Influence of reinforcing filler on thermal conductivity and thermal stability of epoxy composite: A Review

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Abstract – *In this present review discussing the influence of reinforcing filler on thermal conductivity and stability of epoxy composites. Epoxy resins are most employed type of polymer as matric material in composite but having fewer thermally conductive in nature due that metallic, carbon based and ceramic fillers are incorporated as reinforcing into epoxy resin to enhance thermal characteristic of composite. With the incorporation of metallic filler and carbon based filler into epoxy will lead significantly enhancement of thermal characteristic of composite but increases the weight and electrical conductivity too. Ceramic fillers are introduced into epoxy where electrical conductivity and weight is concerned but having low thermal conductivity. Hybrid filler can also be employed as reinforcing to enhance thermal characteristic of composite.*

Key Words: **Thermal conductivity, thermal stability, epoxy resin, reinforcing fillers, hybrid fillers, composites,stea.**

1. INTRODUCTION

Thermal management is concerned important factor in microelectronic as when electrical circuit start to work heat is generated within. This heat should be dissipated to avoid failure of component. Microelectronic packaging plays crucial role in thermal management. For electronic packaging application materials with high thermal conductivity are required to dissipate the heat generated by the electronic component effectively. Polymers are often used in these application in addition to being employed as substrate material because of their low dielectric properties and ease of processing[shin].polymer composite materials have been widely used in the microelectronic industries the aerospace industries, household electrical appliances industries and so on due to their advantages of low density high corrosion resistance, easy processing however their intrinsic low conductivity limits their application to a larger scale currently[12]. Heat conductive filler such as aluminum oxide, boron nitride, silicon nitride, graphite, metal particles, carbon nanotubes, graphene etc. are introduced into polymer to increase thermal conductivity. For application that require both high thermal conductivity and electrically insulating properties, electrically insulating filler such as aluminum oxide, boron nitride, aluminum nitride etc. can be used while graphite, metal particle, carbon nanoparticles and graphene are often utilized in application where electrical insulation is not required[2]. Composite comprises of matrix material and reinforcing material and Composite materials are usually classified by the material used as matrix material such as metal matrix composite(matrix material: copper, lead, magnesium, cobalt, silver, alloys of alluminium, titanium, iron etc.), ceramic matrix composite(matrix material: silicon carbide, silicon nitride, alumina, and various cement etc.), carbon/carbon fiber matrix composites(matrix material: carbon fiber, graphite, grapheme, diamond etc.) and polymer matrix composites(matrix material: polyepoxide, polyester, polypropylene and polyamide etc.). At a present day compactness in electronic packaging is most concerned and polymer matrix composites are lighter in weight and having excellent insulating property and hence most widely used kind of composite [2,28].

1.1 matrix material

Epoxies, bismaleimides, cynate easters, aromatic thermoplastic, thermoplastic polymides are utilized as matrix material for service temperature ranging upto 250 ^{0}C and for above temperature polymides with high transition temperature are existed. Usually uncured epoxy resins have poor mechanical and heat resistance properties however properties could be improved by curing which form three dimensional cross linked thermoset structure.

Table 1 various kind of polymer and their properties

Polymers	Temp	Thermal conductivity (w/mk)
Polyauryllactum(nylon12)		0.25
Nylon6,6(hexamethelene adadipamide)	303	0.43
Polycarbonate polysters,polythers & polycetones		0.3
Polybutylene terephthalate(PBT)	293	0.29
Polycarbonate bisphenol A	293	0.2
Polyoxymethelene	293	0.23
Epoxy resin	293	0.19
Polypropylene	293	0.12
Polyvinylidene chloride	293	0.13
Polyvinyledene fluoride	*293*	0.13
Hydrocarbon polymer polybutane	*293*	0.22

Hydrocarbon polymer polybutadine		*293*	0.22
Polyethylene	Low density		0.33
	Medium density		0.42
	High density		0.52
Polyethelene propelene			0.355
Natural rubber	Unvulcanized		0.13
	Pure gum vulcanize		0.15

1.2 Reinforcing filler materials

Reinforcing material or filler such metallic, ceramic and carbon are incorporated into polymer to enhance thermal conductivity of composite. These filler are employed in continuous fiber, discontinuous fiber, whisker, particles and form.

High conductive metals such Nickel, copper, alluminium, gold, silver etc. are employed as filler. There is quite influence on thermal conductivity of composite because of highly conductive nature of metallic filler simultaneously electrical conductivity also increases thus metallic filler can be utilized where electrical insulation and dielectric breakdown strength are not major concern. Carbon based filler have extremely high thermal conductivity than metallic filler. Due to highly conductive characteristic carbon based filler can be quite effective to enhance thermal conductivity of composite. Carbon based filler are light in weight and hence most widely utilized where weight is major concern such aerospace application. Since carbon based fillers having excellent electric conductivity hence may lead to increase electrical conductivity and dielectric breakdown strength. Ceramic fillers have excellent thermally conductivity and electrically insulating properties. Ceramic having lack of free electron so phonons are causes of heat transfer.

Table 2 various fillers and their conductivity

Filler material /reinforcing material	Thermal conductivity(w/mk)
Copper	483
Silver	450
Aluminum	234
Nickel	158
Gold	345
Silica	1.5
Silicon nitride	200-300
Silica coated aluminum nitride	160-260
Silicon carbide	>270, 490
Boron nitride	250-300
Aluminum oxide	20-29
Beryllium oxide	260, 215.5
Black phosphorus	60-80
Diamond	2000
Carbon black	6-174
Carbon fiber	300-1000
Carbon nanotubes	2000-6000
Multiwall carbon nanotubes	>3000
Single wall CNT's	~3500
Graphene	4840-5300
Graphite	100-400
Graphite(bulk)	~500-2000

2. Influence of fillers on thermal conductivity and stability of composites, associated models and standard test methods

2.1. Metal filler epoxy composite

K shrinivas et al. [4] simultaneously found maximum conductivity 0.68 and 0.62 for Al-epoxy and Cu-epoxy at 40% fraction of filler on composite.

Susumu nikkeshi[5] analyzed thermal properties of epoxy resin composite for various volume fraction of Ni powder and observed decrement on C_p with increasing of volume fraction while conductivity was increased with volume fraction and maximum 0.3w/mk at 29.4 vol%.

Yuan xiang fu et al.[6]prepared epoxy resin adhesive with different filler and found that thermal conductivity of adhesive was about 0.74 w/mk with Cu filler at filling load of 68.25 wt% and 1.11 w/mk of Al powder filler at 69.69 wt% and for diamond epoxy resin thermal conductivity was 0.35 w/mk at 29.46 wt% filling load. Also maximum thermal conductivity of graphite epoxy adhesive reached to 1.68w/mk at 44.3wt% filling load of graphite powder.

Wei yu et al [7] analyzed effect of graphene and aluminum filler on thermal conductivity of thermal grease. With hybrid size alumina filler grease, thermal conductivity increased with increase of small aluminum particle and reached maximum 2.21w/mk at small particle volume fraction of 15%.further increment in small particle volume fraction led to decrease in thermal conductivity of composite. Thermal conductivity of grease reached 2.70w/mk at 63 volume% of hybrid filler. Also high conductivity graphene enhanced the thermal conductivity of composite. With addition of 1 wt% graphene, thermal conductivity of composite reached about 3.45w/mk at 63 total volume fraction% of fillers.

Yi Hsiuan Yu et al [8] fabricated highly conductive film by blending silver nanowires and silver nanoparticle with epoxy resin. Thermal conductivity of composite reached approximately 8w/mk when density ratio of silver nanowires (50phr) and silver nanoparticle (300phr) was 1.512:3.65 and suggests that silver nanowires were better option as interface material rather than silver nanoparticles.

Dilek kumlutas et al [9] studied the thermal conductivity of particle filled polymer composite and found that for Tin particle filled HDPE, thermal conductivity increases from 0.554w/mk for pure to 0.681w/mk and 1.116w/mk for 8 vol% and 16 vol% of filler loading composite.

2.2. Carbon filler epoxy composite

Zhaofu wang et al. [10] studied the effect on thermal conductivity of expanded graphite as filler on epoxy composite and found that thermal conductivity of composite reached 1.0w/mk at filling load of 4.5 wt% and also marked that thermal decomposition temperature of epoxy composite filled with expanded graphite improved to 348 ^{0}C from 318 ^{0}C of epoxy without filler.

Ya fei zhang[11] invested the thermal behavior of graphene foam filled polymer composite and observed that thermal conductivity of composite decreased with increasing volume fraction of foam while conductivity increased with effective radius. Also marked that thermal conductivity of graphene foam epoxy composite almost not relied on contact thermal resistance but when thermal contact resistance surpassed 10^{-7} m^2/kw reduction in thermal conductivity was noticed.

Wenkai xiao et al [12] investigated the effect of ratio of radius to thickness, interfacial thermal conductivity and contact thermal conductivity on thermal conductivity of graphene nanoplatelets-epoxy composite. They observed that thermal conductivity of composite linearly dependent on ratio of radius to thickness.

Asma yasmin et al [13] fabricated Anhydride-cured diglycidyl ether of bisphenol A with graphite platelets filler. Glass transition temperature of composite increased with increasing platelets concentration. Thermal stability was determined by thermogravimetric analysis. Pure graphite exhibits very high thermal stability upto 800 ^{0}C. it was found that epoxy enhanced thermal stability with graphite plalets incorporation.

2.3 ceramic filler epoxy composite

R.kochetov et al. [14] prepared epoxy composite with high thermal conductivity nanopowder (AlN and BN) filler ranging from 0 to 10 wt% filling load. Results showed that for any given filler loading AlN filled sample had higher conductivity than BN filled sample. With 10 wt% filling of AlN, thermal conductivity of composite reached 0.198w/mk which is 18% higher than value 0.168w/mk of pure epoxy while 10 wt% filling of BN, thermal conductivity of composite reached 0.191 w/mk which is 14% higher than value 0.168 w/mk of pure epoxy.

Yunsheng Xu et al [15] prepared electrically insulating polymer matrix composite. Polyvinylidene fluoride (PVDF) or epoxy was matrix material while AlN whisker or particle and silicon carbide whisker were chosen as filler. Predicted that thermal conductivity increased with filler volume fraction of various type of AlN particle and greatest mean particle size of 115 μm gave composites with highest thermal conductivity. The combination of whisker and particles at appropriate ratio gave highest thermal conductivity of 11.5w/mk at 60% total filler volume fraction PVDF matrix composite. Also found that combination of SiC whisker and AlN particle at volume ratio of 1:6 gives PVDF matrix composite of thermal conductivity than SiC and AlN PVDF matrix composite alone.

Alok Agrawal et al [16] developed mathematical model of thermal conductivity of particulate filled polymer composite and validated it by experiment taking AlN and Al_2O_3 as filler for epoxy based composite. Found that 50 wt% of AlN enhanced thermal conductivity of epoxy by 446% whereas Al_2O_3 by 443%.

Sudipta halder et al [17] examined thermophysical properties of ZrO_2 epoxy nanocomposite using thermogravimetric analysis and significant increase in decomposition temperature at 5wt% particle concentration was observed. thermostabilty and activation energy of decomposition also enhanced.

Prem prakash et al [18] analyzed heat conductivity of zirconium filled epoxy composite numerically using finite element method and noticed that thermal conductivity of composite increases with increase in vol% of zirconium powder filler. At 11.31vol% of zirconium powder filler thermal conductivity was increased by 73% of pure epoxy.

Yeon Kyung shin et al [19] investigated the effect of BN content and particle size on thermal properties of BN-high density polyethylene composite and predicted that thermal diffusivity and thermal conductivity increases with increase in BN content and size of particle.

Yicheng wu et al [20] analyzed numerically thermal conductivity of epoxy matrix composite incorporated of ZrB_2 and found that thermal conductivity increased with increasing of ZrB_2 loading ranging from 0 wt% to 16 wt%.

2.4 Review on hybrid filler epoxy composite

K. srinivas et al. [4] studied the effect of mono Al/Cu and hybrid Al-Cu particulate on thermal conductivity of epoxy composite. Maximum thermal conductivity such as 0.68 W/mk and 0.62 W/mk were observed for Al-epoxy and Cu-epoxy simultaneously at 40% fraction of filler. Also observed that hybrid 35Al-5Cu- epoxy exhibits higher thermal conductivity of 0.69 W/mk. Hence result shows that epoxy composite with hybrid filler enriching aluminum content exhibits higher thermal conductivity than filler Al/Cu alone. Alok agrawal and Alok sathapathy[21] evaluated mathematical model for effective thermal conductivity of polymer composite with hybrid filler of two dissimilar particulates and estimation were validated through experimental result. Epoxy and polypropylene were picked as matrix materials and AlN and Al_2O_3 were conductive filler and pine wood dust and rice husk were chosen as insulative filler. Effective thermal conductivity of polymer with conductive and non-conductive particulate filler were estimated using the proposed model which shown agreement with experimental model.

Wenhue yuan et al. [22] fabricated thermal conductive adhesive epoxy composite taking AlN, natural graphite, and graphene oxide and hybrid graphene oxide-AlN as filler. Enhancement in thermal conductivity of adhesive was simultaneously marked as 2.24w/mk at 70wt% filling of AlN, 0.88w/mk at 40wt% filling of natural graphite and 1.22w/mk at 8wt% filling of graphene oxide. Highest thermal conductivity about 2.77 w/mk of adhesive filled with hybrid filler containing 50wt% AlN and 6wt% graphene oxide was observed. Result remarkably shown that enhancement in conductivity of adhesive composite filled with hybrid filler exceeding adhesive composite filled with particulate alone.

Fan long jin and Soo Jin park [23] investigated the thermal properties such as curing behavior, thermal stability and thermal mechanical property for epoxy resin/hybrid filler composite fabricated from diglycidylether of bisphenol-A as the epoxy resin and nano Al_2O_3 and nano-SiC as filler and noticed that Thermal stability at 800 ^{0}C of both composite increased linearly with increasing filler content. The T_g of both composite was detected 10 ^{0}C higher than neat epoxy.

Geon Woong Lee et al [24] studied the polymer composite filled with various inorganic filler with different shape and size of single particulate or combination.lee et al. found that composite containing hybrid filler of spherical and fibrous shape have enhanced thermal conductivity at low to intermediate filler content. Also examined the effect of filler treatment on thermal conductivity and observed that due to introduction of coupling agent thermal conductivity had been improvement for all composite.

Krishnamachar Srinivas and Mysore Siddalingappa Bhagyashekar[25] studied the thermal conductivity of epoxy resin composite containing three different particulate filler such as graphite, silicon carbide and hybrid graphite-silicon carbide. Maximum thermal conductivity of Gr- epoxy and SiC-epoxy were observed 0.64w/mk and 0.68w/mk respectively at a filling load of 40 wt%. Also thermal conductivity of hybrid composite containing 20SiC-20Gr-epoxy was observed 0.71w/mk which is highest among composite containing only particulate.

Srinivas V Giddappanavar et al.[26] studied the influence of filler material like SiC and ZrO_2 on thermal properties of carbon epoxy composite. Thermal diffusivity of carbon-epoxy composite containing 20% SiC increased to 0.412 mm/sec at 40 ^{0}C and carbon epoxy containing 20% ZrO_2 increased to 0.272 mm/sec respectively which higher than value 0.221 mm/sec of carbon epoxy without filler at 40 ^{0}C. Thermal conductivity of carbon epoxy containing 20% SiC and 20% ZrO^2 increased from 0.332w/mk to 0.605w/mk and 0.548w/mk respectively.

Wenying Zhou et al. [27] noticed that thermal conductivity increases to 1.49w/mk with addition of SiC into Si_3N_4 filler silicon rubber which is slight higher than the value 1.34w/mk of Si_3N_4 silicon rubber composite at a total volume fraction of 50%. Also found that the initial decomposition temperature of hybrid filler silicon rubber composite enhanced than pure silicon rubber.

2.5 thermal stability test of epoxy composite

Zhoufu wang et al [10] evaluating the thermal stability of pure epoxy and epoxy composite by TGA and found that initial decomposition temperature (corresponding to the decomposition of 5 wt%) of pure epoxy, 4.5 wt% expanded graphite filled epoxy and 4.5 wt%(KH550@expanded graphite) filled epoxy composite were recorded 318 ^{0}C, 329 ^{0}C and 348 ^{0}C respectively and observed that KH550@expanded graphite filled epoxy composite exhibits better thermal stability than pure epoxy and untreated expanded graphite filled epoxy.

Yuezhan feng et al [31] plotted TGA curve of epoxy/Al_2O_3 and revealed that their thermal degradation behaviors have little change by adding fillers except for the yield of residual char and observed that Incorporating Al_2O_3 into epoxy results in a noticeable decrease in thermal stability due decreasing of heat capacity of epoxy. By contrast, thermal stability of epoxy was improved by Al_2O_3@HGO hybrids due to restriction of chain movement by the reinforced interfacial interaction.

Huei Ruey Ong et al [32] analyzed thermal behavior of the epoxy by TGA at a constant heating rate of 10^{0}C/min in the temperature range of 40 to 600 ^{0}C and studies were conducted in inert atmosphere to eliminate the diffusion of oxidant. 5–10% weight loss was observed in 100–150 ^{0}C range of temperature due to the adsorbed solvent. The TGA curves were plotted and noticed that blend resins were degraded in a single step pattern leaving 10-17.5% residue above 580 ^{0}C. Initial degradation temperature for alkyd/epoxy (30:70) was 280 ^{0}C which is shifted to 300 ^{0}C with incorporation of Cuo particle into alkyd/epoxy blend.

P. Anithambigai et al [33] traced TGA curve of cured DGEBA polymer filled with 50 wt% and 60 wt% Al fillers respectively. From the thermograph it is observed that first degradation steps of both cases ware similar approximately 108⁰C. The second degradation step was recorded to be at 207 ⁰C for 50 wt% Al filled epoxy but in case of 60 wt% Al filled sample this decomposition occurred at much earlier temperature of 199 ⁰C. from the observation they suggested that the chosen DGEBA polymer composite filled with Al can be employed as thermal interface material for the LED as the decomposition temperature of composite was much higher than the maximum temperature that can be attained by the LED.

Votarikari Naveen kumar and VSS Shrinivas[34] conducted a TGA on sample of natural rubber based nanocomposite filled with Boron nitride in the temperature range of 0-600 ⁰C. It was found that with the dispersion of boron nitride into rubber significantly enhanced the thermal stability of composite.

2.6 theoretical models of thermal conductivity developed for epoxy composite

Many mathematical and empirical models have been proposed for binary and hybrid mixture to predict thermal conductivity of composite. Some models are listed below:-
(1)Parallel model (linear mixing rule)

$$k_e = (1-\emptyset)k_m + \emptyset k_f$$

(2)Series model (inverse mixing rule)

$$\frac{1}{k_e} = \frac{1-\emptyset}{k_m} + \frac{\emptyset}{k_f}$$

These models are also called as rule of mixture.
(3)Maxwell model

$$\frac{k_e}{k_m} = 1 + \frac{3(k_f - k_m)}{k_d + 2k_f}\emptyset$$

(4)Bruggemen model

$$(1-\emptyset)\frac{k_m - k_e}{k_m + 2k_e} + \emptyset\frac{k_f - k_e}{k_f - 2k_e} = 0$$

(5)Agari-Uno model

$$\log k_e = \emptyset C_2 \log k_m + (1-\emptyset)\log(C_1 k_f$$

(6)Effective medium theory (EMT)

$$k_e = \frac{1}{4}\left(\gamma + \sqrt{\gamma^2 + 8k_f k_m}\right)$$

$$\gamma = (3\emptyset - 1)k_f + [3(1-\emptyset) - 1]k_m$$

2.7 standard testing methods for thermal characteristic composites

Steady state method, thermal conductivity is evaluated based on heat flux and temperature gradient measurement. On the other hand in transient method thermal conductivity is calculated by thermal diffusivity and rise in temperature depends on time to reach maximum [2, 30].

Table 4 various thermal conductivity test method

Method		Temp range	Conductivity range	Test standard
Steady state method	Guarded hot plate method	80-800K	>0.8	ASTM C177, ISO 8302, EN 12667
	Axial flow method	90-1300K	0.2-200	ASTM1225
	Heat flow meter method	253-532K	>10	ASTM C518 ASTM E1530 ISO8301 EN12667
	Pipe method	293-2770K	0.02-200	ISO8497
Transient method	Laser flash method	373-3273K	>0.01	ASTM E1461 ISO 22007-4 ISO 18755
	Transient hot wire method	293-2773K	<25	ASTM-C 1113 ISO 8894-1 ISO 8894-2
	Transient plane source method	20-1273K	0.005-1800	ISO 22007-2

3. CONCLUSIONS

Thermal conductivity and stability of epoxy composite incorporated various kind of Filler and their concentration o is reviewed in this review paper and based on this review following thing can be concluded.

- Epoxy resins having excellent incorporating tendency of fillers so homogenous composite can be achieved hence most employable kind of polymer as matrix material.
- Thermal conductivity and stability of epoxy composite is solely rely on both matrix material and filler and hence with increasing of filler

concentration thermal conductivity of composite will also increases.

- Metallic fillers such as copper, silver, alluminium etc. having excellent thermal conductivity in nature. Increasing of filler loading leads noticeable enhancement in thermal conductivity of composites.
- Carbon based fillers have larger thermal and electrical conductivity and hence small amount of weight fraction significantly enhance the thermal conductivity of composite so where electrical conductivity is not major concern can be utilized.
- Ceramic fillers such as BN and AlN filler are most applying filler due to their light in weight and insulating characteristic but fewer thermally conductive than metallic fillers and carbon based fillers and hence enhancement on thermal conductivity is low compare to metallic and carbon based filler but enhancement of conductivity can be improvised by hybridization

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