Effective Thermal Conductivity of Yttria stabilised Zirconia Filled Epoxy Composites

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Submitted by

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C E R T I F I C A T E

This is to certify that the work in this thesis entitled Effective Thermal Conductivity of yttria stabilised zirconia Filled Epoxy Composites by **Chandra Prakash** has been carried out under my supervision in partial fulfilment of the requirements for the degree of Bachelor of Technology in Mechanical Engineering during session 2014 - 2015 in the Department of Mechanical Engineering, National Institute of Technology, Rourkela.

To the best of my knowledge, this work has not been submitted to any other University/Institute for the award of any degree or diploma.

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CONTENT

List of Figures

List of Tables

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ABSTRACT

Particulate filled polymer composites have enhanced thermal, mechanical properties as compared to the neat epoxy polymer. This project exhibits an increment in the thermal conductivity due to the addition of particulate filler. A mathematical expression for the spherical and also cubic fillers, distributed uniformly in the epoxy matrix is developed to evaluate theoretical thermal conductivity of the polymer composites. Yttria stabilised zirconia is used as a filler material in various proportions. The resultant composite is of enhanced thermal conductivity in comparison with pure epoxy matrix. Unitherm ™ Model 2022 tester is used to experimentally determine the thermal conductivity of the composite fabricated. Hand lay-up technique is used to fabricate yttria stabilised zirconia-epoxy composite. Experimental and theoretical results are compared and the reason behind this difference is also discussed. Currently, various applications of particulate filled composites are also studied. Future scope and various other influencing properties of the composites were discussed in short. It is discovered that particulate filled polymer composites can be used in micro-electronic circuit.

Nomenclature

K_{eff} = Effective thermal conductivity of the composite.

K_m = Thermal conductivity of matrix material.

K_f = Thermal conductivity of filler material.

ϕ = Volume fraction of filler material.

R_T = Total thermal resistance of cube.

R_1, R_2, R_3, R_4, R_5, R_6 = Resistance of layer 1, 2, 3, 4, 5 and 6 respectively.

H = side length of the cube.

a = side length of cubical filler.

PMC = Polymer matrix composite.

CMC = Ceramic matrix composite.

Chapter-1

Introduction

1.1-Introduction:

The modern era of composites began with the development of plastics. Till that time, natural resins obtained through animals and plants were the sole means of getting binders and glues. By the beginning of 20th century, plastics like polystyrene, vinyl, polyester and phenolic came into existence. The newly developed synthetic materials turned out to be more advanced than the resins obtained through the natural process. Though, only plastics could not generate more rigidity needed in the structural application. Reinforcements were required for generation of the rigidity and strength. Fiber glass, after combining with the polymers of plastic provides an amazing structure which has strength as well as is light in nature. Thus, the FRP industry came into limelight.

A large portion of the best developments in this field was the outcome of the war. Generally as the Mongolians started using the materials made from composites, it brought the FRP business into the real world. Now, it was acknowledged that composites had different advantages other than being light in nature and rigid.

Composites came with full force in the market when the world war 2 was about to end. As there was little scope in military items, composites pioneers aggressively attempted in bringing composites uses to the different scenario. Later on, composites were used in business vessel structure and other boats. With the time, composites were used in car panels, aircraft wings and microelectronic circuits.

1.2-Composites Continuous Advancement:

During the early part of 1970, the composites business started to develop. With the advancement in technology good plastic resins and the reinforcing fiber was produced.

Kevlar- an aramid fiber which was being introduced by Dupont, was extensively used in the defensive establishments because of kevlar’s high tendency to persist. Carbon fiber’s were additionally grown during this period; which resulted into the subsequent replacement of material as it turned out to be more advantageous.

The evolvement of the composites is continuous and nowadays it is mainly focussed around the clean energy sector. For example, in the wind energy sector, turbine blades are nowadays fabricated of the composite material and it is requisite for the blades to be small and made up of highly efficient material, effective design and precisely manufactured. Composites have enhanced the efficiency of the wind energy sector. Solar panels are also being fabricated of the composite material. With the time, nano materials will be inducted into the composites as it would definitely enhance the strength, rigidity, the thermal conductivity of the composites. Nano materials have been found extensive useful in the all the applications and integration of it into the composites would definitely present a major change in the world of material science. There are various college products going around the world, which works on the development of the composites and fabricate them to generate more enhanced material. Also, nowadays composites are being fabricated keeping in mind of the environmental concerns. Composites have turned out to be a boon for the world to make it a wonderful place to spend.

1.3-Definition:

A composite is made up of two or more materials with different physical and chemical properties which when combined produces an altogether different material which has different physical and chemical characteristics in comparison to the individual components. This definition holds good for all the composites. But, there is a recent definition which describes “composites” as reinforced plastics.

1.4 -Various types of composites materials:

- Matrices:
    - Organic Matrix Composites (OMCs)
        - Polymer Matrix Composites (PMCs)
        - Carbon-Carbon Composites
    - Metal Matrix Composites (MMCs)
    - Ceramic Matrix Composites (CMCs)

- Reinforcements
    - Fibres reinforced composites
    - Laminar composites
    - Particulate composites

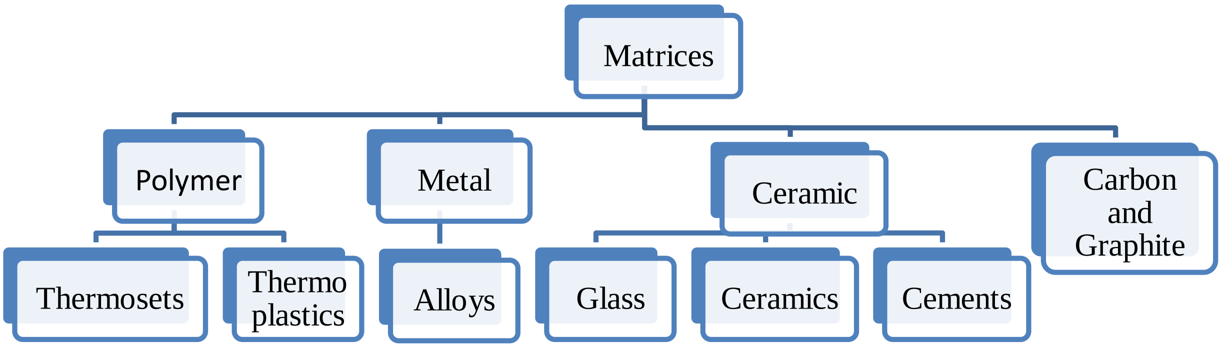

Fig. 1.1- Classification of the composites

1.5-Advantages:

- They have low density
- They have higher directional mechanical strength
- They have higher toughness than ceramics and glasses
- They have high Fatigue endurance
- They have higher toughness as compared to ceramics and glasses.
- They are versatile.
- Machining is easy.
- They can combine with other properties.

Organic Matrix Composites

- Polymer Matrix Composites

Polymer matrix composites (PMCs) are those in which the matrix material is a polymer, bounded together by continuous fibers. It has high strength to weight ratio, which is the main reason behind its use in aerospace industries. Reinforcement in a PMC provides high strength and stiffness to the material. The PMC is planned so that the mechanical load to which the structure is imposed in service is supported by the reinforcement.

Metal Matrix Composites:

In Metal Matrix composites are those in which the matrix is metal and filler materials can be ceramic, metals etc. It is more advantageous than the monolithic metals. It has excellent properties, for example, high specific strength, and low coefficient of thermal expansion. Because of its effective thermal properties, it can be widely used in cable manufacturing, housing etc.

Ceramic Composites:

Ceramic composites are those in which the matrix material is made up of ceramics and filler materials can be ceramics, glass, metals. Mechanical properties of the ceramic composites are very efficient. Their strength, rigidity, hardness and crack resistance is very high.

Particulate composites:

Particulate acts as a reinforcement, which enhances thermal, mechanical properties of the composites. Particulates are used to decrease the rigidity of the matrix and increase the modulus of the matrix. Current investigation of the project is on the particulate filled polymer composites.

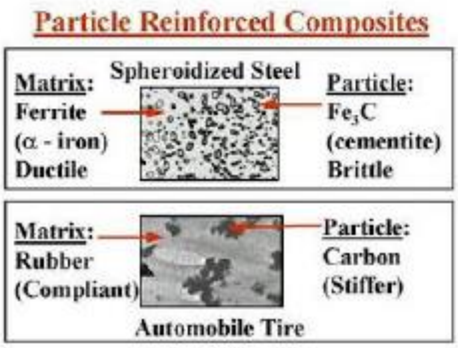

Fig. 1.2 - Particle reinforced polymer composites

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Chapter-2

Literature Review

Literature Review:

The present chapter discusses the literature studied during the course of research. The basic motto behind the literature review is to give a short idea and background data on the various issues dealt in the present investigation. This chapter elaborately discusses the particulate filled polymer matrix composites and its thermal characteristics. Following three points are discussed in this chapter:

- Particulate filled polymer matrix composites
- Thermal conductivity of particulate filled polymers
- Thermal Conductivity Models

2.1-Particulate Filled Polymer Matrix Composites:

The PMCs comprise of the polymers added with the filler materials in different concentrations to enhance the thermal properties and various other characteristics. It is also effective in reducing the cost, enhances the electrical, optical, magnetic properties of the composite materials. Mechanical properties such as hardness, rigidity and resistance to wear are also improved significantly. To enhance the wear resistance significantly, metal or ceramic particulate fillers and fiber fillers are extensively used [1]. Particulate fillers, for example, metal particles or ceramics are utilised to enhance mechanical properties of the material, for example, wear resistance [2]. These are used in electronics departments such as in making chips, thermal electrodes. This is due to their thermal resistance at very high temperature [3]. Now a day it is used in the printed circuit boards and other micro-electronic circuits. It is used for effective heat dissipation of the generated amount of heat. Our current investigation is also on the ways to enhance the thermal conductivity so as for effective heat dissipation. Particulate filled composites comprising of the ceramics fillers have been a subject of extensive investigation for the past 20 years. Ceramic or inorganic fillers are used basically for the cost reduction and improvement in the stiffness [4] [5]. The shape, size, other dimensions, filler particles' volume fraction enhances the mechanical, thermal and magnetic properties of the material [6]. Yamamoto et al [7] tells about the dimensions and topology of the particles, which have a noteworthy impact on mechanical properties, for example, fatigue resistance, malleability and fracture resistance. Fiber reinforced composites and particulate filled composites have been found useful in numerous operational requirements. Bonner [8] reported that with the incorporation of smaller scale measured particulates into polymers, high filler content (commonly more than 20% vol) is by and large needed to bring the above expressed beneficial outcomes into play.

Polymer matrix composites comprising of silica particles play a very significant role in enhancing the electrical, mechanical and thermal properties of the resultant composite [9] [10]. It was studied by Yamamoto et al. [11] that structure of silica particles is such that it greatly enhances the mechanical properties. State of the silica particles also plays an important role in doing so. Moloney et al. [12–14] and Adachi et al. [15] reported from their investigation that filler material's volume fraction also impacts the mechanical properties of the polymer composites.

2.2-Thermal Conductivity of Polymer Matrix Composites:

A couple of hypothetical and exact models to determine composites' effective thermal conductivity have already been proposed. Maxwell model and Rule of mixture [16] models are used to compare the thermal conduction inside the polymer composites, which demonstrates that irregular scattering of the small spheres with low filler concentration inside the composites play a significant part in determining the effective thermal conductivity. Bruggeman [17] found a mathematical expression of thermal conductivity in relation to the strong stacking of spherical shaped fillers in the polymer matrix. Lewis and Nielsen [18] obtained a hypothetical model which is a modified form two-phase system equation, which were earlier proposed by Halpin-Tsai equation. Recently, in a study Griesinger et al. [19] has found out that if we keep orientation ratio of polyethylene at 50, thermal conductivity increments from 0.35 to 50 W/M-K. The studies have been done to know about the thermal conductivity of the polymers instead of their molecular arrangement. Including the thermally conductive filler materials or fibers to enhance the thermal conductivities of the polymer composites are the most efficient way. A lot of studies have been done on the impact of inclusion of thermally conductive fillers on the thermal conductivity of the material. Most of them incorporate experimental results of effective thermal conductivity of filler reinforced polymer composites [20-24].

2.3-Thermal Conductivity Models:

For a two-part composite, according to heat flow, the mathematical expression for thermal conductivity for materials organized in series or in parallel.

For parallel conduction model:

$$k_{eff} = (1 - \phi_f)k_p + \phi_f k_f \tag{1}$$

For series conduction model:

$$1/ k_{eff} = (1 - \phi_f)/k_p + \phi_f / k_f \tag{2}$$

The nature of the expression displayed by Equation (1) and (2) is found out on the basis of the Rules of the mixture. For arbitrarily distributed homogeneous spheres in a medium, which are non-interacting, Maxwell got an accurate result for the thermal conductivity.

$$\frac{k_{eff}}{K_p} = \frac{k_f + 2k_P + 2\phi_f(k_f - k_p)}{k_f + 2k_P - \phi(k_f - k_p)} \tag{3}$$

Using this model, thermal conductivities of the polymer composites with the lower filler concentrations are found out very well. However, when there is an increment in the filler concentrations, molecules begin to interact with each other and there is a formation of the chain towards the direction of heat flow, which is the basic reason behind the decrement of importance of this model.

Agari and Uno [25] determined mathematical expression which considers both parallel and series conduction mechanism. He gave the following expression to determine the effective thermal conductivity.

$$\log K_c = \phi C_2 \log K_f + (1 - \phi)\log(C_1 k_m) \tag{4}$$

C_1,C_2 are experimentally determined constants.

2.4-Aim of the present work:

(1) To develop a mathematical model to evaluate effective thermal conductivity of a class of particulate filled polymer composite.

(2) To fabricate the boron nitride filled epoxy composite with two different concentrations by hand layout technique.

(3) To experimentally find out the value of effective thermal conductivity of these composites.

(4) To study the effect of incorporation of micro-sized boron nitride on the heat conductivity of epoxy.

(5) To validate the theoretical model by comparing the results with measured values.

(6) To identify the potential applications of these composite in microelectronics.

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Chapter-3

Materials and Methods

Materials and Methods:

Current section depicts materials and methods used in fabrication and characterizing of the composites, which is being studied. Present chapter displays all the tests, which is associated with the physical, mechanical, micro-structural and thermal characterization of the epoxy and polypropylene composites arranged for this examination.

3.1-MATERIALS:

Matrix Material

Epoxy LY 556, which is a nomenclature of Bisphenol-A-Diglycidyl-Eather, generally comes under the so-called "epoxide" family has been used as a matrix material. Epoxy resin comes in the group of thermoset along with the silicones and polyesters. Matrix is Epoxy, which is chosen on the fact that it has low density (1.1 gm/cc) and low thermal conductivity (0.63 w/mk) and it is one of the extensively used polymers. It has excellent electrical insulating capabilities, which is the main reason behind its use in microelectronics circuits. Polymer are most usually used as matrix material due to its cost efficient nature, simplicity of creating complex shapes with lesser expense of tools and additionally they have effective properties at room temperature after contrasting with matrices of metals and ceramics. Polymer matrices could either be thermoplastic or thermoset in nature [26].

Thermoplastic polymers are joined through chain because of inter-molecular forces and these forces allow thermoplastics to be remoulded in light of the fact that the inter-molecular interactions increments after cooling and restore the bulk properties. These arrangements of polymers are ordinarily created in one stage and afterward are made into products in a further process.

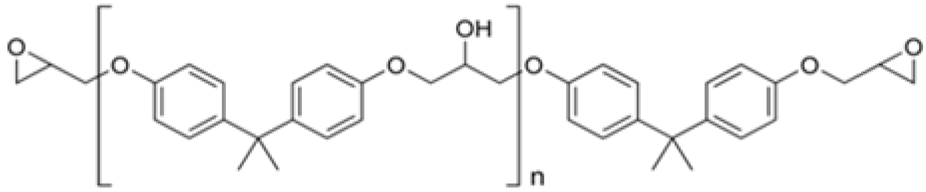

Fig. 3.1 Unmodified epoxy resin chain ('n' denotes number of polymerized unit)

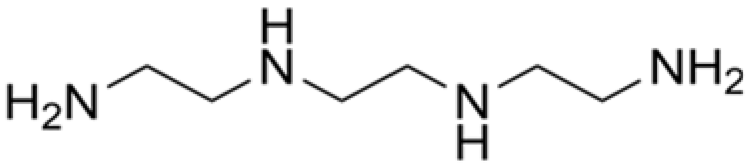

Fig. 3.2 Tri-ethylene-tetramine (hardener used for epoxy matrix)

Table 3.1: Properties of epoxy

Characteristic Property	Inference
Density	1.1 gm/cc
Compressive strength	90 Mpa
Tensile strength	58 Mpa
Micro-hardness	0.085 Gpa
Thermal conductivity	0.363 W/mK
Glass transition temperature	98^0C
Coefficient of thermal expansion	62.83 ppm/ ^{0}C
Electrical conductivity	0.105 X 10^{16} S/cm

3.2-Filler Material:

Zirconia, which is an abbreviation of zirconium dioxide, is stabilised at room temperature by the addition of yttrium oxide. Some of the zirconia ions are replaced by the yttria ions in the lattice of zirconia. This leads to vacancies in the lattice, hence there is conduction of electricity. This property enhances with the temperature.

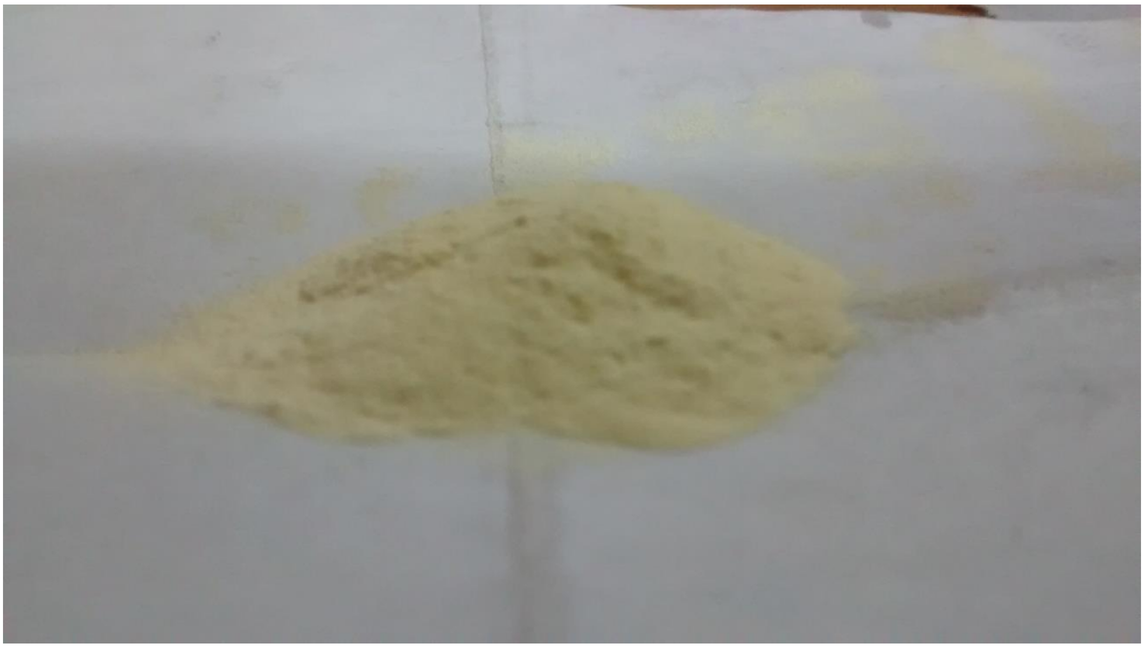

Fig. 3.3- Yttria stabilised zirconia powder

Applications:

- It is chemically inert and hard.
- It is used as a refractory.
- It is used as a gas turbine as thermal barrier coating.
- It is used as electro ceramic.
- It is used in the production of solid oxide fuel cells.
- It can be used in printed circuit board to enhance the heat dissipation.

3.3-Analytical Modelling:

An analytical model is being proposed to evaluate the effective thermal conductivity of epoxy-yttria stabilised zirconia composite theoretically. A filler of cubic shape was assumed to be uniformly distributed in the matrix. Since, FCC arrangement is the most efficient one, hence the arrangement is assumed to be FCC packed. Let the side length of the matrix cube is 'H' and the side length of the filler cube is 'a'. Following expression was used to determine the volume fraction of the filler in matrix:

$$\phi = \frac{4a^3}{H^3} \quad (1)$$

If we put H=2a, we get ϕ=0.5. We have considered following three cases:

i) ϕ = 0 (pure matrix material)

ii) ϕ = 0.1 (less than 0.5)

iii) ϕ= 0.5 (Exactly filled)

Cube is divided into similar layers in each of the above cases and thermal resistance is assumed along the direction of heat flow. Total resistance of the cube is given by:

$$R_T = \sum_{i=1}^{n} R_i \quad (2)$$

Note – We have ignored contact resistance to make the calculation simpler.

Case-i: (ϕ =0)

In this case, overall resistance will be that of the resistance of the matrix.

Case-ii: ϕ = 0.1 (less than 0.5)

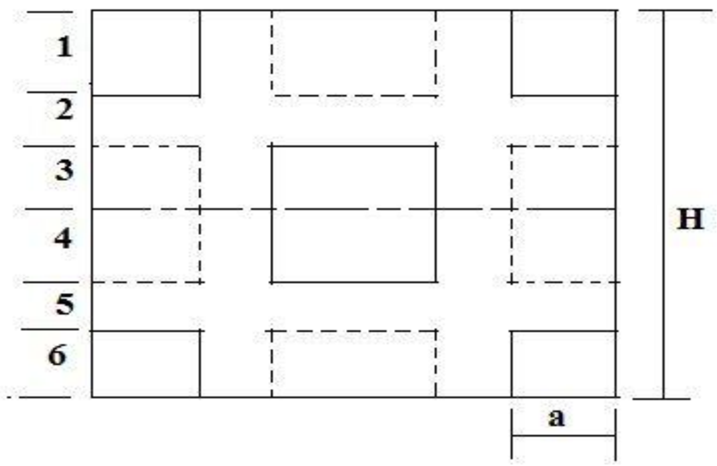

Fig. 3.4- Front View of cubic filler in Composite for ϕ = 0.1

$$R_2 = R_5 = \frac{H-2a}{k_m H^2} \tag{3}$$

$$R_1 = R_2 = R_4 = R_6 = \frac{a}{4(k_m - k_f)a^2 + K_m H^2} \tag{4}$$

$$R_T = \frac{a}{4(k_m - k_f)a^2 + K_m H^2} + \frac{(H-2a)}{k_m H^2} \tag{5}$$

$$k_{eff} = \frac{H}{R_T H^2} = \frac{1}{R_T H} \tag{6}$$

<u>Case-(iii)</u>: ϕ = 0.5

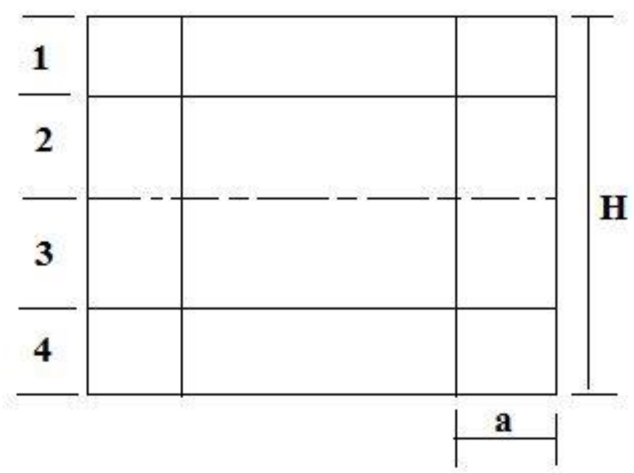

Fig. 3.5- Front view of cubic filler filled composite for ϕ = 0.5

$$R_1 = R_2 = R_3 = R_4 = \frac{a}{4(k_m - k_f)a^2 + K_m H^2} \tag{7}$$

$$R_T = \frac{4a}{4(k_m - k_f)a^2 + K_m H^2} \tag{8}$$

$$k_{eff} = \frac{H}{R_T H^2} = \frac{1}{R_T H} \tag{9}$$

3.4-Experimental Procedure:

Various samples of composites' are being manufactured through hand lay-up method. This technique is termed as the most simple for composites fabrication. The epoxy-yttria stabilised zirconia composites are prepared by following steps:

- At first the spray is sprinkled in the container to be used.
- Epoxy of requisite quantity is weighed on the weighing machine.
- Epoxy is then put inside the container to be used for mixing.
- Yttria stabilised zirconia is weighed on the weighing machine according to volume fraction and weight fraction determined theoretically.
- Then, the filler material is poured into the container in which epoxy is present.
- Filler and epoxy are mixed thoroughly and few drops of hardener are also added in the mixture.
- Mixture is kept still for a complete day
- Composite is fabricated and then effective thermal conductivity is calculated experimentally.
- The comparison is done between the theoretical and experimental results.

Fig. 3.6- Fabricated Composite

3.5-Experimental Determination:

Unitherm ™ Model 2022 tester is used to determine the thermal conductivity of different materials such as polymers, composites, ceramics etc. Determination of experimental thermal conductivity is done according to the ASTM E-1530 standard.

Fig. 3.7 - Unitherm ™ Model 2022

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Chapter-4

Results and Conclusions:

4.1-Results:

This chapter compares between the experimental and theoretical thermal conductivity of the epoxy-yttria stabilised zirconia composites. Bar chart (drawn below) shows the difference between the experimental and theoretical values at a different volume fraction of the filler materials. Further, causes of difference in values are also discussed in this chapter.

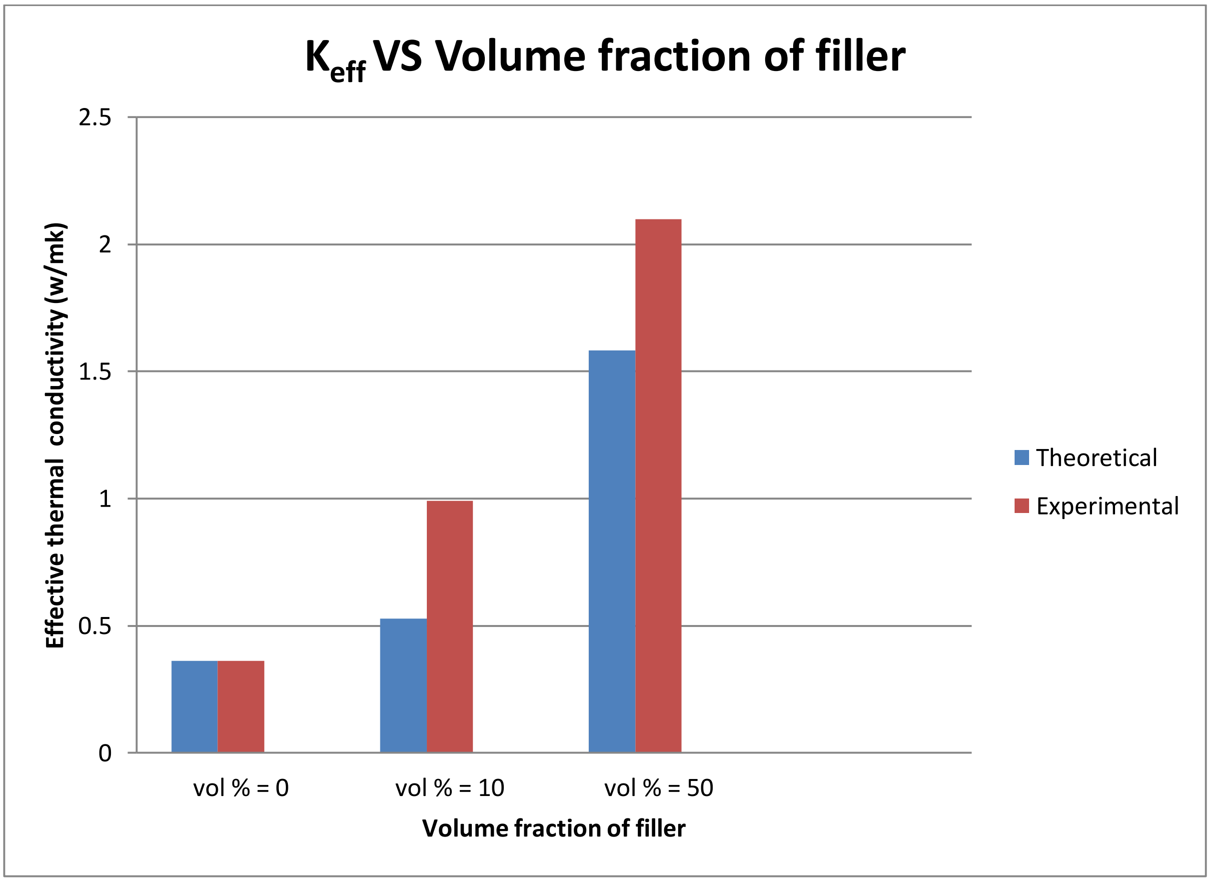

Fig. 4.1- Variation of thermal conductivity with filler content

4.2 - Inferences from the above graph:

- Effective thermal conductivity at 0% of filler content would be that of the thermal conductivity of the epoxy material.
- For volume fraction of 10%, there is an increment of 45.45 percent in the experimentally determined effective thermal conductivity from the pure epoxy material. However, there is a slight difference between theoretical value and experimental value.
- For volume fraction of 50%, there is an increment of 335.67 percent of experimentally determined effective thermal conductivity from a pure epoxy composite.
- There is a difference in the experimentally determined value and theoretically determined value.

4.3-Conclusions:

i) As the effective thermal conductivity of the composite is increased significantly by the addition of filler materials, it can be used in electronics packaging, which requires high heat dissipation.

ii) Composites can also be used in manufacturing of printed circuit boards. As PCB is denser and it requires a highly efficient heat dissipation mechanism, composites are best suitable for this purpose.

iii) Composites can also be used in glob top encapsulation, which is a covering type on the PCB. It prevents from contamination and also provides mechanical strength to printed circuit boards.

Scope for future Work:

In the current investigation, only the impact of volume fraction on the effective thermal conductivity is studied. However, the thermal conductivity can also be influenced by varying the shape, size and filler materials. Nano-particles have brought a revolution in the present scenario. Using nano particles as filler material can effectively change the thermal conductivity.

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