A review on thermal properties of epoxy composites as thermal interface material

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Abstract - *Heat dissipations in electronic devices have become a challenge and area of research for thermal management. Thermal interface materials are used to improve the heat transfer in electronic devices. Epoxy resin is widely used as a thermal interface material because of its excellent property over other thermal interface materials. The past researches show that the incorporation of filler materials of high thermal conductivity improves the thermal conductivity of epoxy composites, consequently improves the heat transfer in electronic devices. In this review paper the need of thermal interface material and epoxy as a thermal interface material are discussed. The review paper also includes the various types of filler material used for thermal conductivity enhancement of epoxy composites and their effect on thermal properties of epoxy composites.*

***Key Words*:** Thermal conductivity (TC), Thermal interface material (TIM), Epoxy resin, Filler material, Thermal properties, Thermo-gravimetric analysis (TGA).

1. INTRODUCTION

Thermal management is critical to the performance, lifetime, and reliability of electronic devices. With the miniaturization, integration of electronics and the development of new filler materials lead to the emergence of new applications such as light emitting diodes, aerospace electronic packaging and in various electronic devices thermal dissipation becomes a challenging problem. Addressing this challenge requires the development of novel polymer-based composite materials with enhanced thermal conductivity [1, 2].

Thermal management has become one of the most critical issues in the field of electronic devices owing to the continuous increase in power density and consumption and the transfer of heat to heat sinks are indispensable for protecting modern electronics from thermal failure. The plan area of CPUs has been steadily decreasing while the power dissipation has been increasing to cover the demand of modern age. When electronic products are operated, tremendous heat is generated. For example, die size of the Pentium 2 was 25.4 mm square and the power dissipation was about 33 W. However, the Pentium 4 die is 12.5 mm square and will dissipate up to 80 W. It has been seen that the power dissipation from the CPU are in the range of 20 to 150W [3].

It is well known that the reliability of an electronic device is exponentially dependent on the operating temperature of the junction, wherein a small increase of 10-15°C can result in a two times decrease in the lifespan of the device [4,5]. And also the stability of an electronic device will be decreased 10% by per 2°C rise in temperature when they are working for long time [6]. Thermal management in conventional electronics was difficult because the conducting path between heat sources and heat sinks were made of rigid materials that possess high thermal conductivity, such as metals. It is hard to make a perfect contact between heat sinks and electronics because of the differences in the flatness between the two surfaces because there are only three contact points between two rigid planes, which causes very poor thermal contact [7]. Therefore, there is need of a soft, deformable material capable of forming low-thermal-resistance contacts in the interface between the electronics and heat sinks. The general method to do this is to fill the air gap by using an elastomeric material with high thermal conductivity in the interface between the heat sink and the heated device. Such materials are called thermal interface materials (TIMs). TIMs conduct heat more effectively to the heat sink than air and thus they reduce the resistance to heat transfer caused by air voids in the device.

1.1 Theory of thermal interface material

Fig (1) shows the location of TIM in typical electronic packaging. Thermal interface resistance is a measure of how well heat is transferred across the interface of two mating rigid surfaces, such as a CPU and a heat sink baseplate; for lower value of resistance the heat transfer will be higher (fig.2).
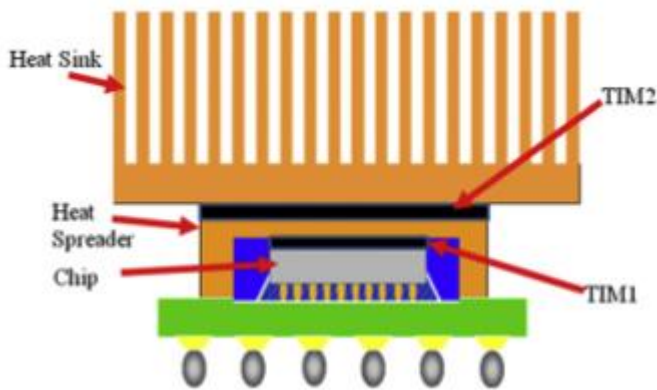
Fig-1: Typical chip package, highlighting location of TIM1 and TIM2

Gwinn et al. [3] has explained more clearly the concept of TIMs. In the case of the CPU–heat sink interface, the interface resistance (per unit area) R_{int} is defined as

$$R_{int} = \frac{T_{cpu} - T_{hs}}{Q} \qquad (1)$$

Where T_{cpu} is the CPU (or electronic packaging) surface temperature, T_{hs} is the heat sink base plate temperature and Q is the power per unit area of heat emitting component.

The interface resistance is generally expressed in K cm^2/W (or in K in.^{2} /W). Fig. 2(b) and (c) shows that the CPU and heat sink surfaces being joined contain a combination of surface roughness and surface non-flatness. The micro roughness on a macroscopic non-planar is in the form of a concave, convex or wavy surface, resulting in as much as 99% of the interface area between the heat sink and CPU being separated by air filled gaps (Fig. 2a) unless a TIM is used. Theoretically, the mating surfaces will only contact each other at discrete points and remaining is filled with air. Because air is a very poor thermal conductor (k_{ai}=0.026 W/m K at room temperature), it acts as a thermal barrier preventing efficient heat transfer across the interface.

If no TIM is used to enhance heat transfer across the interface, the total interface resistance, R_{int} is due to contact resistance, $R_{contact}$ as shown in Fig. 3a. Contact resistance, $R_{contact}$, is composed of two parallel resistances: (1) conduction resistance at the points where the mating surfaces contact and (2) conduction and/or radiative resistance across the gaps of non-contacting area. The existence of TIM material adds a thermal conduction resistance across the material of thickness t.

$$R_{int} = R_{contact1} + R_{cond} + R_{contact2} \qquad (2)$$

And

$$R_{cond} = \frac{t}{k_{TIM}A} \qquad (3)$$

Where $R_{contact1}$ the contact resistance between the TIM and the CPU surfaces is, $R_{contact2}$ is the contact resistance between the TIM and the heat sink baseplate and R_{cond} is the conduction resistance across the thickness of the TIM. k_{TIM} is the TIM bulk thermal conductivity, t is the thickness of the TIM and A is the surface area of the electronic component across which heat transfer occurs.
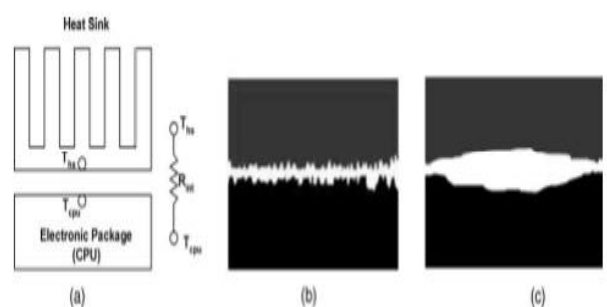

Fig -2: (a) definition of thermal interface resistance, R_{int} (b) heat sink and CPU surface roughness, and (c) poor heat sink and CPU surface flatness.

1.2 Methods to reduce contact resistance

The contact resistance in the interface area may be reduced by the following two methods: (1) increasing the area of contact points, accomplished by (a) increasing contact pressure which will 'flatten' the peaks of the micro roughness and reducing any non-flatness or (b) reducing the roughness of the surfaces before the interface is formed by grinding the surfaces to remove non-flatness and (2) using a TIM of high thermal conductivity that can able to conform the imperfect surface features of the mating surface [3].

Load constraints on electronic components and circuit boards make it difficult to use high contact pressure. Manufacturing highly finished surfaces to eliminate micro roughness is not practical due to cost constraints. Therefore, the practical alternative is to use a TIM with high thermal conductivity. Any interstitials substance that fills the gap between the mating surface and whose thermal conductivity is higher than air will reduce the contact resistance. Gwinn et al. [3] has mentioned the following characteristic that The ideal TIM (Fig. 2b) should have:

1. High thermal conductivity.
2. Easily deformed by small contact pressure to fill the all uneven areas of both mating surfaces, including surface pores, reducing $R_{contact}$.
3. Minimal thickness i.e. viscosity.

4. Would not leak out of the interface.
5. Would maintain performance indefinitely.
6. Non-toxic and eco-friendly.
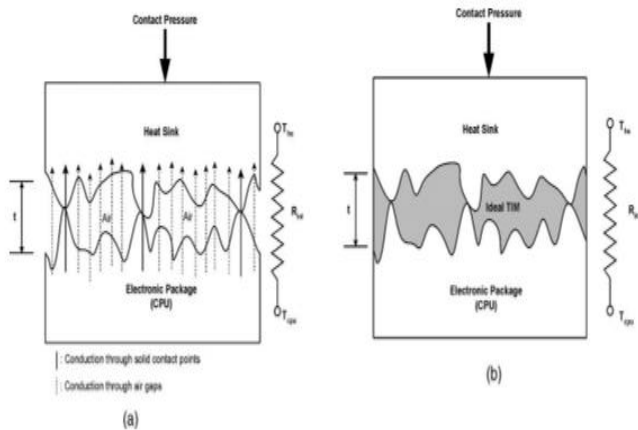7. Manufacturing friendly (easy to apply and remove).
8. Low cost.

Fig -3: Exploded view of thermal interface when (a) no TIM is used. Note R_{int} is composed only of contact resistance $R_{contact}$, and (b) when an ideal TIM is used.

Ideal TIM has not yet been discovered, but there are several options available to provide reduced interface resistance. There are many types of TIM commercially available like thermal greases, elastomeric pads, solders, phase change materials (PCM), gels and adhesives [3, 7, 8 and 9]. Out of them Epoxy resin has been extensively applied in electronics, insulators, printed circuit boards, semiconductor and packaging materials as adhesive due to superior properties such as low shrinkage, low thermal expansion coefficient (TEC), good adhesion and resistance to thermal, physical and chemical stability [10], heat resistance, high adhesive strength, good impact resistance, high strength and hardness, and high electrical insulation [11]. However, the thermal conductivity of epoxy resin is generally low (0.2 W/m k). Therefore, it is necessary to exploit effective method to improve the thermal conductivity of the epoxy resin. An attractive method is to add high thermal conductivity substances which are called fillers to the epoxy matrix.

Epoxy resins were discovered in 1909 by Prileschajew. Epoxy resins can be defined as low-molecular-weight pre-polymers containing more than one epoxide group of the form.
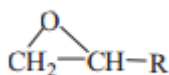
Epoxy resins are thermosetting polymers, which are cured by using wide variety of curing agents via curing chemical reaction. Generally the word 'epoxy resin' employs for both of resin and its curing agent. The properties of epoxy resins mainly depend on specific combinations of the type epoxy

resins and curing agents used. Jin et al. [12] have studied various types of epoxy resins and curing agents and also discussed their synthesis and application area.

2. THERMAL PROPERTIES OF EPOXY COMPOSITES FILLED WITH DIFFERENT KINDS OF FILLER

2.1 Various filler materials used for enhancement of thermal conductivity of epoxy resins

From past few decades, extensive researches are going on the heat transfer improvement of low thermal conductivity materials by adding a few percentages of high thermal conductivity materials which are called filler, especially in the topic of the Nano fluids [13] and polymers [14]. Also, metal meshes and foams have been used in the thermal storage systems to improve the thermal conductivity of the systems. A few theories have been suggested on the heat transfer enhancement in Nano fluids or composite materials.

Fillers may be in the form of fibres or in the form of particles uniformly dispersed in the polymer matrix. The thermo-physical properties of fibre-filled composites are anisotropic, except for randomly dispersed fibres; while the thermo-physical properties of particle-filled polymers are isotropic [15]. The size of the fibre or particle may be of the order of Micro or Nano. Various types of fillers used in epoxy composites are as follows-

(i) Metallic fillers

Various metallic fillers, such as nickel [16, 17], copper [17, 18], aluminium [18], silver [18] etc. have been widely used in epoxy composites for improving TC. In general they could be quite effective in increasing TC of composites compared to the pure epoxy matrix. Adding metallic fillers into epoxy may also cause substantial increase in electrical conductivity.

(ii) Carbon based materials

Carbon-based materials, including graphite [15, 18, 19, and 20], graphene [21, 22], diamond [18], carbon nanotube (CNTs) [23], carbon fibre [24] etc. have been widely studied as heat conductive fillers because of their high intrinsic TC. Even a very small loading of carbon-based fillers can significantly increase the TC of composites.

(iii) Ceramic fillers

Ceramic fillers have been widely studied for thermally conductive and electrically insulating composites due to their inherent properties. Ceramic fillers include metal and non-metal compounds like fillers such as alumina (Al_2O_3) [18, 25], ZrB_2 [26], aluminium nitride (AlN) [27], boron nitride (BN) [27, 28], silicon carbide (SiC) [29] etc. have been commonly used as fillers into epoxy composites.

(iv) Hybrid fillers

Hybrid fillers are commonly used in the thermally conductive composites, formed by mixing fillers with different sizes or types in order to form large thermally conductive network by building bridges between fillers and maximizing the filler packing density. One more advantage of such a system is that it may help to significantly reduce the overall filler loading, thus reducing the system viscosity which improves the thermal conductivity of the matrix.

For the some research above-mentioned, very high filler loading has been incorporated into epoxy matrix to achieve percolation thresholds and form continuous heat conducting path in the epoxy matrix, currently higher than 60 wt%. However, high filler content into epoxy leads to high density and inferior mechanical properties, resulting in negative effects for the adhesive.

Incorporation of high filler loading into epoxy matrix, currently higher than 60%; to achieve percolation thresholds and to form continuous heat conducting path in the epoxy matrix leads to loss of the mechanical integrity of the matrix [4] and inferior mechanical properties, resulting negative effects for adhesive [6]. In addition with these high filler loading causes poor processability and high cost.

2.2 Thermal properties of epoxy composites

The thermal conductivity measurement techniques can be divided into two groups. One is steady-state methods and another is transient methods. Steady-state methods are used when the system has achieved stability, while the transient methods are applied during the process of heating up or cooling down a material.

Chen et al. [1] and Suganthi et al. [12] have described these two types of measurement methods but Chen et al. [1] has investigated further the different techniques associated with these two methods. Table 1 show various information's regarding thermal conductivity measurement techniques of polymers.

Table-1: comparison of different thermal conductivity test methods [1]

Methods		Temperature range (K)	TC (W/m K)	Accuracy	Test standards
Steady state methods	Guarded hot plate method	80–800	<0.8	2%	ASTM C177 ISO 8302 EN 12667
	Axial flow method	90–1300	2-200	2%	ASTM E1225
	Heat flow meter method	253–523	<10	3%	ASTM C 518 ASTM E1530 ISO 8301 EN 12667
	Pipe method	293–2770	.02-200	2%	ISO 8497
Transient method	Laser flash method	373–3273	>.01	3–5%	ASTM E1461 ISO 22007-4 ISO 18755
	Transient hot wire method	293–2273	<25	1-10%	ASTM-C 1113 ISO 8894-1 ISO 8894-2
	Transient plane source method	20–1273	.005-1800	5%	ISO 22007-2

2.2.1 Testing performed on epoxy-metal composites

Nikkeshi et al. [14] has taken Ni powder as filler and prepared composite at various volume fractions. They used laser flash method thermal conductivity measurement and calculated thermal conductivity 7 times higher than the pure epoxy at volume concentration of 24.5%.

Mamunya et al. [15] has investigated on copper and Nickel powder as filler into epoxy separately. Thermal conductivity testing carried out under steady state method with the ITEM-1M instrument.

Fu et al. [16] has prepared eight kinds thermal conductive adhesives by filling the epoxy resin (E-51) with conventional fillers such as natural graphite, Cu, Al, ZnO, BN, Al_2O_3, diamond, and Ag powders. Among these three are metallic filler. He measured the thermal conductivities of all samples by Hot Disk TPS-2500 thermal constants analyser, and demonstrated the variations and the enhancement of the thermal conductivities of these thermal conductive adhesives as the filling load of the fillers are increased. They measured the following results:

Filler	Filler concentration in wt.%	Thermal conductivity Wm^{-1}K^{-1}	k_c/k_m
Cu	68.25%	0.74	4.35
Al	69.69%	1.11	6.53
Ag	34.25% approx.	.35	2.06

Where k_c is the thermal composite and k_m is the thermal conductivity of the epoxy matrix.

2.2.2 Testing performed on epoxy-carbon material composite

There are many researchers who have paid attention on carbon materials as filler materials for thermal conductivity enhancement of epoxy adhesive. Carbon nano materials are widely used as fillers in epoxy composites because of their outstanding properties. The thermal conductivity testing performed on carbon materials are discussed in the table below:

Table-2: TC testing performed on epoxy-carbon material.

Filler	Method	Filler loading	TC(Wm^{-1}K^{-1})	k_c/k_m	References
Exfoliated graphite	Laser flash	20 wt.%	5.8	28	Ganguli et al. [15]
Graphite	Transient plane source	44.3 wt.%	1.68	9.88	Fu et al. [18]
Expanded Graphite	Laser flash	4.5 wt.%	1.0	4.4	Wang et al. [19]
Functionalized graphite	Transient plane source	30 wt.%	1.698	8	Gu et al. [20]
Reduced graphene oxide	Laser flash	20 wt.%	6.1	30.5	Sun et al. [21]
Graphene	Laser flash	30 wt.%	4.9	24.5	Tang et al. [22]
Diamond powder	Transient plane source	29.4 wt.%	.35	2.06	Fu et al. [18]
Single wall carbon nano tube (SWCNT)	Steady state method	1 wt.%	-	2.25	Mcnamara et al. [23]
Multi wall carbon nano tube (MWCNT)	Steady state method	0.5 wt.%	-	1.45	Mcnamara et al. [23]
Carbon fibre	Laser flash	60 wt.%	2.992	-	Mu et al. [24]

2.2.3 Testing performed on epoxy- ceramic filler composites

Ceramic fillers cover all the metal and non-metal compounds including oxides, carbides, nitrides etc. Ceramic fillers are used when there is requirement of thermal conductivity in addition with electrical insulation. Many researchers have done investigation on thermal conductivity enhancement of epoxy resin using various types of ceramic fillers. Some of their analyses are discussed here:

Table-3: TC testing performed on epoxy-ceramic filler composites

Filler	Method used	Filler loading	TC (Wm^{-1}K^{-1})	k_c/k_m	References
Al$_2$O$_3$	Transient plane source	67 wt.%	0.57	3.47	Fu et al. [18]
Al$_2$O$_3$	Laser flash	70 vol. %	13.46	-	Hu et al. [25]
ZrB$_2$	Laser flash	16 wt.%	0.355	2.31	Wu et al. [26]
Aluminium Nitride (AlN)	Laser flash	60 vol.%	11	-	Xu et al. [27]
Boron Nitride (BN)	Laser flash	57 vol.%	10.3	-	Xu et al. [27]
Hexagonal Boron Nitride (hBN)	Laser flash method	44 vol. %	9	-	Yu et al. [28]
Silicon Carbide(SiC)	Laser flash	14 vol.%	0.5 approx.	-	Zhou et al. [29]

All the results discussed above in table 2 and table 3 are at room temperature. Many of researchers discussed in this review paper have further investigated the dependence of thermal conductivity of epoxy composites with temperature and they found that the thermal conductivity of epoxy composites increases with temperature at same filler loading. Many of them have validated and compared their results with various thermal conductivity models or with the past results.

The test results of thermal conductivity of epoxy composites are wide and not so conclusive because many have used various preparation techniques for making composites. The techniques employed for preparation differ in mixing of epoxy and filler materials or in curing conditions.

Many of the researchers discussed above have done surface treatment of epoxy composites using different kinds of chemical agents like acetone, acids, silane etc. and have compared the results with untreated epoxy composites. They found that the thermal conductivity of treated composites is higher than the untreated one.

2.2.4 Testing performed on epoxy-hybrid composites

As mentioned earlier hybrid composites means combination of fillers of different types or of different shapes or of different sizes. Presently the hybrid fillers into epoxy have become area of interest for research. It is seen that the thermal conductivity of epoxy composites are higher than that of epoxy composites with conventional fillers. Some researches on epoxy composites with hybrid fillers are discussed here:

Ahn et al. [4] has prepared copper nanowire/epoxy composites for use as thermal interface materials. The surfaces of the copper nanowire particles were coated with TiO_2 after synthesizing copper nanowire with by reducing $Cu(NO_3)_2$ with hydrazine to enhance the mechanical and thermal properties in the polymer matrix. They used laser flash method for TC measurement and found that the thermal conductivity 1.1248 W/m K at 15 wt.% higher than the untreated composites at same filler loading.

Yuan et al. [6] have used AlN particles with different size, graphite, graphene oxide (GO) as filler into epoxy composite. When they loaded epoxy composites with 8 wt.% GO or 70 wt.% 5µm AlN particles, they got the thermal conductivities of adhesives about 6.42 times and 11.8 times that of pure epoxy respectively. To further improve the thermal conductivity, they proposed hybrid filler 50 wt.% 5µm AlN particles and 6 wt.% GO, the final thermal conductivity reached up to 2.77 W/m k, that was 14.6 times of that of neat epoxy.

Guan et al. [30] have prepared composite by using spherical alumina (Al_2O_3), magnesium hydroxide and graphene Nano platelets (GNPs) as thermally conductive fillers into epoxy adhesive. The epoxy composite with 68% Al_2O_3, 7% modified GNPs (m-GNPs) and 5% magnesium hydroxide is determined as the optimum composition with a high thermal conductivity of 2.2 W/(mK), 11 times of that of neat epoxy.

Chen et al. [31] have investigated on epoxy resin based thermal interfacial materials (TIMs) by filling Ag nanoparticle-decorated graphene Nano sheets (GNSs) as thermal conductive fillers. They measured thermal conductivity by thermal conductivity analyser based on the modified transient plane source principle and got enhancement up to 272.9% observed for Ag-GNS loading at 5.0 wt.% in epoxy resin as the quantity of Ag nanoparticles decorated on GNS is 1.5 mol%.

Thermal Gravimetric Analysis (TGA):

 Thermo-gravimetric analysis or thermal gravimetric analysis (TGA) is a method of thermal analysis in which changes in chemical and physical properties of materials are measured as a function of increasing temperature (with constant heating rate), or as a function of time (with constant temperature and/or constant mass loss). TGA can give information about chemical phenomena, such as desolvation (especially dehydration), decomposition and solid-gas reactions (e.g. oxidation or reduction). Similarly TGA can give information's about physical phenomena such as second order phase transition including vaporization, sublimation, absorption and desorption.

TGA can be used to evaluate the thermal stability of a material. It is generally used to investigate the thermal stability of polymers. In a desired temperature range, if material is thermally stable, there will be no observed mass change. Negligible mass loss corresponds to little or no slope in the TGA trace. TGA also gives the upper application temperature of a material. Beyond this temperature the material will begin to degrade.

Many researchers have done TGA analysis in epoxy composites. Some of them have discussed here:

Wang et al. [19] has investigated the thermal stability of pure epoxy and epoxy composites filled with 4.5 wt.% expanded graphite and with 4.5 wt.% (KH550@EG) using TA Q50 TGA system. The sample was heated to 600℃ at the rate 10℃/min under nitrogen atmosphere. The initial decomposition temperatures were found to be 318℃, 329℃ and 348℃ respectively. They concluded that surface treated samples have better compatibility than untreated samples, which consequently improves the thermal stability.

Gu et al. [20] has investigated the thermal gravimetric (TG) analyses of the samples of Graphene Nano Plates (GNP) and functionalized Graphene Nano Plates (f-GNP) were carried out at 10 C/min (argon atmosphere), over the whole range of temperature (40–800℃) by STA 449F3. They found that the heat-resisting index is 192℃ (pristine E-51), 195℃ (0.5 wt.% f-GNPs), 199℃ (10 wt.% f-GNPs), 200℃ (20 wt.% f-GNPs) and 203℃ (30 wt.% f-GNPs), respectively and concluded that the thermal stabilities of the f-GNPs/ E-51 Nano composites are gradually improved with the increasing addition of f-GNPs

 Xu et al. [27] has done investigation on TGA analysis of the composites filled with boron nitride and aluminium nitride particles with or without of surface treatment using a PERKIN ELMER TGA7 and measured the weight loss heating up to 600℃ at 20℃/min under nitrogen atmosphere. They found that weight loss of the samples treated with silane agents are higher than the samples which were treated with acetone and acids (nitric and sulphuric acid).

Yu et al. [28] has tested the amount of h-BN in the composites using thermo-gravimetric analyzer TG 209 F1 (NETZSCH, Germany) under air atmosphere with a heating rate of 10 °C/ min. The samples were heated up to 700°C and three significant drops were found from the weight loss curve. The first one, which appeared at around 145 °C-270 °C, attributed to the pyrolysis of the low molecular weight components. The second peak at 270 °C-420 °C should be ascribed to the decomposition of epoxy resin. The last obvious weight loss happened at 420 °C-620 °C due to the combustion of the residual carbon of epoxy resin in air atmosphere.

Liang et al. [32] have prepared Nano composite samples with 1 wt.% loading of either SiO_2 fillers or Al_2O_3 fillers and micro-composite samples with 20 wt.% loading of either SiO_2 fillers or Al_2O_3 fillers. Nano-micro (hybrid) composite samples (either use SiO_2 or Al_2O_3 fillers) were prepared by dispersing 1 wt% Nano fillers and 20 wt% micro fillers in the epoxy resin. The TGA analysis was performed on Perkin-Elmer TGA7 to obtain information on thermal stability. The TGA analysis was done under the environment of Nitrogen gas. During the test, the temperature in test chamber increased from 30°C to 800°C at a heating rate of 10°C/min. From 800°C to 850°C, atmosphere change from nitrogen to air and the heating rate was 10°C/min. They found that the weight loss rate of micro-composite and nano-micro composite is much lower compare to neat epoxy and the SiO_2 filled specimens displayed a very similar pattern to Al_2O_3 filled specimens.

3. CONCLUSIONS

This review paper is prepared on the thermal properties of the epoxy resin filled with different kinds of filler as a thermal interface material. Based on the reviewed research papers following conclusion can be drawn:

1. Epoxy resins have better properties over other thermal interface materials. So it is better to use as thermal interface material than other polymers.
2. The thermal properties of epoxy composites vary with the type of epoxy resin and curing agent and also with the curing conditions.
3. The thermal conductivity of an epoxy composite depends on filler loading. The thermal conductivity of epoxy composites increase with increase in filler loading into epoxy composite.
4. High filler loading into epoxy composite causes poor processablity, loss of mechanical integrity and inferior mechanical properties of epoxy composite. Also, High filler loading leads to high cost.
5. Size, shape and the orientation of the fillers into epoxy composites affect the thermal properties of epoxy composites.
6. The techniques employed for preparation of epoxy composites including mixing of epoxy and filler,

curing conditions etc. also affect the thermal properties of epoxy composites.

7. Hybrid fillers, such as mixture of fillers of different type, size, and shape can help to achieve higher TC at a lower filler loading level. They can also help to lower viscosity of epoxy composite, thus improving processability.

Filler surface treatment with different kinds of chemical agents can help improve filler dispersion, while also decreasing the overall viscosity. Even more, it decreases thermal interfacial resistance; therefore, higher TC can be obtained.

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