffusivity of epoxy with basalt fibre report 0.228  $\times 10^{-6}$  m<sup>2</sup>/s<sup>(23)</sup>. Also, 0.22  $\times 10^{-6}$  and 0.58  $\times 10^{-6}$  m<sup>2</sup>/s had reported from literature for 1wt% and 10wt% of G with epoxy<sup>(24)</sup>. An increase in graphene content increases the diffusivity of the material. A maximum diffusivity had obtained for EG4 with 2wt% of G. The increase in heat diffusion into the material happens due to low specific heat capacity and enhanced TC of material. Improved diffusivity value of EG composite material depends upon filler interface with epoxy and material synthesis<sup>(25)</sup>.

## 4 Conclusion

The EG composite was fabricated successfully by varying graphene content without surface treatment. The thermal properties of EG composites had studied as per ASTM standards. The epoxy with EG4 had a maximum value with thermal conductivity of 0.43 W/mK which represent an improvement of 225%, and thermal stability enhanced to 94% compared with unfilled epoxy. The thermal diffusivity increased linearly and exhibited a maximum value of  $0.686 \times 10^{-6} \text{ m}^2/\text{s}$  for EG4 compared with other compositions indicating better heat dissipation with the addition of G nanoparticles. Enhancement in the properties with an increase in graphene content was achieved compared with neat epoxy. Better compatibility with resin, the transmission of phonon, and distribution of filler in epoxy, and the addition of high TC graphene improve composites' TC and thermal stability by decreasing specific heat capacity, which are highly desirable parameters for thermally conductive materials. Thermally conductive material with reduced coefficient of thermal expansion, less heat storage, and better heat dissipation are all significant features of thermal management materials.

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