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Epoxy-Red Lead Oxide and Hybrid Composites Thermal Properties

Dandapani, Devendra K

***Abstract:** Epoxy resins are used as Lightweight Automotive components, hydrophobic coating, corrosive-resistant thermosetting linings, and other applications. To understand the effect of epoxy resin with Graphene(G)-red Lead oxide (Pb3O4) filler with the application of heat, the thermal behavior of the hybrid composite material is studied in detail. Microstructure characterization of the produced composites had performed employing EDX and SEM. Analyses of the epoxy matrix microstructure have confirmed a relatively uniform distribution of fillers. TGA, DSC, and Longitudinal heat flow methods, were used to determine the thermal behavior of prepared materials by ASTM standards. Heat resistivity and Thermal conductivity of the material increase by adding 0.5 wt% of G initially increase but decreases with an increase in the density of the composite. Specific heat capacity and CTE increase with density for hybrid material. A decrease in Diffusivity indicates a proven thermally insulating material. A simple method adopted for fabrication tends to reduce cost. Epoxy-based Graphene-red lead oxide with modified properties has proven to be a good insulating material.*

***Keywords:** Graphene, Epoxy-Graphene-Lead Oxide, Specific Heat, Thermal Diffusivity, Digital Scanning Calorimeter.*

I. INTRODUCTION

In the composite industry, epoxy resins are among the most important and widely used organic matrixes. They are exceptionally durable and chemically and thermally stable, have excellent adhesive and mechanical strength, are corrosion-resistant, and shrink less during curing than other organic matrixes. Regardless, some disadvantages are low resistance to the formation of cracks, resistivity to wear is low, brittleness, coatings of cured epoxy had low intrinsic toughness, and limited applications. These problems had overcome by adding different kinds of filler to epoxy systems to improve thermal as well as mechanical properties. Recent studies proved that toughness is enhanced by incorporating thermoplastic resin, copolymers, rubber, elastomer, and nanoparticles. The addition of rubber into epoxy resin tends to deteriorate the thermal stability of composites due to the separation of phases during the hardening process [1]. Recent advancement in thermosetting plastic reveals graphene by-products to be the most promising fillers for epoxy matrix reinforcement. Due to graphene's many unique thermal, electronic, and mechanical properties, it has garnered a great deal of interest as a two-dimensional nanomaterial. Graphene nano particles (GnPs) have been reported for hardening polymers and improving electrical and thermal properties and are also employed as tougheners in polymers.

The synergic effect of polymer matrix with graphene nanoparticle has established an excellent EMI shielding material [2], also shown as a potential composite for the extreme environment due to its outstanding corrosive resistance, high stability to heat, flame retardance, and good chemical compatibility [3]. Lead is well-known shielding material, especially for x beams and gamma rays. Even so, these radiations induce a whirlpool effect in a conductor due to the presence of a rapidly changing attractive field. Thus, utilization of lead is unsatisfactory as protecting composite to be an insulator. This is overcome by mixing powders of metal into a polymer grid or a conducting material graphene. Lead (Pb) poses several disadvantages incorporating it for any material, including its toxicity to the environment, health risks, high cost, and difficulty of storage and transportation. To manufacture thermally stable and shielding materials, there is a need for materials with characteristics such as resistance to radiation degradation, nontoxic, and cheap. Thus, Its unique mechanical, optical, and electronic properties make lead oxide a potential industrial application compound [4]. To achieve homogeneous dispersal of additives many researchers used a stirrer with constant speed at the mixing process.

Epoxy-lead oxide shows an excellent radiation shield composite used in diagnostic radiology due to the ease of dispersal of lead oxide in an epoxy grid [5]. PbO Nanostructured formed by chemical bath deposition technique during 10 min deposition revealed high performance in gamma-ray shielding with 70% radiation protection efficiency [6]. Epoxy-lead oxide of 1- 15 µm showed good radiation attenuation properties [7]. The overall attenuation properties of Epoxy-lead oxide remain the same at 1000 kGy but morphological behaviour and mechanical strength initiate fading at 600 kGy [8]. Epoxy with 4 wt% GnPs revealed a 160% increase in flexural modulus and a 100% improvement in thermal conductivity for 5 wt% GnPs composites [9]. Epoxy coating on lead has proven to be a corrosive resistance material by SEM characterization [10]. No attempt had made to study the thermal behaviour of Epoxy- Pb3O4. Thus, the main aim of this work is to investigate the thermal behaviour of Epoxy-Pb3O4 with Graphene(G) following ASTM standards.

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* Correspondence Author (s)
Dandapani*, Department of Mechanical Engineering, VTU-RRC Belagavi (Karnataka), India. Email: dandapani2510@gmail.com
Dr. Devendra K, Department of Mechanical Engineering, SKSVMACET-Laxmeshwar (Karnataka), India. Email: devenk93@gmail.com

II. LITERATURE REVIEW

Gamma-ray active isotopes are now being used more and more in industry, medicine, and agriculture, and various materials of technological and biological significance have to investigate for mass attenuation coefficients.

Materials that can act as shielding structures under harsh conditions of nuclear radiation exposure are always needed. In the case of lower or higher gamma energies, materials with a higher atomic number produce a more effective shield [4], [11]. There is a wide range of effectiveness in radiation shielding materials. The circumambient is typically protected from radiation by materials with a high density, such as concrete and lead. The wide band gap energy and numerous phases of lead oxides make them attractive compounds. PbO-ERGO nanocomposites developed by electrochemical deposition method without reducing agent on ITO electrode are potentially suitable materials for next-generation photovoltaics [12]. Experimentations clearly demonstrate that the polymeric coating used on leaded brass is highly corrosion-resistant. The electrochemical impedance spectroscopy of epoxy-coated leaded brass study proves high protection efficiency against corrosion [10]. Over a certain photon energy range of 0.08MeV to 1.332 MeV it is investigated that epoxy with 40% and 50% lead oxide filler composite exhibits medical gamma-ray waning characteristics, making it suitable for shielding biological radiation against gamma rays at this energy range [13]. Epoxy with lead oxide composite materials of equal proportion demonstrated good mechanical and thermal stability, which made them more suitable for use at about 350 0C without any mass loss. And also reveals good shielding efficiency for gamma rays between 660 and 1371 keV [14]

III. METHODOLOGY

2.1 Materials

Bisphenol-A epoxy (LY556) with TEAT harder (HY951) used a polymer matrix with a ratio of 5:1. Pb_3O_4 of 2 microns with Graphene nanoparticles of 5-10 um had used for the synthesis of composites. To lower thermal transfer resistance between resin and filler particles and also to cure composite at ambient temperature TEAT is used.

2.2 Preparation of Epoxy/Pb_3O_4(EPb) and Epoxy/ Pb_3O_4/Graphene (EGPb) composite.

The investigation concentrated on the thermal properties of synthesized composites; the G loading is kept constant for observation based on previous work. The magnetic stirrer had used with 650 rpm for all compositions for 1 hour and 30 minutes for the homogeneous dispersal of filler in resin. The initially measured quantity of red lead oxide is mixed thoroughly in acetone for about 20 minutes and poured into epoxy resin, the resin-filler mixture is stirred for 90 min then TETA is added to it and stirred further for proper dispersion for 5 min and poured it into the mould to obtain desired dimensions. The procedure is followed again for hybrid material EGPb by adding acetone at regular intervals to recede viscosity. The composite removed from the mould after a day and then heated to remove any residues deposited on the surface of the prepared composite. E+0.5 wt% Pb3O4(EPb), EG+0.5 wt% Pb3O4(EGPb1), EG+1 wt% Pb3O4(EGPb2), and EG+1.5 wt% Pb3O4(EGPb3) was prepared for detail study.

2.3 Characterization.

Field Emission Scanning Electron Microscopy (FE-SEM) using Zeiss GeminiSEM 300 equipment was used to analyse the EPb and EGPb composites' morphology characteristics and chemical compositions. TGA is used to measure the thermal stability of EPb and EGPb composites. TMA is used for investigating CLTE at 60 ^{0}C with ASTM E228 and ASTM D791 for density. DSC is used to find specific heat capacity. TC determined by guarded heat flow technique.

IV. RESULTS AND DISCUSSIONS.

3.1 EDS and SEM Analysis.

Fig 1(a) depicts red lead oxide appearing as a bright circular particle on a rough surface. The Pb fillers are embedded firmly in epoxy due to good chemical compatibility and moderately smaller size particles with epoxy matrix. Only a few agglomerations were found with the EPb composite even though the filler weight fraction was greater quantity than epoxy. The indication of minor clumps is in blurry white patches [15]. The strong tendency of G surfaces to adhere to each other and stack that the nanoparticles had arranged into smooth and glassy structures had shown in Figure 1(b). The EGPb composite morphology shows an even and uniform distribution of fillers into the matrix without clumps. The elemental composition analysis of EPb and EGPb1 composites had exposed in Fig.2(a) and (b). Epoxy with Pb_3O_4 consists of C, Pb, O, and N elements with 64.06, 0.21, 22.09, and 13.64 wt%, respectively. EGPb1 consists of C, Pb, O, and N with 74.55, 0.13, 16.5, and 8.81 wt%, respectively. Lead and oxygen existence ensure the presence of red lead in EPb. An increase in wt% of Carbon in EGPb1 evidence the presence of Graphene nanoparticles as G consists of a single layer C atom.

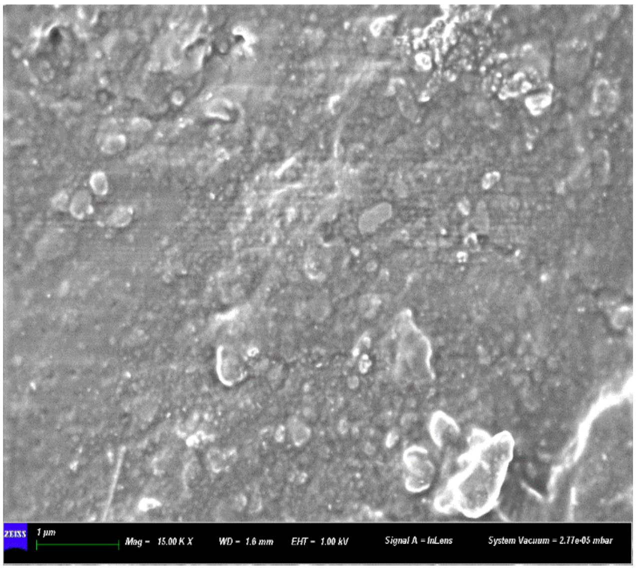

Fig.1 SEM image of a) EPb

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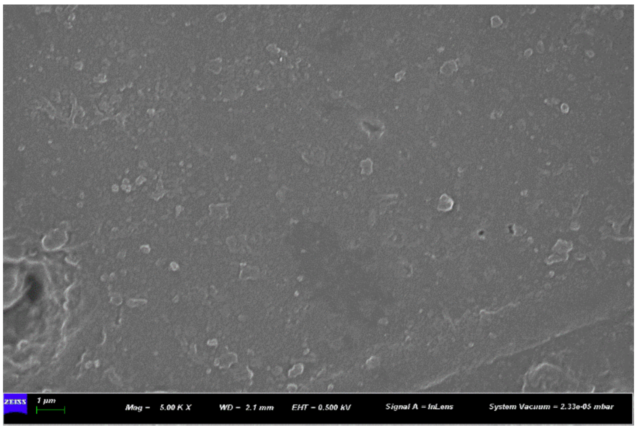

Fig.1 SEM image of b) EGPb1

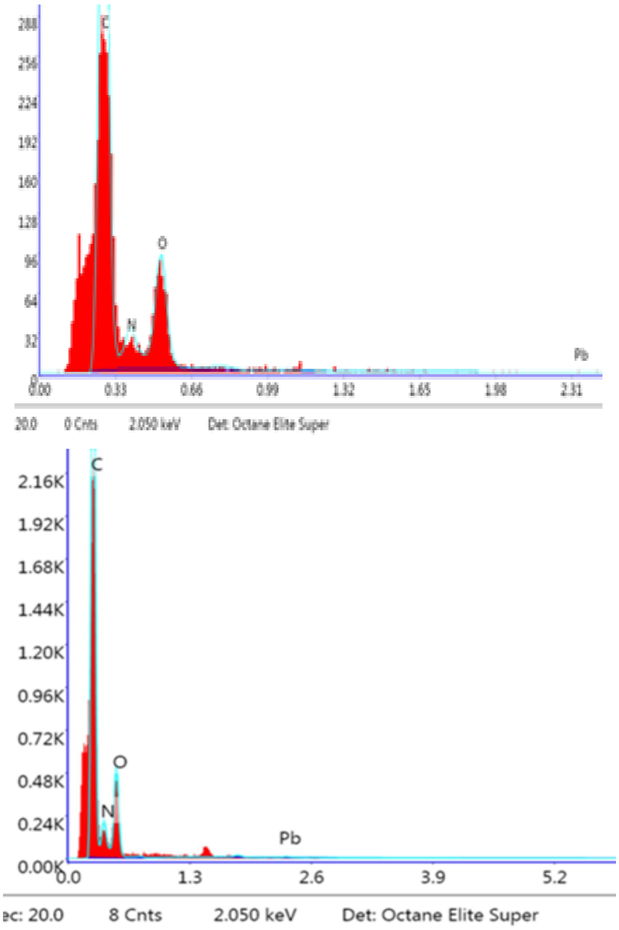

Fig.2 EDS Analysis of a) EPb and b) EGPb1

3.2 Coefficient of liner thermal expansion and density of EPb/EGPb composites

Fig.3 illustrates red lead filler content increases the CTE of EPb composites which is 6.26 x 10^{-5} m^2/s, compared to unmodified epoxy of 6.1 x 10^{-5} m^2/s. Thermal contact resistance at thermal interface materials, which connect different thermal elements, must be minimized to deliver effective heat transfer. The addition of Pb_3O_4 into the polymer matrix results in an increase in the expansion coefficient due to heat. Further, the incorporation of G and Pb_3O_4 indicates an increase in thermal expansion. This proves that EGPb is not a suitable conductive material [16]. A good conductive material should have low thermal expansion coefficient.

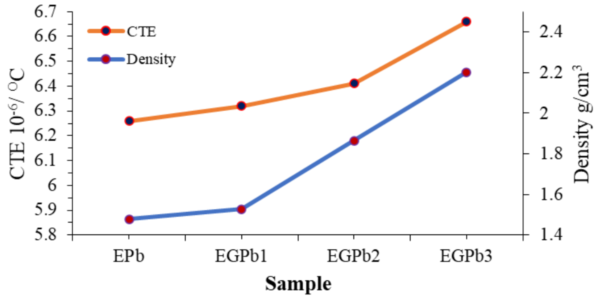

Fig.3 CTE and Density of EPb and EGPb1

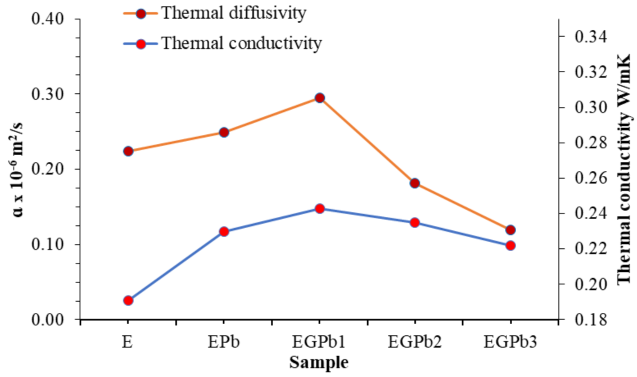

Fig.4 TC and TD of EPb/EGPb composites

3.3 Thermal stability and specific heat capacity of EPb/EGPb composites

The Thermogravimetric analyzer had used to investigate the thermal performance of EPb and EGPb components. The sample had subjected to only heating without considering cooling effects under a nitrogen atmosphere, and a heating program was all set to 25 ^{0}C to 800 ^{0}C with a heating rate of 10 ^{0}C min-1[17].

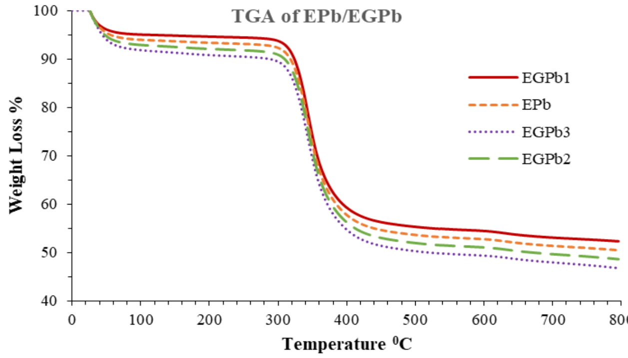

Fig.5 TGA of EPb/ EGPb composites

A sensor had placed to note minute changes happening during the examination. Results from Fig.5 conclude that the maximum decomposition of synthesized composites was above 310 ^{0}C. The enhanced decomposition temperature (T_d) of EPb/EG Pb composites compared to neat epoxy of 170 ^{0}C. The thermal stability of the composites improved to 182% compared with unmodified epoxy. The maximum T_d of 322 ^{0}C obtained for EGPb1 is due to better dispersal of filler particles and excellent chemical bonding between the surface of the filler and epoxy matrix.

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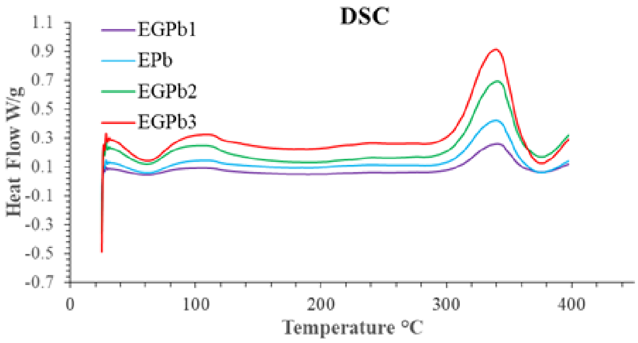

Fig.6 DSC of EPb/EGPb composites

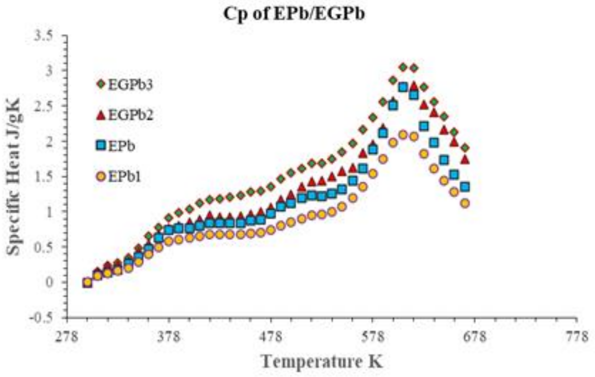

Fig.7 C_p of EPb/EGPb composites

The Digital Scanning Calorimeter had used to determine the specific heat of the red lead oxide composites [18]. The DSC had operated to perform a scan from 0 to 400 ^{0}C at 20 ^{0}C min^{-1} heating rate. To achieve better scan results evenly distribution of filler exists by morphology properties, and the residual is removed during synthesis. The results from Fig.6 exhibit that no mass loss occurs heating EPb/EGPb composites up to 310 ^{0}C, following a similar trend line of the TGA thermogram. The chemical stability of lead oxide composite is excellent however, the addition of G with an increase in Pb_3O_4 results in poor heat flow. EGPb3 reports from Fig.7 a higher CP value compared to EPb composite as G present in composite propagates better heat dissipation.

3.4 Thermal conductivity (TC) and Thermal Diffusivity (TD).

The thermal conductivity had measured by a guarded comparative longitudinal heat flow technique with ASTM standard C 518 with a specimen of Diameter 50 mm and 3 mm thickness. TC of 0.5 wt% increased to 0.23 Wm^{-1}K^{-1} from 0.191 of unmodified epoxy, further increasing the maximum of 0.243 Wm^{-1}K^{-1} with the addition of GnP. When compared to other compositions, EGPb1 exhibited the most effective heat conduction path. Further increases in red lead oxide tend to decrease TC but not less than neat epoxy. In the network with Pb_3O_4, there are too many thermal transfer paths, and they are inefficient in the perpendicular direction [19]. Thermal conductivity enhancement (TCE) had calculated using the equation.

TCE = $(K_c - K_e)/K_e \times 100$, where K_e and K_c are TC of neat epoxy and composites. The highest TC obtained for EGPb1 of 0.245 Wm^{-1}K^{-1} represents an enhancement of 28.27 %. Therefore, the composites synthesized in this work are competitive with neat epoxy.

The TD of the composites is calculated by $\alpha = K/(\rho C_p)$ where K = TC in Wm^{-1}K^{-1}, α = TD in m^{2}s^{-1}, C_p = Specific heat in Jkg^{-1}K^{-1}, and ρ = bulk density in kgm-3 [20]. The TD of composites follows the same trendline of TC following a maximum increment of TD for EGPb1 of 0.295 m^{2}s^{-1}compared to neat epoxy of 0.224 m^{2}s^{-1}. Composites result in a minimum of 0.12 m^{2}s^{-1} for EGPb3. The decrease in TD of composites with an increase in the filler is due to an increase in Specific heat, bulk density, and a decrease in TC. Also due to lead oxide being an insulator, the EGPb1 composite shows maximum TD as the phonon transfer path is most effective for the low filler content of lead oxide. EGPb2 and EGPb3 composites exhibit a good insulating material.

V. CONCLUSIONS

The epoxy with red lead oxide and hybrid epoxy composite had synthesized successfully following a simple technique. The results exhibit a homogeneous dispersal of filler with no agglomeration formation by structural analysis. The hybrid composites EGPb exhibits thermal stability enhancement of 182% for maximum mass loss of composites. The TC of EGPb1 enhanced to 28.27% and TD to 31.7%, and the Cp and CTE value decreased with other composites proving that the low content of Pb_3O_4 with G is a suitable conductive material. Increases in Pb_3O_4 decrease TC, and TD but increase its CTE, Cp, and density, indicating a better insulating material than the neat epoxy as thermal stability had enhanced with all composites.

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AUTHORS PROFILE

Dandapani, Master's in Mechanical Engineering. Author has published 4 papers in international conferences and 2 papers in international journals. Research area of interest includes Thermal science and Nanocomposites. Publication includes 1) "Thermal Properties of Epoxy Nanocomposite materials". Journal of Physics conference series, 2070 (2021) 012171, 2) "Computation of Stress Factors for Counter-Sunk Bolt Fixings in Self Supporting Skins in accordance with DIN 18008-3", 12th International Conference on Researches in Science, Management and Engineering 2017.

Dr. Devendra K, B. E, MTech, Ph.D. in Mechanical Engineering, Working as Professor in, SKSVMACET-Laxmeshwar, India. Author has published several papers in international journals and in international conferences. Publication includes 1) "Thermal Properties of Epoxy Nanocomposite materials". Journal of Physics conference series, 2070 (2021) 012171, 2) Thermal stability and electromagnetic interference of Epoxy-graphene/hybrid composite materials. Mater Today Proc. 2022.

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