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The Effect of Aluminum and Copper Powder on the Physical properties of the Epoxy Composite

A Thesis submitted
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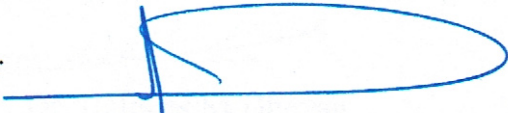
بِسْمِ اللهِ الرَّحْمنِ الرَّحِيمِ

﴿قُل لَّوْ كَانَ الْبَحْرُ مِدَاداً لِّكَلِمَاتِ رَبِّي لَنَفِدَ الْبَحْرُ قَبْلَ أَن تَنفَدَ كَلِمَاتُ رَبِّي وَلَوْ جِئْنَا بِمِثْلِهِ مَدَداً﴾

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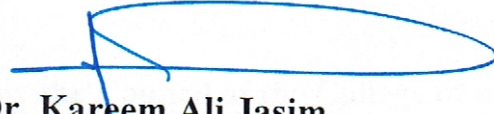
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We certify that we have read this thesis, entitled "*The Effect of Aluminum and Copper powder on the Physical properties of the Epoxy composite*" and as examination committee, we examined the student "*Rihab Nassr Fadhil*" on its contents, and that in our opinion it is adequate for the partial fulfillment of requirements for the Degree of Master of Science in Physics.

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Dedication

To my life light (mother & father) with my love & respect

To my beloved husband (Ayad) for his encouragement & support

To whom have filled me with their kindness and blessing my (Sister& Brother)

To my daughter (Hadeer)

To my sons (Abdullah and Ahmed)

Acknowledgments

Abstract

Epoxy resin with various mix ratio of concentration of Al and Cu powders, was used to prepare epoxy composites, in order to study the physical properties (flexural strength, modulus of elasticity, maximum fracture force, maximum deflection and Yield point and Hardness, thermal conductivity and Glass transition temperature, Conductivity $\sigma_{a.c}$ (w), dielectric constant, dissipation factor and Capacitance).

The results were obtained to form Three point bending test illustrated that the modulus of elasticity of EP/Al and EP/Cu composites were increased with increasing concentration of metal powders. Flexural strength, maximum Fracture Force and Deflection decreases with increasing of the Al percentages compared with pure Epoxy, except the ratio (15%). While Flexural strength and maximum Fracture Force decreases with increasing of the Cu percentages compared with pure Epoxy, except the ratio (25%) and (35%). Deflection decreases with increasing of the Cu percentages compared with pure Epoxy. Yield Points for all Al and Cu percentage increased with increasing ratio fillers. Increase hardness with Al and Cu concentration increase.

Thermal conductivity for EP/Al and EP/Cu composites was increased with increasing weight percentage of filler. The Glass transition temperature (T_g) of EP/Al composites were increased with increasing concentration of Al powder. While the maximum value (T_g) of EP/Cu composites at (35% Cu). Information's of measurements for mechanical and thermal properties could be beneficial in applications automotive

Electrical properties for EP/Al and EP/Cu composites, conductivity $\sigma_{a.c}$ (w) increases with increasing frequency, conductivity increases with increasing Al and Cu powder concentration. The $\acute{\epsilon}$, $\grave{\epsilon}$, tanδ and Capacitance decreases with increasing frequency. Information's of measurements for electrical properties could be beneficial as Capacitors to energy storage in the laser systems.

Contents

List of Tables

List of Figures

List of abbreviations and Latin symbols

Abbreviation/ Symbol	Meaning
E	modulus of elasticity (Young modulus)
F	Force
A	Area
σf	flexural stress
ϵ_f	flexural strain
M	bending moment
Mm	moment of resistance
I	span of specimen
w	width of the specimen
S	slope of the straight-line portion of force -deflection curve
D	deflection
K_{th}	thermal conductivity coefficient
d	thickness
r	radius of disc
DSC	Differential scanning calorimetry
Tg	glass transition temperature
ω	angular frequency
ε_o	permittivity of free space
έ	dielectric constant
C	capacitance
ɛ̆	dielectric loss
P	polarization
Ẽ	electric field strength
α	total polarizability

αe	the Electric polarization
αi	The ionic polarization
αd	The dipolar polarization
αs	The Space charge polarization
Q	charge
V	potential difference
σ	conductivity
R	resistance
Ep	Epoxy
Cu	Copper
Al	Aluminum
ASTM	American Society for Testing and Materials
SEM	scanning electron microscope
AFM	Atomic force microscope
бy	Yield Point
бf	flexural strength
f	frequency

CHAPTER ONE

Introduction

&

Literature Survey
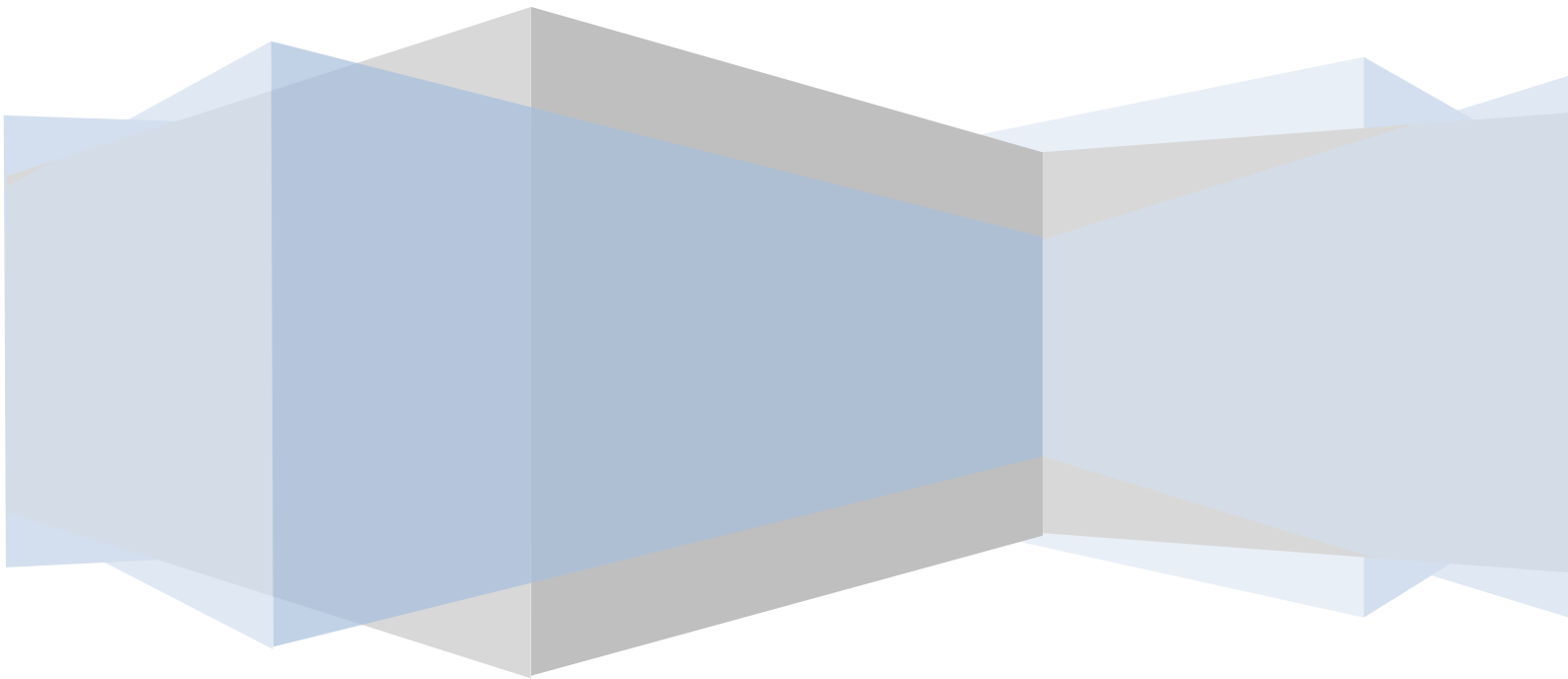
CHAPTER ONE

Introduction

1-1 Introduction

Modern technologies require many of materials with unusual combinations of properties [1], Polymers play a very important role in numerous fields of everyday life due to their advantages over conventional materials [2-3], engineers and designers today easily turn to polymers since they possess a number of features which are not available in any other materials. Polymers characterizes by resistance to corrosion, resilience, color fastness, transparency, ease of processing, lightness, etc [4-5].

The development of polymers science has started to increase. Scientists seek to produce, inexpensive, flexible and multi-use polymers. They are used in housing, electrical equipment, automobiles and various industrial applications. [6-7].

Polymers have replaced metals in many applications in being chemically inert and erosion resistant. Most polymers are presently manufactured as lightweight, rigid and foam materials and are used as insulators due to low thermal and electrical conductivity. Polymers have almost no free electrons but recent scientific and technical break-throughs have succeeded in production some modifications on regular polymers and brought into existence a new generation of polymers that combine the electrical features of the conductive and semi-conductive materials and the mechanical and chemical for polymer. [7-8].

In 1936, Dr. P Castan succeeded in synthesizing resin (Epoxy), at its hardened with phthalic acid anhydride. While developed these resins (epichlorohydrin and bisphenol) to coatings resistant to alkali in 1939.

Also we noted developed epoxy synthesis from 1960 to and to the present where it used in the automotive industry, aerospace and electronics etc. [9].

1-2 Literature Survey

In 1999, (Agari) et al [10], have studied thermal conductivity of a polymer (PS and PS composite), filled with particles (quartz or Al_2O_3) of wide range of volume concentration. They are obtained results show increasing in thermal conductivity within a wide range (from low to super-high) of filler in order to obtain materials with desired thermal, mechanical, electrical and physical properties, polymers [mixed with different kinds of fillers (fibers or particles)] are used as matrix material. The thermal conductivity of composite materials, which are represent as a multiphase substance, depends upon thermal conductivity, proportion, and the distribution of the phase.

In 2008, (Z M Elimat) et al [11], have studied the a.c electrical properties of (Al–Ep) composites as a function of the composition, frequency and temperature.

In 2010, (V. K. Srivastava) and (Ansul Verma) [12], have studied the Mechanical Behavior for (Cu) and (Al) Particles Reinforced Epoxy Resin Composites, where it was added to the epoxy resin (1%), (5%), (8%) and (10%) wt%, (Cu and Al) filler. It was noted the Mechanical properties increased with an increase of Cu and Al wt% for fillers.

In 2010, (Asmaa Shawky) et al [13]**,** have studied The Effect of (Cu, Al and Fe) additive on K_{th} of Epoxy Resin**.** The result of K_{th}, for Ep/metal composites show that, K_{th} increase with increasing ratio of wt%, For EP/ Al and EP/Cu composites. The results show that the best values for K_{th} is for EP/Cu composite compare with EP/ Al and EP/ Fe.

In 2011, (H. I. Jafar) et al [14], have studied the a.c Electrical Properties of Ep/Al Composites have been measured at the range (10^2 -10^6) Hz at RT. It was found that s(w) increases with increasing frequency according to the

relation $s(w)=Aw^s$ has been observed , with values of exponent (s) less than unity for all spacemen's and decrease with the increase of frequency and lie between (0.3 to 0.6) unit.

In 2012, (Jianing Gao) et al [15], have studied The Mechanical Properties of Epoxy Composites Filled with Rubbery Copolymer Grafted (SiO_2), noted improvement of mechanical properties for systems.

In 2014, (Yuan-Xiang Fu) et al [16], have studied the K_{th} enhancement with different fillers for epoxy resin adhesives**,** the K_{th} of all the spacemen's were measured by Hot Disk (TPS-2500) thermal constants analyzer, the highest K_{th} up to (1.68 (W m^{-1} K^{-1})) at (44.3) wt% of the eight thermal adhesives.

In 2014, (Ahmed Jadah Farhan) and (Naajla jerjak) [17], has studied the Preparation and Study of Some Physical Properties for Polyester Reinforcement by (Al) Powder Composites. In this study (UPE) mix with ratio (10, 20, 30 and 40) wt % of aluminum chip. The results show increased K_{th} of the (UPE) spacemen's by increased thermal conductivity coefficient value after addition this material and of K_{th} increased with increasing of additive wt % of Al chip. Dielectrically properties of (UPE) and (UPE/Al) composites show; the dielectrically properties of UPE/Al spacemen's decrease with increasing of the wt % of Al chip.

In 2015, (Harith I Jaffer) et al [18], have studied the Electrical Insulation of Polymeric Composite, Electrical insulators in this research ware made from reinforced epoxy with three kinds of fiberglass (wool, low surface density and high surface density). Reinforced epoxy with wool fiberglass has highest ρ= (28.4*10+1Ω cm) and increasing in insulation voltage for reinforced epoxy with high surface density fiberglass (30 kv).

In 2015, (Anshul Verma) and (V.K. Srivastava) [19], have studied the Mechanical behavior of metal particles reinforced polymer matrix composites,

Cu and Al as filler particles, having size less than (100μm). Mechanical behavior for composites increased with an increase in filler content.

In 2015, (Sihama Issa Salih) et al [20], have studied the Evaluation Mechanical Properties of Polymer Composites Reinforced by different Metal Powders, This research covers reinforcement of unsaturated polyester resin by metal powders Copper (15.598 μm) and aluminum (21.533μm) at selected volume fraction of (0%), (5%), (10%), (15%), (20%) and (25 %). The study of mechanical properties, the results show a noticeable increase in values of flexural strength, flexural modulus and fracture toughness reach to maximum value at (10%) volume ratio for both types composites, however elongation at break shows decrement as the volume fraction of metal powder is increased for both types composites.

In 2015, (Hind W. Abdullah) et al [21], have studied the study of Bending Property for Epoxy / Kevlar - Glass Fibers and Hybrid Composite, In this research a study of the effect of quality, sequential and directional layers for three types of fibers. The results shown that for Bending test the sample which reinforced (Kevlar - regular glass-Kevlar) characterized the highest bending (8mm) and The less bending reinforced sample was (regular glass-Kevlar- regular glass) (4.5mm).

In 2015, (Waleed A. Hussain) et al [22], have studied the Dielectric Properties and a.c Conductivity of Ep/Alumina Silicate NGK Composites, Al_2SiO_5 powder which is extracted from the obsolete spark plug NGK has been used to produce Ep/Al_2SiO_5 composite. The dielectric behavior of the composite substance is analyze as a function of the filler content, temperature and frequency. A.C conductivity and impedance are also studied. The results show that the permittivity, $\acute{\epsilon}$ and tanδ for all composites increase with increasing filler content.

In 2016, (Baraa H.) et al [23], has studied the effect of utilization Si and Cu powders on the mechanical and physical properties for epoxy resin.

Improvement the physical and mechanical properties for epoxy resin by fillers. The thermal conductivity increased with increasing wt% for Si and Cu fillers content be for Si (24.65 w/m.c^{-1}) and the Cu (25 w/m.c^{-1}). As well as the electrical conductivity increased with increasing wt% for Si (0.87 s/m) and Cu (0.967 s/m). The impact resistance increases for epoxy resin to add silicon particles and Cu but be better resistance at the rate of 8% Si, where up to (32.5 J/m^2) while the addition 8% Cu arrived to (14J/m^2).

In 2016, (Safaa. Abed. S) and (Widad. Hamdi.J) [24], have studied the Comparing the Behaviors of ZrO$_2$ and MgO Nanoparticles in Mechanical Properties Improvement of epoxy composite, Epoxy with various mix ratio of concentration (1%), (2%), (3%), (4%), (5%), (7%), (10%), (15%) and (20%) of ZrO$_2$, MgO nanoparticles and ZrO$_2$, MgO microparticles were used to prepare epoxy microcomposites and epoxy nanocomposites in order to study the properties, mechanical ZrO$_2$ nanoparticles enhance all mechanical properties of epoxy matrix with most vol. fraction of addition , The best value of addition of ZrO$_2$ nanoparticles which is used to improvement of mechanical properties like (flexural strength, fracture toughness and impact strength) at (2%) and (flexural modulus ,hardness) at (3% vol.) fraction. MgO nanoparticles enhance all mechanical properties of epoxy matrix with most vol. fraction of addition. The best value of addition of MgO nanoparticles is (4%) which is used to improvement of mechanical properties like (flexural strength, flexural modulus, fracture toughness impact strength and hardness).

1-3 The objective of This study

The objective of this study to Improvement the physical properties represented by mechanical, thermal and electrical behavior of epoxy reinforced by Aluminum and Copper powders. Information's of measurements for mechanical and thermal properties could be beneficial in applications automotive. As for information's of measurements for electrical properties could be beneficial as Capacitors to energy storage in the laser systems.

CHAPTER TWO

Theoretical Background
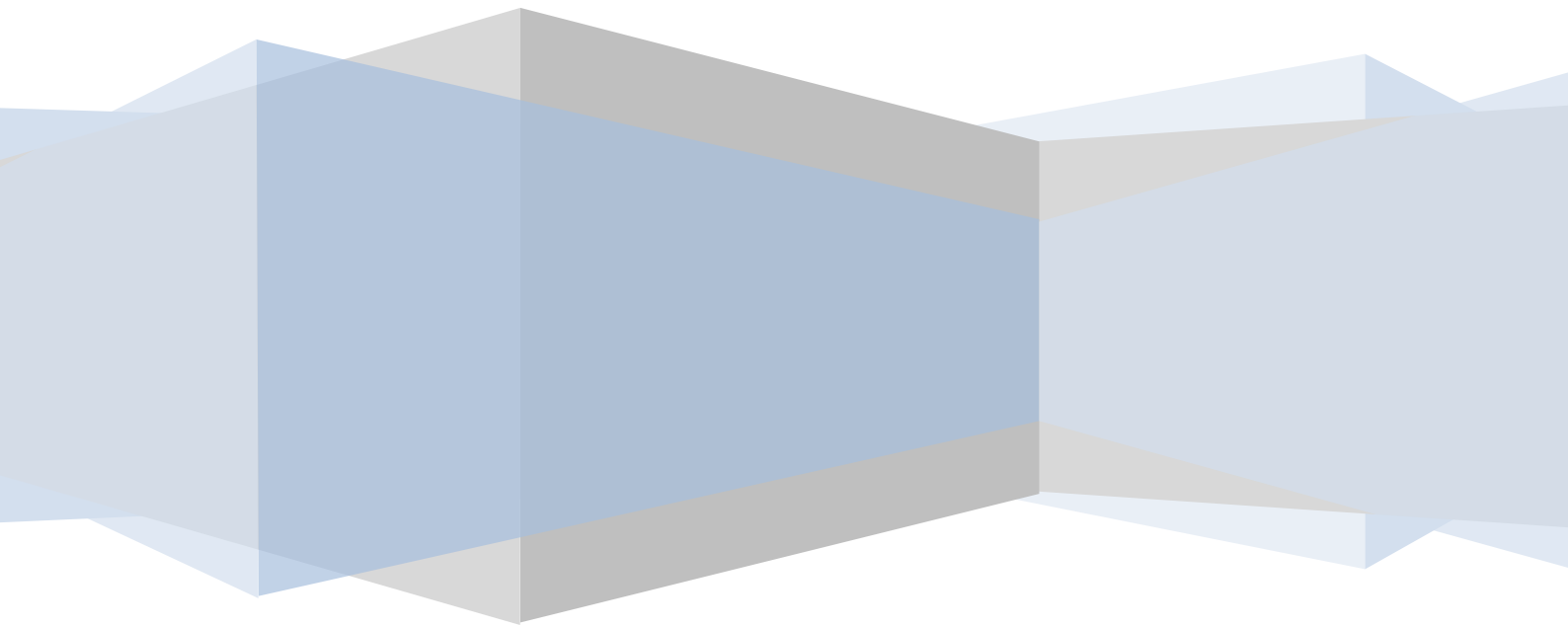
CHAPTER TWO

Theoretical Background

2-1 General Concepts

Polymers are macromolecules built up by the linking together of large numbers of much smaller molecules. The small molecules that combine with each other to form repeating molecules are termed monomers, and the reactions by which they combine are termed polymerizations. There may be hundreds, thousands, tens of thousands, or more monomer molecules linked together in a polymer molecule [25].

Polymers have used in a wide range of applications as coatings, as adhesives, as engineering and structural materials, for packing, and for some clothes. There is now an increasing demands for highly specialized materials for use in for example optical and electronic applications and polymers have been singled out as having particular potential in this regard [26].

2-2 Polymer structure

Since most polymers are organic in origin, must be reviewed some of the basic concepts relating to the structure of their molecules. First, many organic materials are hydrocarbons; that is, they are composed of hydrogen and carbon. Furthermore, the intermolecular bonds are covalent. Each carbon atom has four electrons that may participate in covalent bonding, whereas every hydrogen atom has only one bonding electron [27].

A single covalent bond exists when each of the two bonding atoms contributes one electron. Double and triple bonds between two C atoms include the sharing of two and three pairs of (-e). Like, C_2H_4, as in structural form [28].

$$
\begin{array}{ccc}
\text{H} & & \text{H} \\
| & & | \\
\text{C} & = & \text{C} \\
| & & | \\
\text{H} & & \text{H}
\end{array}
$$
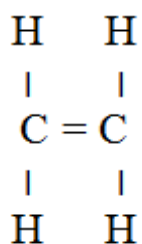
Double and triple bonds between molecules refer to an unsaturated state [29].

2-3 Classification of Polymers

There are several ways of classification of polymers based on some special considerations; the following are some of the common classifications of polymers:

2-3-1 Classification Based on Source:

This type includes three sub categories.

a- Natural polymers:

These polymers are compounds, which come from animals or plants. Like starch, proteins, cellulose, wool, some resins and rubber.

b- Semi-synthetic polymers:

The usual examples of this sub category are cellulose derivatives as cellulose acetate and cellulose nitrate, etc.

c- Synthetic polymers:

Which are prepared from simple chemical compounds and represent the most industrial important polymers, involve plastics, synthetic leather, nylon fabrics, synthetic rubbers (Buna - S), and some other dyes [30]

2-3-2 Classification Based on the chemical nature of polymer:

This type includes three sub categories [27].

a- Organic polymers

b- **Pure inorganic polymers consist in structure (Si, N, P-N, -B- N), like silicon polymers.**

c- Organic – Inorganic Polymers, Silicon Rubbers.

$$HO-\left(Si-O\right)_n-H$$

with R above and R` below the Si.
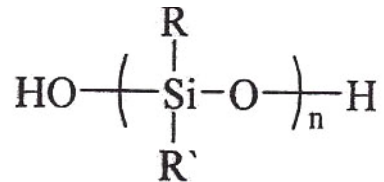
2-3-3 Classification Based on the behavior of polymers with rising temperature.

1- Thermoplastic polymers:

Thermoplastics soften when heated (and eventually liquefy) and harden when cooled-processes that are totally reversible and may be repeated.

2- Thermosetting polymers:

Thermoset polymers are generally harder and stronger than thermoplastics and have better dimensional stability.

3- Elastomers polymers:

It's one a rubber material that can be stretch to twice its original length and back to its length (original) [31-32].

2-3-4 Classification Based on the structure of polymer:

1- **Linear polymers:** Van der Waals bonding between chains as illustrated in figure (2-1a). Examples: polystyrene, poly (vinyl chloride), nylon and polyethylene.

2- **Branched polymers:** Chain packing efficiency is reduced compared to linear polymers - lower density as illustrated in figure (2-1b). Such as (LDPE) and (HDPE).

3- Cross-linked polymers: Chains are connected by covalent bonds. Often achieved by adding molecules or atoms that form covalent links between chains, as in figure (2-1 c). Many rubbers have this structure.

4- Network polymers: as shown in figure (2-1d) [33-34].

(a)   (b)

(c)   (d)
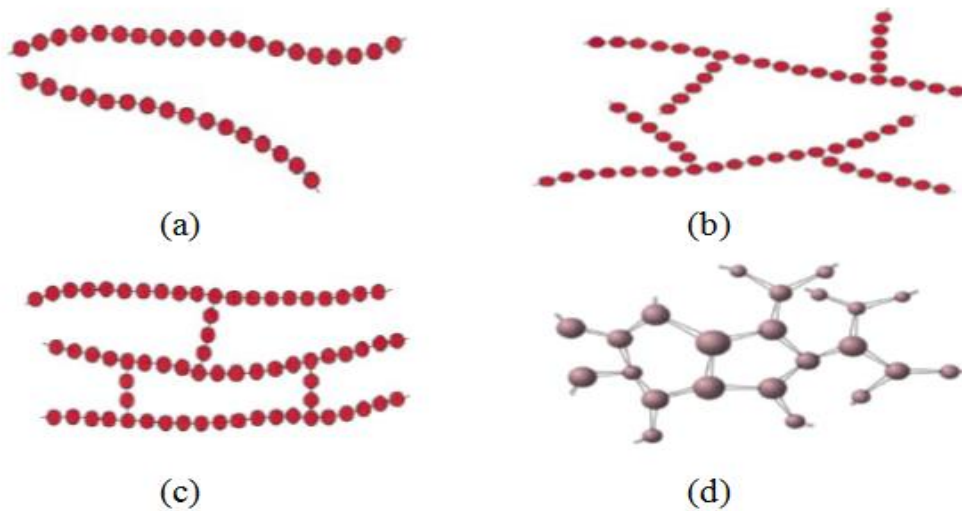
Figure (2-1) Schematic representations of (a) linear, (b) branched, (c) cross linked, and (d) network molecular structures. Circles specify individual repeating units [33].

2-3-5 Classification Based on the Homogeneity of Polymers:

1. **Homo Polymers**

   If the polymer has all the same monomers then it is said to be a homopolymer, as in poly therphethals ethylene.

<div align="center">-A-A-A-A-A-A-</div>

2- Copolymers:

Where the building blocks of a polymer are more than one type, as in the Polymer.

a) Uniform as,

<div align="center">-A-A-A-A-A-A-B-B-B-B-B-B-</div>

b) Random copolymer (rubber) contains a random distribution of monomer units like,

-A-B-B-B-A-A-A-B-B-B-B-A-A-

3- Alternate:

Some polymers like nylon (6, 6) or poly (ethylene terephthtalate) are made from two monomers, but both monomers must alternate if they are to produce a polymer so these types of polymers are also defined as homo polymers.

-A-B-A-B-A-B-A-B-
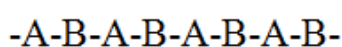
4- Polymer Blends: contain from two or more polymer types [35- 36].

2-3-6 Classification of Polymers Based on the Polymerization Reaction:

1- Classification Based on the old classification:

    a. Condensation polymers.

    b. Addition polymers like polystyrene.

2- Classification Based on the Mechanism of Chain Growth , Involve:

    a. chain growth polymerization

    b. Step growth polymerization [36].

2-4 Polymerization Process

Its process of transforming monomers without making any variation in the original structure of molecules, this process involve transfer low molecular

weight (monomers) to high molecular weight materials. These processes include two groups:

1- Additional Polymerization.

Also called chain growth polymerization. Where, a polymer is formed at the first stages. Such as in polymerization ethylene to polyethylene [35].

$$CH_2=CH_2 \rightarrow CH_2-CH_2- \rightarrow (-CH_2-CH_2-)_n$$
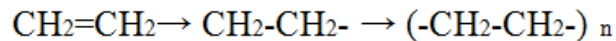
2- Condensation Polymerization

In contrast with the additional polymerization, where called step growth polymerization, the polymer is formed at the last stages of the polymerization process, like, polyesters [37]

2-5 Degree of Polymerization

Molecular Weight = (n) x Molecular Weight for (n)   [38].

(n), called the degree of polymerization

2-6 Composites Material

Composite materials are substance system composed of a combination of two or more than two substance that differs in form on substance composition. The properties of a composite are different from those of its substance [39].

It is composed at least two phases or more, one called the matrix phase and the other called the reinforcement phase. Matrix is percolating phase in which is embedded the reinforcement [33].

2-7 Types of Composites

There are many types of composite classification according to the type of matrix: [33- 40].

1. Metal matrix composite (MMCs).

2. Ceramic matrix composites (CMCs).

3. Polymer matrix composite (PMCs).

The composites may be also classified on the basis of the type of reinforcement they employed as shown in figure (2-2): [41].

1. Short fiber, or whisker reinforced, composites.

2. Particle reinforced composites.

3. Continuous fiber or sheet reinforced.

4. Laminate composite.

Particle-reinforcement, fibers reinforce and structural composites also have had at least two subdivision exits for each. For particle-reinforcement composite the dispersed phase is equaled (i.e., particles in all directions); for fiber-reinforcement composite.

Structural Composites are combination of composite materials and homogenous material [26].
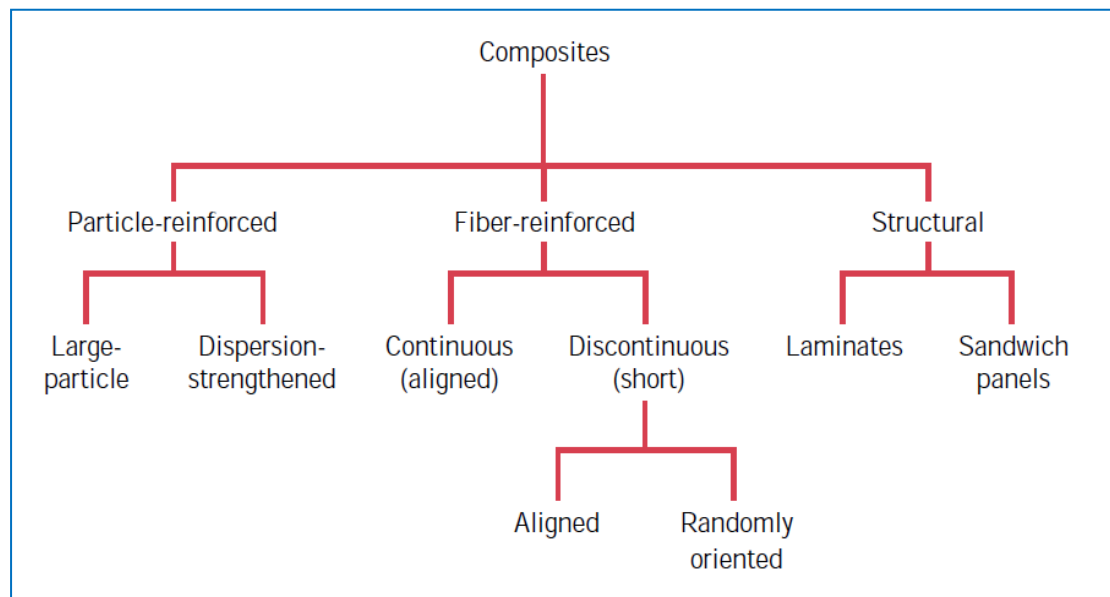
Figure (2-2): classification scheme for the various composite types [26].

2-8 Polymer Matrix Composites

Polymer matrix composites are widely used in building construction, sporting goods, transportation, and in aerospace. The reasons of the wide acceptance of these composites are [42]:-

1. Light weight.

2. Simple installation.

3. Good machinability.

4. Easy of producing simple and complex shapes.

5. Excellent cost-performance ratio.

6. Controlled insulator or conduction.

7. High strength-to-weight ratio (specific strength).

8. Long service life.

- **In this work the polymer matrix used is Epoxy resin.**

2-9   Epoxy resin

Epoxy systems are made up of an epoxy resin and a hardener or catalyst. Epoxy resin from thermosetting materials. [43]. An epoxy resin is defined as a molecule with more than one epoxy group, which can be hardened into a usable plastic. The epoxy group as shown in figure (2-3), called the glycidyl group, has through its characteristic appearance given the name to epoxy [9].

$$CH_2-CH-$$
Epoxy group
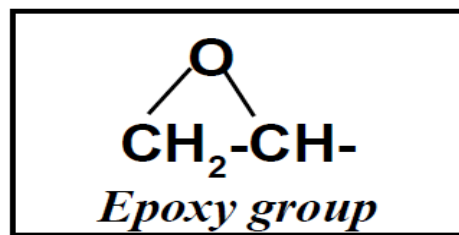
Figure (2-3) Epoxy group.

Epoxy resin is manufactured from simple basic chemical show in figure (2-4) that are readily available.
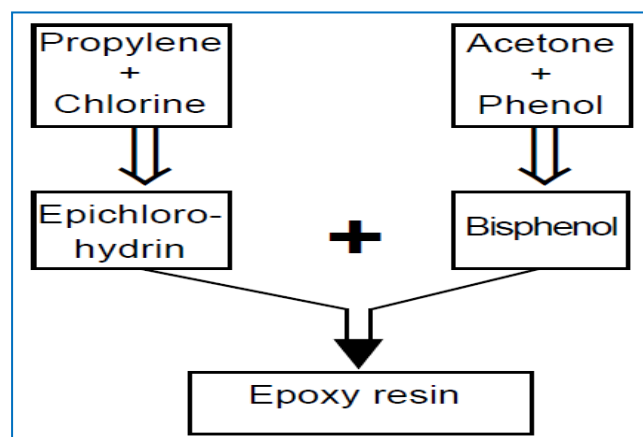
Figure (2-4): Epoxy resin manufactures [9].

To convert epoxy resin to epoxy plastic, a reaction with a suitable substance is required. Such a substance in this context is called a (hardener) [9].

All epoxy resins have these terminating end groups which can bond chemically with each other and with other molecules to form a large three dimensional network during the cure process [44].The structure of Epoxy is explained in figure (2-5):
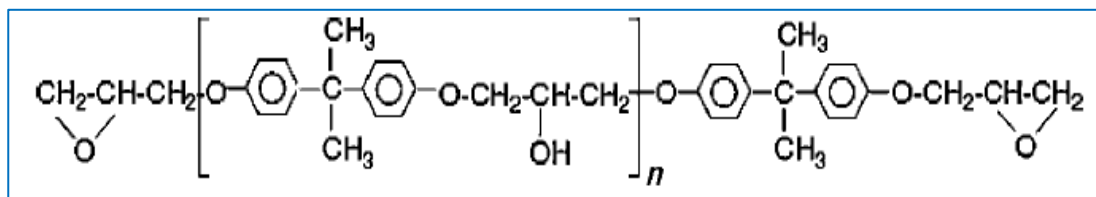
Figure (2-5) Epoxy structure [34].

2-9-1 Physical Properties of Epoxy

Epoxy resins are arguably one of the most versatile polymers with uses across an enormously wide variety of industries. The outstanding physical properties exhibited by epoxy resins include**:**

• Low cure shrinkage.

• No volatiles given off during cure.

• Compatibility with a great number of materials.

• Strength and durability.

• Adhesion.

• Corrosion and chemical resistance.

• Electrical insulation.

Furthermore, epoxy resin systems are capable of curing at either ambient or elevated temperatures, and they require only minimal pressure during the cure. Thus, epoxies can be applied and cured under many adverse conditions including outdoors. These properties provide great added value in many industries engaged in product assembly [44].

2-10 Composites with Particular Fillers

A composite whose reinforcement by relatively micro size particles is called particulate composite, the particles and matrix material in particulate composite can be any combination of metallic and nonmetallic materials. The choice of a particular combination depends on the desired properties. Many types of particles used as reinforcement material such as ceramic, metal and inorganic particles.

The hard particles in a particulate composite place constraints on the plastic deformation of the matrix material between them, because of their inherent hardness relative to the matrix, the particles also share the load. Thus these particles are effective in enhancing the stiffness of the composites, but do not offer the potential for much strengthening. Hard particles placed in brittle matrix reduce the strength due to stress concentrations. Many particle fillers used to improve the other properties of matrix materials such as electrical and thermal conductivity, improve performance of elevated temperature, increase wear and abrasion resistance, reduce friction, improve machinability, increase surface hardness and reduce shrinkage. Two types of particles could be reinforced materials, spherical particles, and platelet particles [42].

2-11 Hand Lay-Up Method

Hand lay-up is an open molding method suitable for making a wide variety of composites products from very small to very large. Production volume per mold is low; however, it is feasible to produce substantial production quantities using multiple molds. Hand lay-up is the simplest composites molding method, offering low cost tooling, simple processing, and a wide range of part sizes. Design changes are readily made. There is a minimum investment in equipment. With skilled operators, good production rates and consistent quality are obtainable. In this technique only one mould is

used, and this may be either male or female. A suitable mould pattern is prepared from wood, metal, plastic [42].

2-12 Mechanical Properties

Polymer is a material having many unique properties like low density, reasonable strength, flexibility, easy processibilty, etc. However, the mechanical properties of these materials are inadequate for many engineering applications. Mechanical Properties of polymeric materials depends on the infrastructure of the molecules of the major chains, Different bonds between atoms and molecules and between chains will lead to change in these characteristics. The mechanical behavior of materials describes the response of materials to mechanical force or deformation [45].

2-12-1 Polymer Fracture

Large ranges of fracture are observed in the polymers, depending on the microstructure [45]. In an amorphous polymer the common form of failure is brittle failure with temperature below their glass transition temperature [46 - 47]. The chains are arranged randomly in amorphous polymer with no orientation. Fracture tends to occur by craze formation in amorphous polymer due to stretching of polymer and that arise gaps between chains [48] and three stages for cracking of an amorphous polymer, as in figure (2-6), [47].
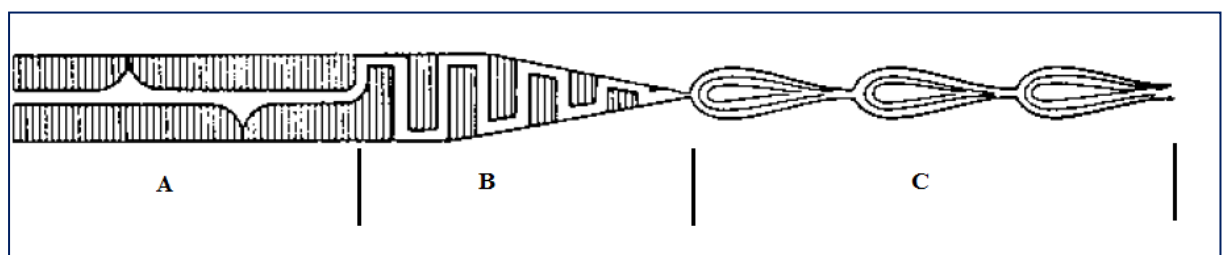
Figure (2-6) Shows diagram of stages of cracking into amorphous polymers. A: crack by void creation; B: crack advance along alternate craze–matrix interfaces; c: crack advance through craze bundle to form hackle bands [47].

The fracture surface appearance and mechanisms for composites depend on the fracture characteristics of the composite (matrix and reinforcement) matters and on the effectiveness of the bonding between the matrix and reinforcement [49]. The damage in composite matters may happen by Interfacial/interphase cracking and de-bonding, Particle pull-out or fibril cracking [45]. The creation of cracks may be a compound fracture process, which strongly depends on the microstructure of a specific crystalline or amorphous solid, imposed loading, and environment. The microstructure plays a very significant role in a fracture process because of dislocation motion, precipitates, grain size, inclusions, and kind of phases making up the microstructure. All these micro structural features are defect and can act as fracture nuclei under negative conditions. For instance, Brittle Fracture is a low-energy process (low energy dissipation). On the other hand, Ductile Fracture is a high-energy process in which a large amount of energy dissipation is related with a large plastic deformation before crack instability happens. Consequently, slow crack growth happens due to strain hardening at the crack tip area [50-52]. Inorganic fillers particles were generally addition to polymer to improve the stiffness, if there is a strong interfacial strength also to rise the yield stress. In case of low or lost interfacial strength (adhesion), no-bonding and cavitation seems during loading. The micro-voids about the filler particles action as stress concentrators (like the elastomeric particles in rubber particle toughening) and can start local yielding processes. In dependence on particle distribution and particle size, several cases often seem as in figure (2-7) [53]:

a. The basic effect is no-bonding/cavitation and local stress concentration.

b. Big particles create great voids with the disadvantage of void coalescence and construction of cracks of overcritical lengths.

c. Agglomerates of small particles can break, constructing sharp cracks.

d. Small, homogeneously distributed particles start local yielding among the particles/micro-voids.
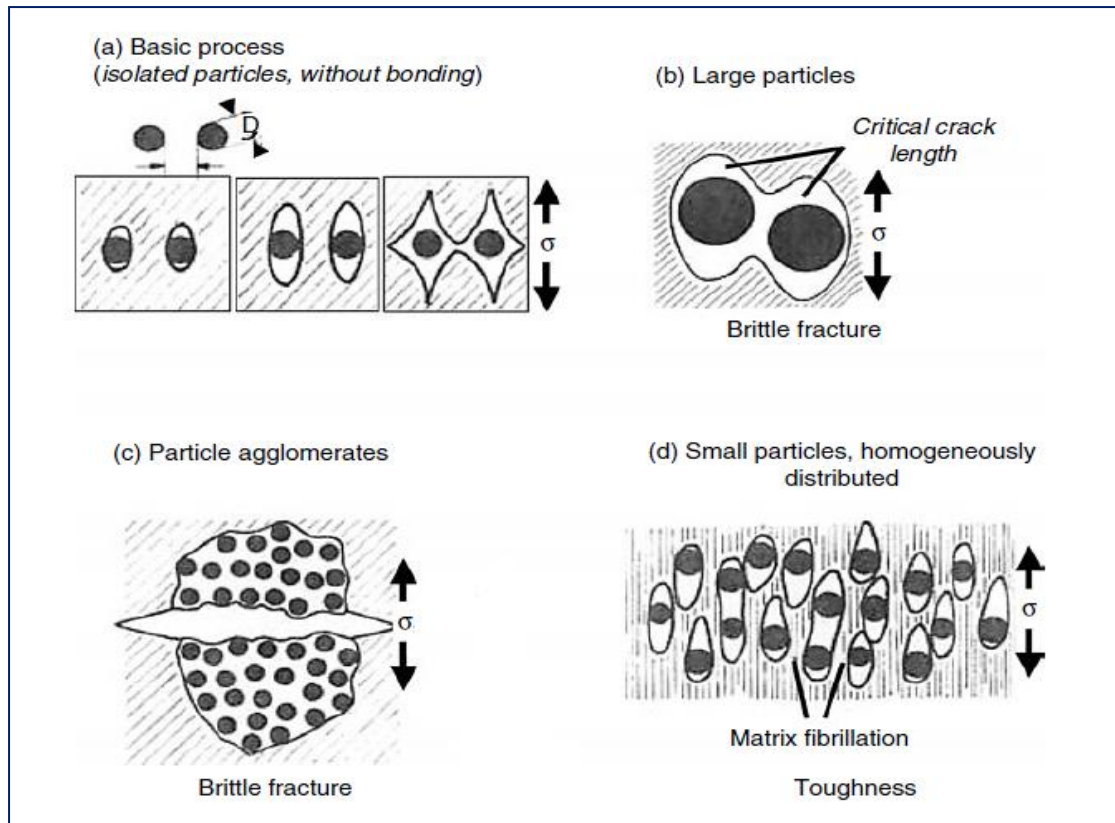
Figure (2-7) Illustrations of particle-filled polymers and changed cases of local processes, depending on distribution and particle size [53].

2-12-2 Three Point Bending

Bending test is exposing the sample to an external load (known as flexure) [54]. In bending, and for rectangular shape specimen, the convex side of the specimen extended, and the concave side is compressed with an unstrained neutral axis through the center [55]. Figure (2-8) illustrates three-point bending [56].
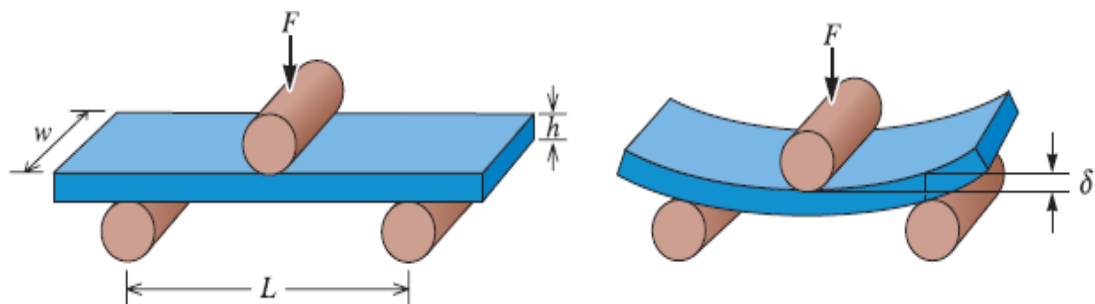
Figure (2-8) (a) the three -point bend test. (b) The deflection δ obtained by bending [56].

The material properties supposed uniform through the thickness. Under these circumstances (force distribution, uniform material) the usual stress varies linearly from a maximum in compression on one surface to an equal maximum in tension on the other surface, When the imposed force at the sample midpoint cause in bending this constitutes a bending moment (M) close to sample ends while (Mm) is the moment of resistance figure (2-9) [57].
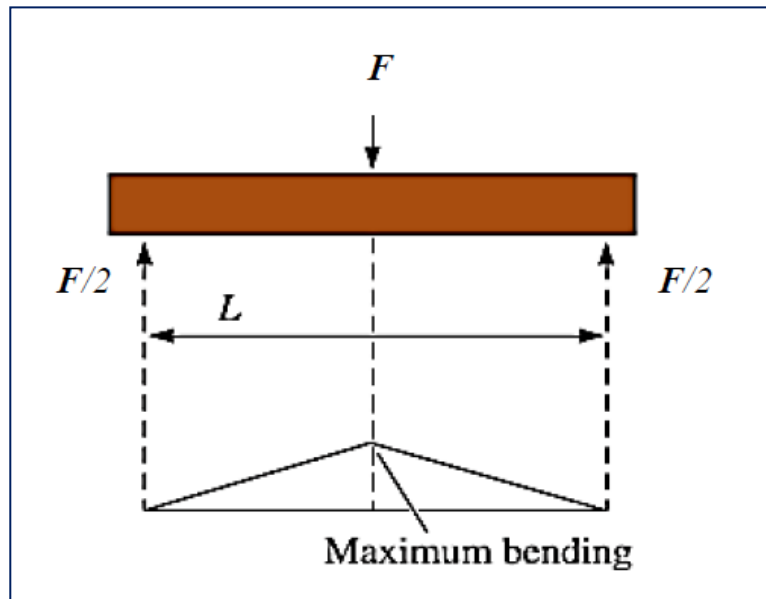
Figure (2-9) Three-point bending test setup and force distribution [57].

Where:

$$M = \frac{Fl}{4} \qquad\qquad (2-1)$$

$$M_m = \frac{wh^2}{6} \qquad\qquad (2-2)$$

For (σf),

$$\sigma_f = \frac{M}{M_m} \qquad\qquad (2-3)$$

$$\sigma_f = \frac{3Fl}{2wh^2} \qquad\qquad (2-4)$$

The E (Young modulus) calculated in the elastic region is [55]:

$$E = \frac{I^3 S}{4wh^3} \qquad\qquad (2-5)$$

Where **I** is the span of specimen, **w** is the width of the specimen, h is the thickness of specimen, S is the slope of force (**F**) - deflection (**D**) [57], stress and strain are proportional to each other through the relationship.

Flexural strain (ϵ_f) is given by:

$$\epsilon_f = 6D\,w/\,I^2 \qquad\qquad (2-6)$$

Stress distribution across the specimen thickness (h=2c) is demonstrated in figure (2-10).

a) Stress distribution in a rectangular beam subjected to elastic bending.

b) Stress distribution in a rectangular beam subjected to bending if yielding occur.
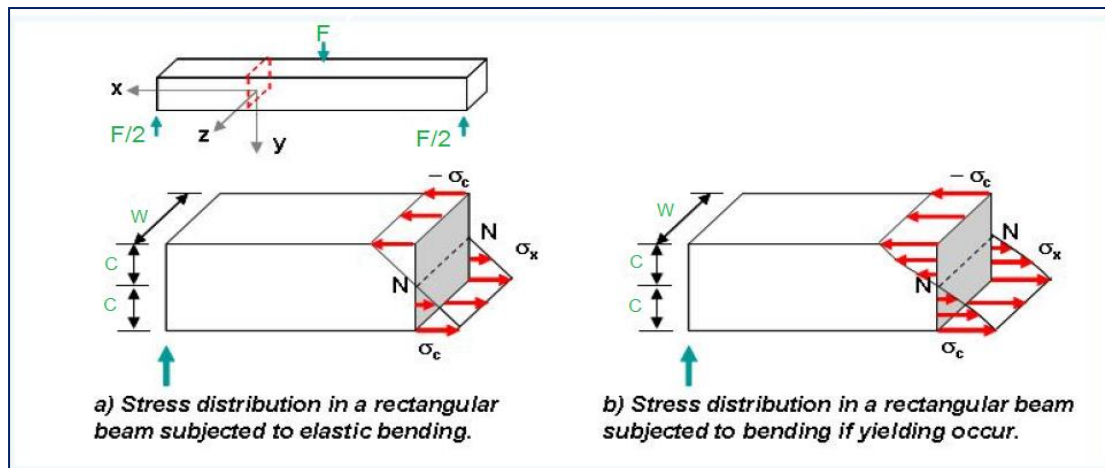
Figure (2-10) Stress distributions in a rectangular bar when (a) elastically bended and (b) after yielding [57].

2-12-3 Hardness

Hardness is the property of a material that enables it to resist plastic deformation, usually by penetration. However, the term hardness may also refer to resistance to bending, scratching, and abrasion. Shore hardness is a measure of the resistance of a material to penetration of a spring loaded needle-like indenter. Shore A scale is used for testing soft Elastomers (rubbers) and other soft polymers. Hardness of hard elastomers and most other polymer materials (Thermoplastics, Thermosets) is measured by Shore D scale. Shore hardness is tested with an instrument called Durometer. Durometer utilizes an indenter

loaded by a calibrated spring. The measured hardness is determined by the penetration depth of the indenter under the load [58-59].

2-13 Thermal Properties

2-13-1 Thermal conductivity

Thermal conduction is the phenomenon by which heat is transported from high to low temperature regions of a substance. The property that characterizes the ability of a material to transfer heat is the thermal conductivity. The basic law of the thermal conductivity is Fourier's law [33]:

$$q = -k\frac{dT}{dx} \qquad\qquad (2-7)$$

Where q: the flux of thermal energy transmitted across a unit area per unit time, K is the thermal conductivity coefficient, and (dT/dx) is the temperature gradient. The units of q and k are W/m^2 and W/m. k, respectively.

Equation (2-7) is valid only for steady – state heat flow, that is, for situations in which the heats flux do not change with time. Also, the minus sign in the expression indicates that the direction of heat flow is from hot to cold or down the temperature gradient. The phonon-aspect of lattice vibrations is particularly appropriate when energy transformation is of concern. Such energy transformation processes include the creation of and annihilation of phonons. Thermal conduction is most conveniently described in terms of the scattering of phonons; by other phonons, by static imperfections, or by electrons. Heat energy can be transmitted through a material via the motion of phonons, photons, free electrons (or free hole), electrons-hole pairs, or exciton (bound electron-hole pair), According to the mechanism of heat transfer [33].

Specifically there are two mechanisms for heat transfer through a solid substance [33].

*Free electrons and lattice vibration in solid conductors are the dominant mechanism of temperature transfer.

*The phonons in solid insulator substances are the unique mechanism.

2-13-2 Thermal Conductivity measurement Principle

The methods used to determine thermal conductivities vary greatly with the nature of the substance under investigation. Three cases may be distinguished [60].

1-Good Conductors; which includes:

  a- A direct method; by Forbes's method

  b- An electrical method; by kohlrausch's method.

  c- A comparative method; by Wiedemann and Franz method.

2-Poor Conductors; by Lee's method

3-Gases; by Hercus a Laby method

The K_{th} of small thin disc of material was determined by Lee's method [61], as shown in figure (2-11).

The heat received per sec. by disc A and given up to air is [61]

$$(\pi r^2 + 2\pi r\ d_A)\ e\ T_A \qquad\qquad (2\text{-}8)$$

The heat received per sec. by sample and given up to air from its exposed surface or passed on to A is [60]:

$$e\ T_A(\pi\ r^2 + 2\pi\ r\ d_A) + 2\pi\ r\ d_s\frac{1}{2}(\ T_A + T_B) \qquad\qquad (2\text{-}9)$$

In terms of I.V can be obtained, since the total heat supplied must be equal to that given up by the various surfaces [60]:

$$H = I.V = \pi\ r^2 e\ (T_A + T_B) + 2\pi r e\ [\frac{1}{2}d_s\ (T_A + T_B) + d_A T_A + d_B T_B + d_C T_C] \qquad (2\text{-}10)$$

I.V: rate of supply of energy to the heater, after the steady state has been reached.

e: heat loss per sec. per sq.cm for each 1° increase of discs temperature, over that of enclosure.

T: excess of temperature over that of the enclosure.

d: thickness of disc.

r: radius of disc.

So, thermal conductivity becomes [60]:

$$K_{th}\left((T_B - T_A)/ds\right) = e\left[T_A + \frac{2}{r}\left(d_A + \frac{1}{4}ds\right)T_A + \frac{1}{2r}dsT_B\right] \qquad (2\text{-}11)$$
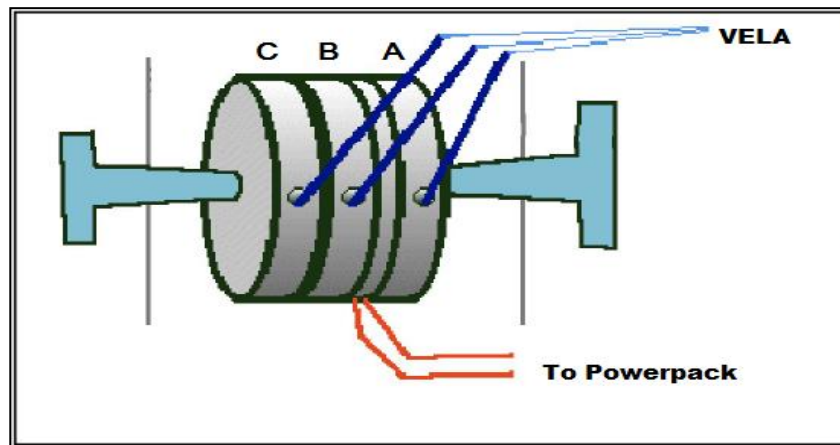
Figure (2-11) Schematic diagram of Lee's disc technique [60]

2-13-3 Differential scanning calorimetry (DSC)

DSC is an analytical technique in which the difference in the amount of heat required to increase the temperature of the sample and reference is measured as a function of the temperature. Both the sample and the reference are kept at approximately the same temperature throughout the experiment. Overall, the temperature program is designed to analyze the DSC so that the temperature of the sample holder increases linearly as a function of time. The reference sample should have a well defined heat capacity over the temperature to be scanned [62].

The glass transition temperature (Tg) of a polymer can be detected by using (DSC), as an endothermic shift from the baseline is observed at the (Tg) in the traces of crystallize able polymers. Such a change results from an increase in heat capacity due to the increased molecular motions in the material. It should

be noted that (Tg) value depends strongly on the heating and cooling rates used in a differential scanning calorimetry run.

The (Tg) is the temperature at which an amorphous polymer ceases to be brittle and glassy and becomes less rigid and rubbery. As a polymer is heated up to (Tg) the molecular rotation about single bonds become significantly easier, the change of volume of a polymer with increase temperature [30] [63].

2-14 Electrical Properties

Electricity is the movement of charged units called charge carriers. These units are the electrons and holes of the matter in the solid state or its ions in the liquid and gas state [64]. Thus, the Materials can be divided into groups or families according to their conductivity: conductors, semiconductors, insulator and Superconductive matters [65].

1- Insulators: its conductivity ranges from (10^{-22} –10^{-12}) (Ω. cm)$^{-1}$.

2- Semiconductors: its conductivity ranges from (10^{-13}–10^{-3}) (Ω. cm)$^{-1}$.

3- Conductors (metals): its conductivity is more than (10^{-3}) (Ω. cm) $^{-1}$.

4- Superconductive matters: its conductivity is (10^{20}) (Ω. cm)$_{-1}$. [65]

To understand the electrical properties of the materials and how electrical conductivity takes place, it is necessary to know more about the energy levels of atom and the electronic distribution in these levels. Electrons are distributed in an atom in different energy levels, electrons closer to the nucleus are closely connected to the atom, the more distanced the electrons from the nucleus, and the less is the connection of electrons to the nucleus. Consequently, electrons that occupy the orbital which is the most distant from the nucleus are the least connected to the nucleus. These electrons are called valence electrons. Valence electrons are distributed among many levels of energy which are called valence band [66].

Though the electrons that occupy the valence band are weakly connected to the nucleus, this connection is making them unable to move. By gaining certain energy, valence electrons can transfer to higher levels, called the conduction band. The difference in energy between the conduction band and valence band is called energy gap (E_g).

Electrons of the conduction band are very weakly connected to the nucleus and that makes them easily move by the influence of external effects such as the exertion of electrical field. The difference of energy (ΔE) determines the electrical properties of matter [67].

Metals: One of the most important properties of metals is their good electrical conductivity, which is attributed to their electronic configuration. Electrons are equally distributed between the conduction band and the valence band so that the last band is occupied with a large number of electrons in nearly normal temperature, ensuring effective transfer of electric current. In metals, the energy gap (E_g) is very small, conductivity in metals does not depend on temperature, on the contrary, increased temperature in metals (conductors) decreases electrical conductivity. Resistance in metals is caused by two reasons:

First, nucleus constituting the metal crystal has vibrating movement which is called phonon. This movement influences the movement of electrons or charges that transfer the current; it scatters and deviates them from their trajectory. Increased temperature decreases electrical conductivity in metal conduction. Second, the resistance resulting from the defect in the metal crystal lattice(crystal defect), is not influenced by temperatures, this defect cuts off the trajectory of electrons and charges that transfer the electric current [64].

Semiconductors: Many solid elements have the properties of conductors and insulators, such as Silicon and Germanium. Such elements are called semiconductors. They are different in their electronic configuration from that of metals, the number of electrons in the valence band is greater than that

in the conduction band because the energy gap is bigger in semiconductors, and the electrons in the valence band need greater energy to transfer to the conduction band [68]. Since the number of excited electrons is increased by temperature, semiconductor conductivity increases with increased temperature. This is exactly the opposite in metals [69].

Insulators: The energy gap (Eg) is very much greater in insulators than in conductors and semiconductors, and the conduction band is empty of electrons, which are distributed in insulators at lower energy levels of the valence band, making them strongly bound to the atom with little movement, to be released, these electrons need high energy to enable them transferring to a higher energy level weaken their binding to the atom and facilitate their movement among atoms [64]. Figure (2-12) shows energy gap in metals, semiconductors and insulators.
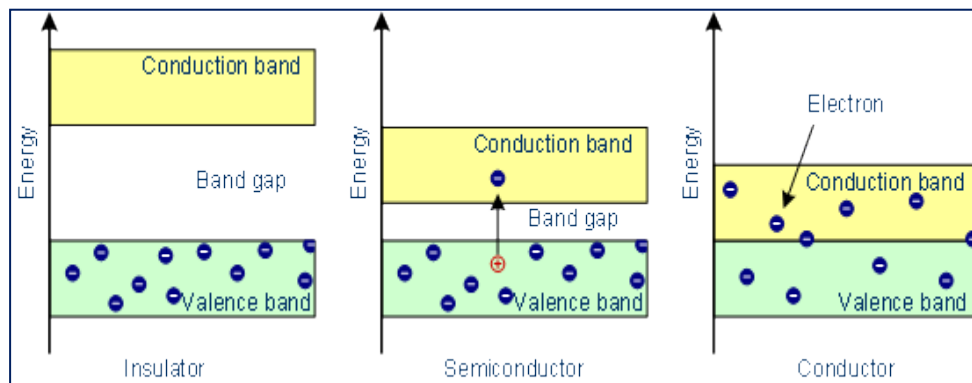
Figure (2-12): The energy gap and distances among the conductor, semiconductor and insulator materials [70].

2-14-1 A.C Conductivity

Information about the nature of the conduction mechanism in material can be obtained from A.C. conductivity. In these experiments, the electrical conductivity is measured as a function of the frequency (f) [70].

The dielectric constant, έ, is calculated from the equation:

$$\acute{\varepsilon} = d \, C / A \, \varepsilon_o \qquad (2\text{-}12)$$

Where:

ε_o is the permittivity of free space ($\varepsilon_o = 8.854 * 10^{-12}$ Farad/m).

d is the thickness.

C is the capacitance.

A is the cross section area.

The dielectric loss ($\ddot{\varepsilon}$) is calculated from the equation:

$$\ddot{\varepsilon} = \acute{\varepsilon} \tan\delta \qquad (2\text{-}13)$$

Where:

$\delta = 90 - \phi$, ϕ is the phase different angle and δ is the phase angle between the electric field and the polarization of the dielectric.

The $\tan\delta$, is a material property, and is not dependent on geometry of a capacitor [71-73].

2-14-2 Dielectric Constant

The ($\acute{\varepsilon}$) is ability of a material to polarize in response to an applied field. [74]. Polymers and the atoms that make them up have their electrons tightly bound to the central long chain and side groups through 'covalent' bonding. Covalent bonding makes it much more difficult for most conventional polymers to support the movement of electrons and therefore they act as insulators [75].

2-14-3 Dielectric Polarization

A dielectric introduce into an electric field undergoes polarization this phenomena, known as dielectric polarization, involves appearance of electric charges on the surface of dielectric. These charge known as induced charges, are not free to move and they generate a field known as the depolarization field in the dielectric, whose direction is opposite to the direction of the field that gives rise to polarization [76 -77].

The degree of polarization P is $\acute{\varepsilon}$ and the $\tilde{E}$ (electric field strength) as follow:

$$\mathbf{P} = \varepsilon_0 (\acute{\varepsilon}\text{-}1) \tilde{E} \qquad (2\text{-}14)$$

Where ε_0 is ($\varepsilon_0 = 8.85 * 10^{-12}$ F/m).

In a most general case the total polarizability α may be expressed as

$$\alpha = \alpha e + \alpha i + \alpha d + \alpha s \qquad \ldots\ldots\ldots\ldots\ldots\ldots \text{ (2-15)}$$

Where: αe: the Electric polarization,

αi: The ionic polarization,

αd: The dipolar polarization,

αs: The Space charge polarization,

These polarization components are explained below [78]:

2-14-3-1 Electronic Polarization αe

This effect is common to all materials, because they include distortion of the charge symmetry of the basic atom. Under the influence of the application field, the nucleus of the atom and the center of the negative charge of electrons shift, creating a small dipole. This polarization effect is small, despite the huge number of atoms within the material, because the momentary arm of the dipole is very short, which probably only contains a small fraction of an Angstrom [73].

2-14-3-2 Ionic Polarization αi

Ionic displacement is common in ceramic materials. Under the influence of an electric field, dipole moments are created by shifting of these ions towards their respective (opposite) polarity of the field. [73].

2-14-3-3 Dipolar Polarization αd

This is a phenomenon that involves the rotation of permanent dipoles within an applied field. In polar molecules, the center of gravity of positive charges and charges of negative charges are not synchronized. A small molecular dipole is shown, although there is a permanent dipole in the ceramic compounds. Dipole orientation is more common in polymers by virtue of its

atomic permit. Orientation polarization belongs to slow or relax polarization types. [78].

2-14-3-4 Space Charge Polarization (Interface) αs

The space charge is produced by the separation of mobile positively and negatively charged particles under an applied field, which form positive and negative space charges in the bulk of the substance or at the interfaces between various substances [79].
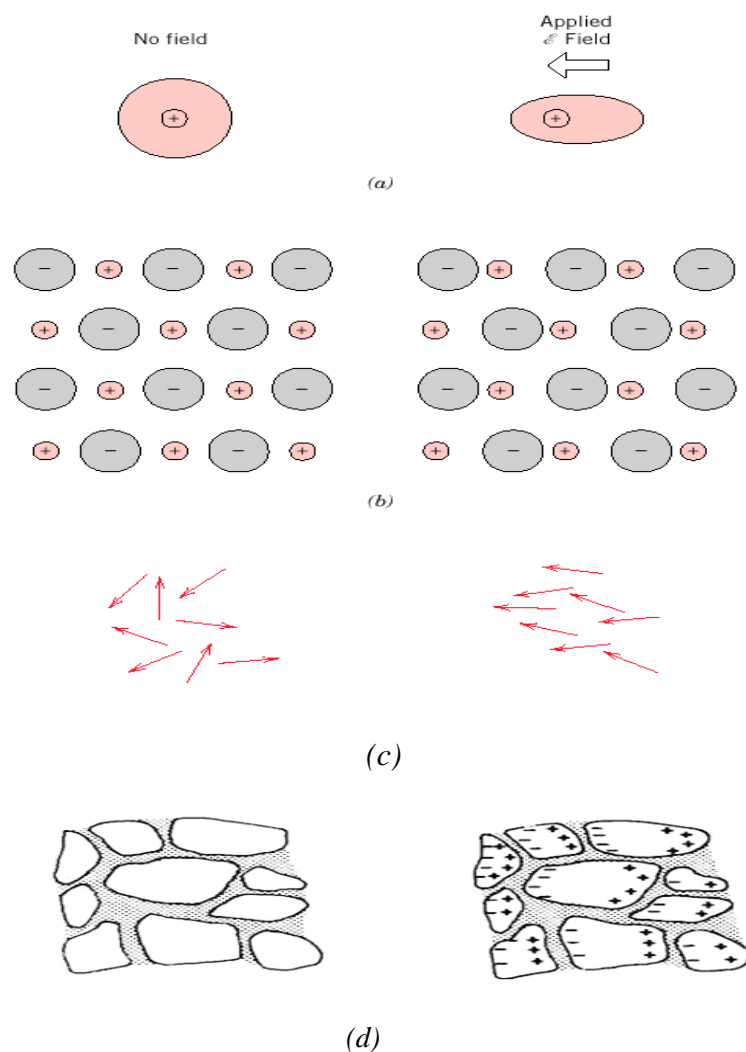
Figure (2-13) Basic kinds of electric polarization: (a) Electronic polarization, (b) Atomic or ionic polarization, (c) Dipolar polarization and (d) Interface or space Charge polarization [80].

2-14-4 Capacitance

The capacitance C of a capacitor is determinate as the ratio of the charge Q on conductors to the potential difference V between the plates of the capacitor.

$$C_0 = Q/V \qquad (2\text{-}16)$$

Capacitance is as well determinate by multiplying the permittivity of free space and electrode surface area A, then dividing by the thickness separating the electrode d. Units are expressed in Farad.

$$C_0 = \varepsilon_0 A/d \qquad (2\text{-}17)$$

If the space between the plates of a capacitor is full of dielectric of permittivity ε the capacitance may be calculate:

$$C = \varepsilon A/d \qquad (2\text{-}18)$$

ε is the absolute permittivity of the dielectric material.

The capacitance is dependent on the kind of substance and its dimensions. The ratio of the capacitance with dielectric to the capacitance it is determinate as the relative dielectric constant ε_r.

$$\varepsilon_r = C/C_0 = \varepsilon/\varepsilon_0 \qquad (2\text{-}19)$$

From equations (2-17) and (2-18) the capacitance is then:

$$C = \varepsilon_0 \varepsilon_r A/d \qquad (2\text{-}20)$$

The equation (2-20) determinate the capacitance in terms of the amount ε of the dielectric and the given geometrical dimension (A, d) [73]

2-14-5 The Effect of Frequency on Dielectric Loss

The difference of the dielectric constant with frequency is like to the difference of polarizability and polarization. At minimum frequencies of the

order of a few Hz, the dielectric constant is made up of contributions from electronic, atomic and space charge polarization [81].

When measurements are perform as a function of frequency, the space charge polarization give rise to a specific frequency and the dielectric constant becomes frequency independent. The frequency beyond which the difference ceases may fall in the specific range. The frequency-independent amount is taken as the true static dielectric constant [78].

By measuring the dielectric constant as a function of frequency, one can separate the several polarization components. Each polarization mechanism has a located characteristic frequency. Electrons have very limit mass and are thus able to follow high frequency fields up through the optical range. Ions are a thousand times heavier but continue to follow fields up to the infrared range. Molecules— particularly those in liquids and solids—are more weight yet and are severely impeded by their surroundings. Most rotational effects, like those in water, are bounded to microwave frequencies. Space charge effects are often in the kilohertz range or even limited Frequency has an important effect on the polarization mechanisms of a dielectric. When the frequency of the applied field is quite large as compared to the inverse of the relaxation time for a particular polarization process, the contribution of that process to the polarizability is negligible. As relaxation time is the maximum for space charge polarization, the space polarization disappears first followed by dipolar, ionic, and electronic contributions. Figure (2-14) shows a typical frequency spectrum of a dielectric containing all four types of polarization [81] [82].
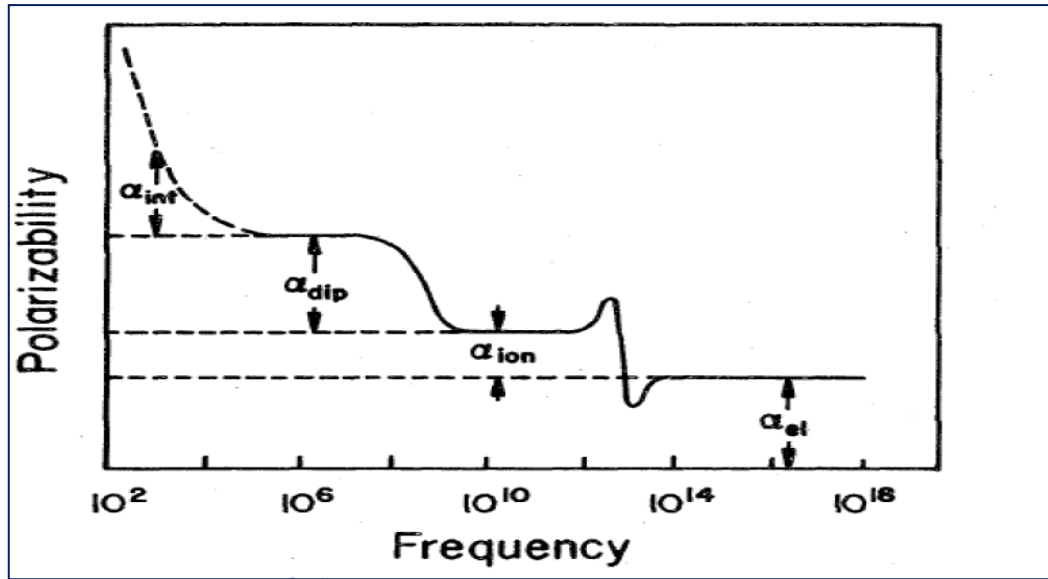
Figure (2-14): The frequency dependence of polarizability showing several contribution mechanisms [80].

2-14-6 Percolation phenomenon

Percolation theory is formulated in terms of simple geometric image such as spheres or particles in lattices. Percolation theory, as a theory of critical phenomena, is not yet a mathematically rigorous science. However, if a rigorous proof exists but appears to be very complicated, it have been decided to replace it here by arguments that will explain the result rather than prove it The present introduction, offers a summary presentation of the theory of percolation and its application to the conductivity of metal- insulator mixture. This mixture which manufactured from an insulator with tinge metallic-inclusions, are now an important object of study. The electrical properties of such materials in the vicinity of percolation threshold are fascinating. For instance, the capacitance of a capacitor filled with such a material could have extremely large value when the fraction of volume filled with the metal tends to percolation threshold [83].

The presence of conductive fillers within an insulating matrix material modifies the electric properties of the composite. The composite becomes conductive above a critical value–percolation threshold that defines the

insulator- conductor transition. The electrical percolation threshold depends on many factors including the size and shape of the filler, matrix properties, preparation method, filler properties, dispersion of the filler within matrix and the interaction between compounds [84]. The dielectric properties of the composites are also characterized by means of the dynamic dielectric spectroscopy. For the frequency dependent AC conductivity $\sigma_{a.c}$ and the real part of the permittivity $\acute{\varepsilon}$ power law equations are given by [75]:

$$\sigma a.c\,(\omega) \sim \omega^S \qquad\qquad (2-21)$$

$$\acute{\varepsilon}\,(\omega) \sim \omega^{-U} \qquad\qquad (2-22)$$

Where s and u denote scaling exponents (in range of 0 - 1), which in vicinity of the percolation threshold are related to each other:

$$s + u = 1$$

The relation of electrical conductivity is given by:

$$\sigma = \frac{d}{R.A} \qquad\qquad (2-23)$$

Where d is the thickness of specimens (cm), R is the resistance (Ω) and A is the affective area (cm^2).

CHAPTER THREE

Experimental Work
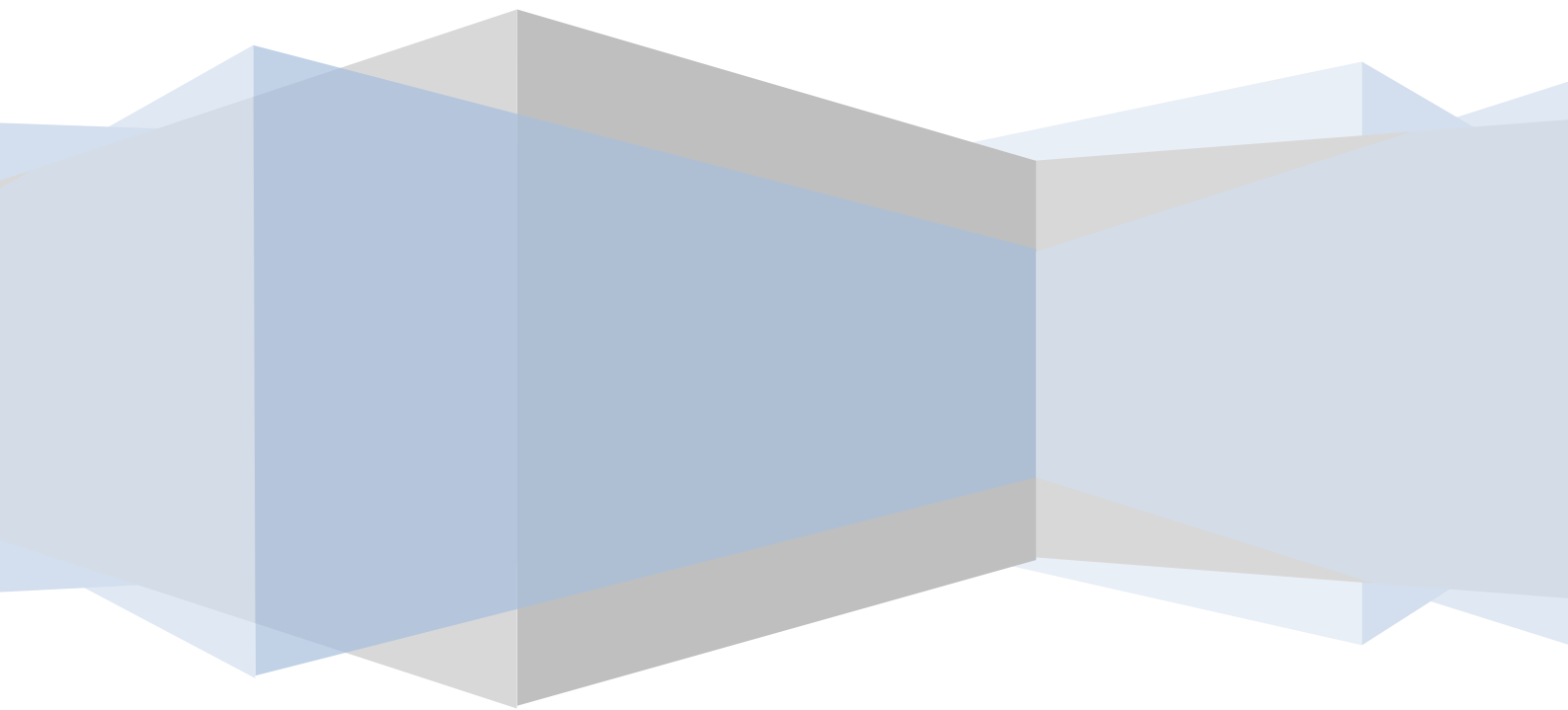
CHAPTER THREE

Experimental Work

3-1 Introduction:

This chapter deals with material processing, sample preparation, instrumentation and measurement techniques. General descriptions of materials used in this work are given. Brief descriptions of sample preparations are given which were designed for physical measurements (mechanical measurements, Electrical and Thermal Conductivity).

3-2 Materials Used:

3-2-1 Epoxy Resin:

The matrix material that was utilized is epoxy resin (EP Euxit 50). It has a trademark (EP Euxit 50) production of Swisschem is a liquid of low viscosity resin, and it is converted to solid state by adding hardener (Euxit 50 KII) at ratio of (5:2), which were supplied by Egyptian Swiss chemical industries company .The properties of epoxy resin used in this work show in Table (3.1) according to the properties of Product Company ASTM D-543 and ASTM C-881-87.

Table 3-1: The properties of epoxy material

Density at 20º C (gm/cm^3)	Viscosity 20º C	Flexural strength (MPa)	Modulus of elasticity (MPa)	Fracture Toughness (MPa)	Colour	Glass temperature º C	Bending property (N/mm^2)
1.05	300	63	2800	0.6	Colorless	57	45

3-2-2 Aluminum powder

In this research, Aluminum powder (Density 2.7 g/cm^3 and purity 99.0), was produced by the Company Central Drug House (P) Ltd, Vardaan House, New Delhi-10002, INDIA. The powder was adding to epoxy with weight percentage (0, 5%, 15%, 25%, 35%, and 45%). The physical and chemical properties of Aluminum used in this work show in Table (3-2) according to the properties of Product Company:

Table 3-2: The properties of Aluminum powder

Appearance	Colour	Density at 25 °C	Purity	Description	Crystal structure	Particle size
powder	Silver	2.7 g/cm^3	99.0	A bright Silver grey powder	face-centered cubic (Fcc)	Fine powder

3-2-3 Copper powder

In this research, Copper powder (Density 8.94 g/cm^3 and purity 99.5), was produced by the Company Central Drug House (P) Ltd, Vardaan House, New Delhi-10002, INDIA. The powder was adding to epoxy with weight percentage (0, 5%, 15%, 25%, 35%, and 45%). The physical and chemical properties of Copper used in this work show in Table (3-3) according to the properties of Product Company:

Table 3-3: The properties of Copper powder

Appearance	Colour	Density at 25 °C	Purity	Description	Crystal structure	Particle size
powder	light red	8.94 g/cm^3	99.5	Bright reddish powder	face-centered cubic (Fcc)	Fine powder

3-3 Specimens Casting

Hand lay –up method were used in this study to prepare specimens of composite material. The adopted technique is summarized as the following;

3-3-1 Mold preparation

The molds were preparation according to ASTM specifications for each test as the following;

1. Used for mechanical specimen (Three Point Bending) of glass molds with over Head paper placed on the glass plate to ensure that specimens were not stuck with the mold, glass panels are installed on the glass base by wax. ASTM-D790 Standard Test Methods for Flexural Properties. As shown in figure (3-1), and table (3-4).

   While, used for Shore D Hardness specimens of circular plastic molds made of Teflon, installed on over Head paper placed on the glass base to ensure that specimens were not stuck with the mold. The molds of specimens depended on [ASTM-D2240], as in figure (3-2).

2. For electrical specimens, used circular molds from plastic, thickness of 3 mm and diameter of 25 mm installed on a glass base by stabilizers and placed inside the mold over Head paper to ensure that specimens were not stuck with the mold. Specimens were prepared according to ASTM-D150. As shown in figure (3-3).

3. For thermal conductivity specimens, used circular plastic molds made of Teflon, thickness 4mm and diameter of 30mm installed on over Head paper placed on the glass base to ensure that specimens were not stuck with the mold. Specimens were prepared according to Lee's disc apparatus. As shown in figure (3-4).

4. Place the molds in a flat place to be ready for casting.
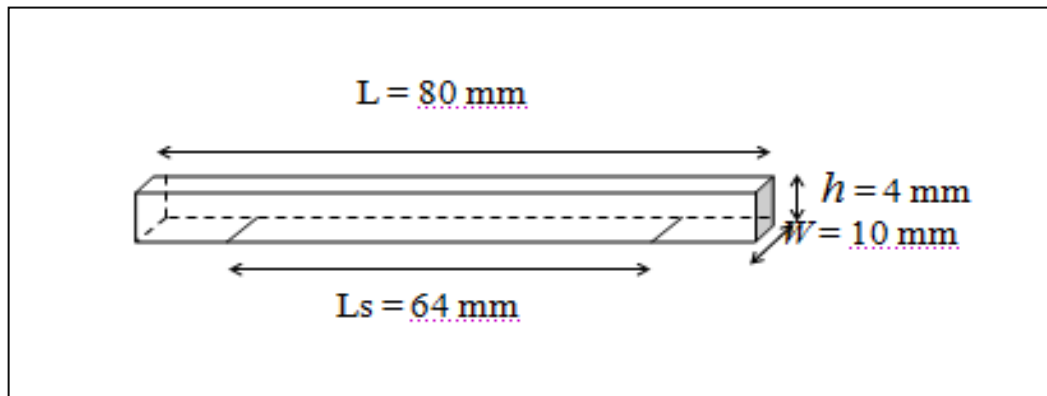
Figure (3-1): Dimension of Flexure Test Specimens.
[ASTM- D790]

Table (3-4): Recommended Dimensions for Test Specimen.

Nominal Specimen Depth, (h) mm	Specimen Width, (w) mm	Width Specimen Length, (L) mm	Length Support Span, (I) mm
1	25	50	16
2	25	50	32
3	25	60	48
4	10	80	64
5	10	100	80
6	10	125	96
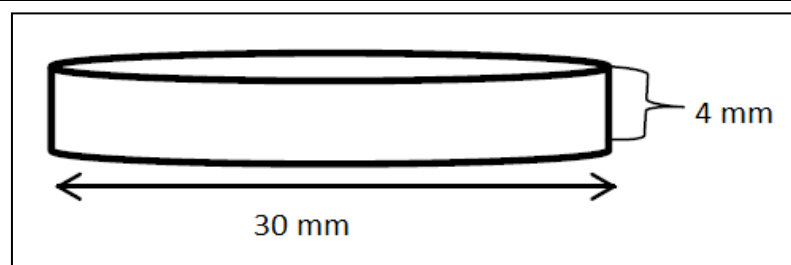
Figure (3-2): Dimension of Shore D Hardness Test Specimens.
[ASTM-D2240]
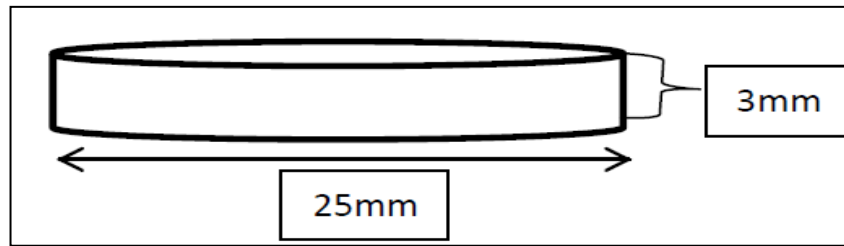
Figure (3-3): Dimension of Electrical Test Specimens.

[ASTM- D150-98]
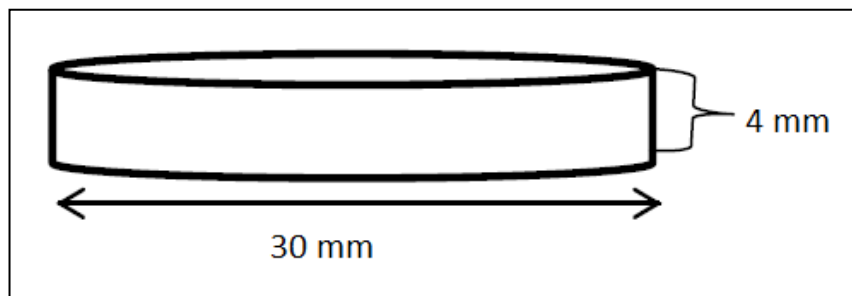
Figure (3-4): Dimension of thermal conductivity Test Specimens.

3-3-2 Epoxy Specimen Casting

As a matrix, blend was prepared by A clean disposable container was used for mixing epoxy and its hardener. The container put on a sensitive electronic balance of accuracy (0.0001 gm) with the required epoxy resin and hardener.

An exact amount of special hardener was added to the Epoxy in the container with ratio (5:2) the content was mixed thoroughly by homogenizer device at (700 rpm) for (10 min) to have the best homogeneous blend. Rotary vacuum pump producing vacuum less than (10^{-1} bar) was used to remove the bubbles from mixture of epoxy resin before casting, After that, casting mixture into the prepared molds previously, as explain at section 3-3-1, then left to solidify at room temperature for (24 hours). The samples are then released and softened. Samples were left for (28 days) before testing.

3-3-3 Composites Casting

Hand lay –up method were used to prepare specimens of Ep/Al and Ep/Cu composite. As reinforced materials, copper and aluminum powder were used to prepare for composites. The weight percentage of the powders (5%, 15%, 25%, 35%, and 45%), as showing in Table (3-5) and Table (3-6), the method of preparing the composite for each ratio is as follows:

For each weight percentage of the metal powders, the ratio of epoxy and hardener is prepared separately.

1. Metallic powder mixed with epoxy by a Glass Rode for (5 minutes) continuously and slowly and in one direction to avoid bubbles during mixing.
2. After step 2, hardener was added to the content and mix thoroughly by homogenizer device at (700 rpm) for (10 min) for well homogeneous of composite.
3. Rotary vacuum pump producing vacuum less than (10^{-1} bar) was used to remove the bubbles from mixture before using for mold casting.
4. Casting mixture into the prepared molds previously, as explain at section 3-3-1, then left to solidify at room temperature for (24 hours).
5. The samples are then released and softened. Samples were left for (28 days) before testing.

Table (3-5): Show weight percentage for Aluminum powder.

Ep (wt%)	100	95	85	75	65	55
Al (wt%)	0	5	15	25	35	45

Table (3-6): Show weight percentage for Copper powder.

Ep (wt%)	100	95	85	75	65	55
Cu (wt%)	0	5	15	25	35	45

3-4 Devices used:

3-4-1 Scanning Electron Microscope (SEM) device:

SEM Inspect s50 The device used in this study to measure the particle size for Copper and Aluminum powder.

The (SEM) uses a focused beam of high-energy electrons to generate a variety of signals at the surface of specimen.

Characteristic (X-Rays) are emitted when the electron beam removes an inner shell electron from the sample, causing a higher-energy electron to fill the shell and release energy. These characteristic (X-rays) are used to identify the composition and measure the abundance of elements in the sample.

The signals that derive from electron-sample interactions reveal information about the sample including external morphology, chemical composition, and crystalline structure and orientation of materials making up the sample, and composition a 2-dimensional image.

Available in Department of physics, College of science, Al-Nahrain University. Figure (3-5) show Photograph of Scanning Electron Microscope (SEM).

Figure (3-5): Photograph of SEM Inspect s50.

3-4-2 Atomic force microscope (AFM) device:

To measure the average diameter of Copper and Aluminum micro particles was used (AFM) device. In the AFM (Figure: 3-6), the sample is scanned by a tip, which is mounted to a cantilever spring. While scanning, the force between the tip and the sample is measured by monitoring the deflection of the cantilever. Scanning is dynamic process, because the tip is in mechanical contact with the specimen, attractive forces or force gradients between the tip and surface are sensed. A laser beam falls on the carrier, this laser beam rises and falls with High and low tip, and thus, with the variety of the topography of the surface of the rise and fall, the laser beam reflex are captured on the receiver. The topography of the surface are determined according to the movement of reflex laser beam.

Using (AA3000), 2008, USA, Available in chemistry Laboratory, Chemistry department, College of science, Baghdad University.
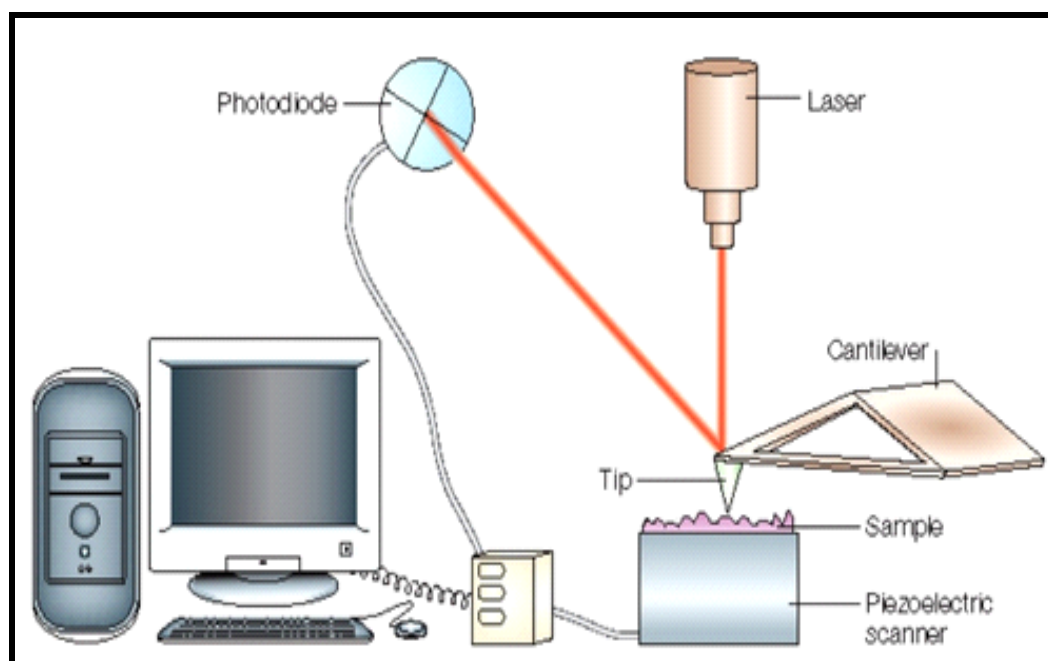
Figure (3-6): Block diagram of atomic force microscope.

3-4-3 Three Point Bending test device:

The loading technique for bending test is shown at Figure (3-7). According to ASTM standard D-790, the support span (distance between the supports) was depending on the specimen thickness as listed in Table 3-4. The load was applied on a specimen at the middle of support span. The load – deflection displacement Curves were recorded by the Instron. A typical set of load – deflection curves are shown in Figures (3-8, 9, 10).

The flexural test (Instron 1122), Available in materials laboratory, Department of physics, College of science, Baghdad University

Figure (3-7): Photograph of Instron (1122).
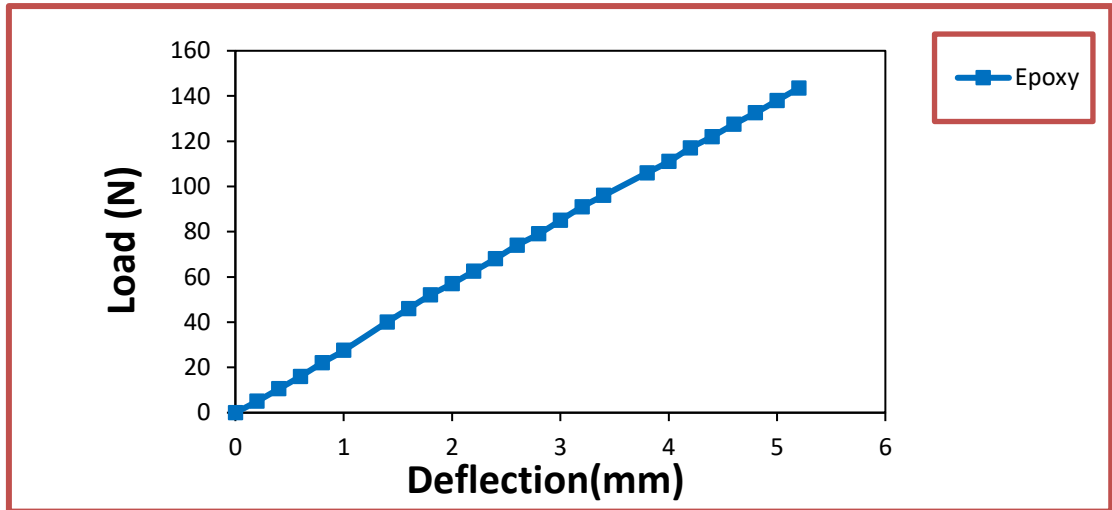
Figure (3-8): load – deflection curve for Epoxy.
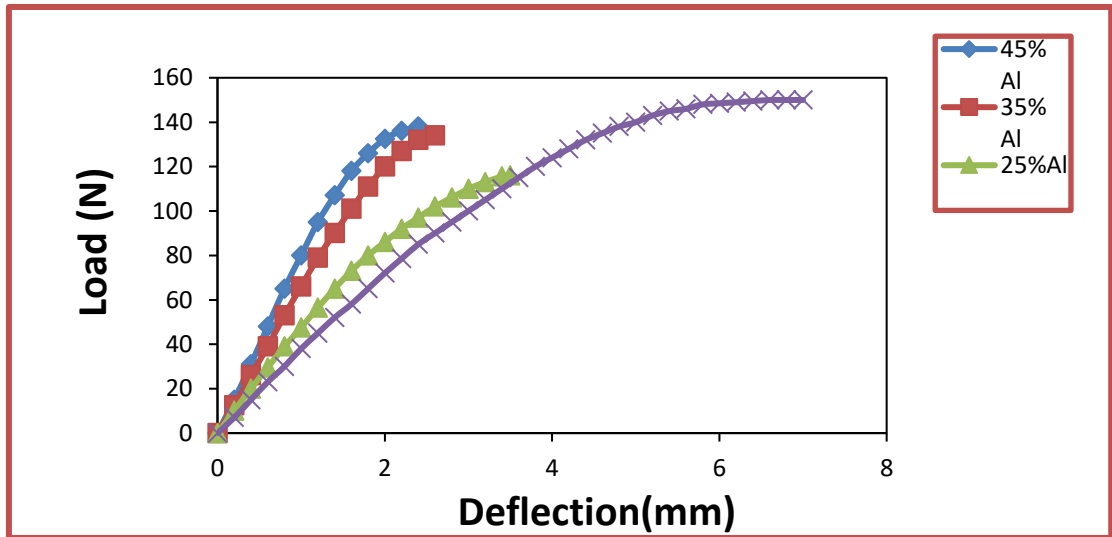
Figure (3-9): load – deflection curve for EP/Al.
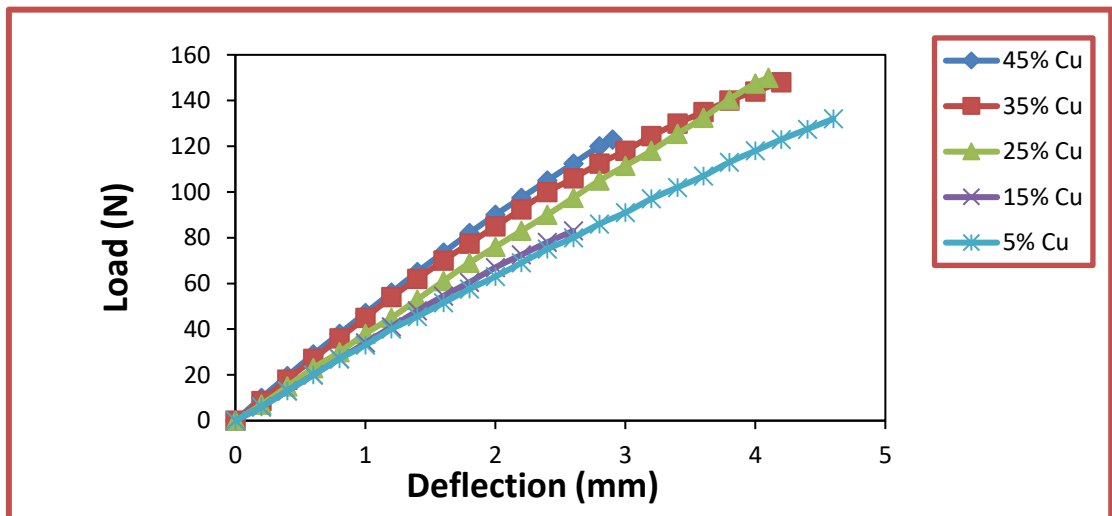
Figure (3-10): load – deflection curve for EP/Cu.

3-4-4 Hardness (Shore D) test device:

Shore D Hardness tester TH210 made by Time Group INC. Used to measure the hardness of the material, the indenter was attached to a digital scale that is graduated from (0 to 100) unit, the specimens were tested by pressing the indenter of the instrument, which is a needle of a sharp head (Length 2.54 mm) into the specimen surface so that the result was appeared on the digital screen attached with the instrument. Available in Applied Sciences Laboratory, Department of Applied Sciences, College of science, University of Technology. As shown in Figure (3-11).

Figure (3-11): Photograph of Shore D Hardness TH210.

3-4-5 Thermal conductivity (Lee's disc apparatus) device:

Lee's disc apparatus used to calculate thermal conductivity of materials. Which contain three discs of copper (A, B and C). The thickness of copper disc 13mm and diameter 30mm, Heating Coil is placed between disc B and C which is connected to a D.C Power Supply. The samples of Epoxy composites were placed between disc A and B. Upon arrival to the state of thermal equilibrium, Record the disk temperatures T_C, T_B, T_A. Thermal conductivity was calculated by relation (2-11).

$$K_{th}((T_B - T_A)/ds) = e\ [\ T_A + \frac{2}{r}(d_A + \frac{1}{4}ds)T_A + \frac{1}{2r}ds T_B\ ]$$

Made by **(Griffin and George),** England. Available in materials laboratory, Department of physics, College of science, Baghdad University. As shown in Figure (3-12).
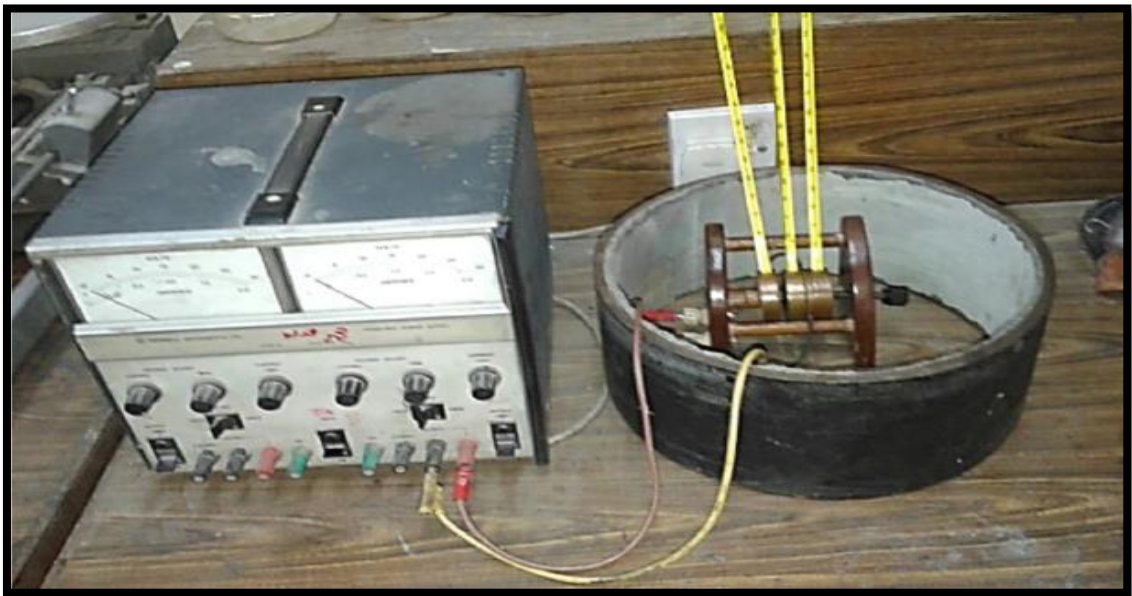
Figure (3-12)**:** Photograph of Lee's disc apparatus.

3-4-6 Differential Scanning Calorimeter (DSC) device:

Differential Scanning Calorimetry (DSC131 EVO) is one of the most popular methods that can be used to study the thermal properties, including the glass transition temperature (Tg) for samples as a function of temperature.

Measurements were performed over a temperature range of (25 – 200) °C at a heating rate of (10°C/min). DSC includes two Aluminum pans. The samples weight was about (10 mg). After placing the sample substance in the crucible and put the lid .The metal crucibles are closed using an appropriate crimping tool (Figure 3-13). The die (A) receives the crucible to crimp and holds it during the crimping operation. The punch (B) crimps the lid on the crucible. After the crimping operation check that the crimping is satisfactory. Check in particular that the lid is not squint. DSC131 EVO Production Company SETARAM, France. Available in materials laboratory, Department of physics, College of Education for pure Sciences/ Ibn Al Haitham. As shown in Figure (3-14).
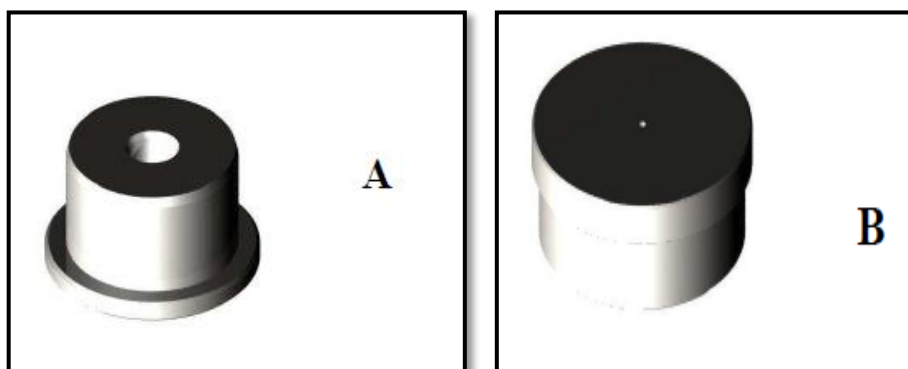
Figure (3-13): Crucible Crimping tool.

Figure (3-14): Photograph of DSC131 EVO.

3-4-7 Precision Impedance Analyzer device:

Precision Impedance Analyzer used for studying the A.C electrical properties may provide valuable information about the conduction mechanisms occurring in the specimens of Epoxy. It covering a frequency range from (40 Hz) to (110 MHz), The composites specimen was placed between two electrodes made of silver with diameters 10mm of a parallel plate capacitor. The measured conductance (σ), from (50 Hz) to (15 MHz) is used to calculate A.C. conductivity σa.c by using the relation (2-23).

$$\sigma = \frac{d}{R.A}$$

Model 4294A, Agilent, USA, Available in Department of Nano, the Ministry of Science and Technology. As shown in Figure (3-15).
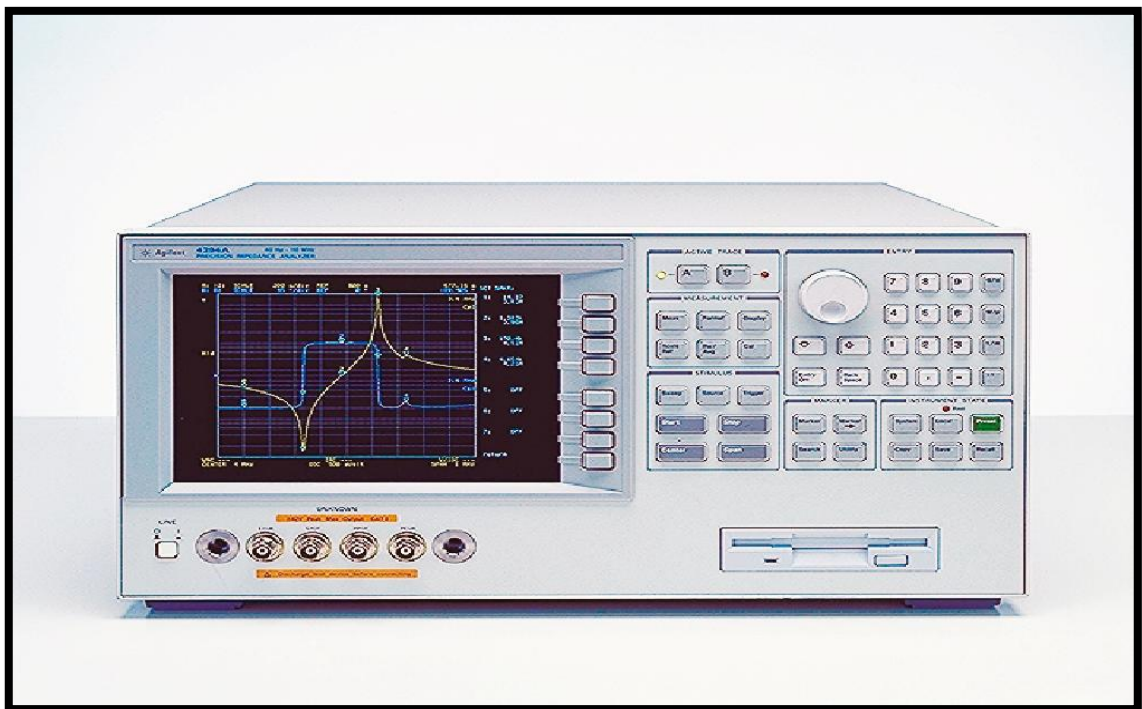
Figure (3-15): Photograph of Precision Impedance Analyzer 4294A.

CHAPTER FOUR

Results & Discussior
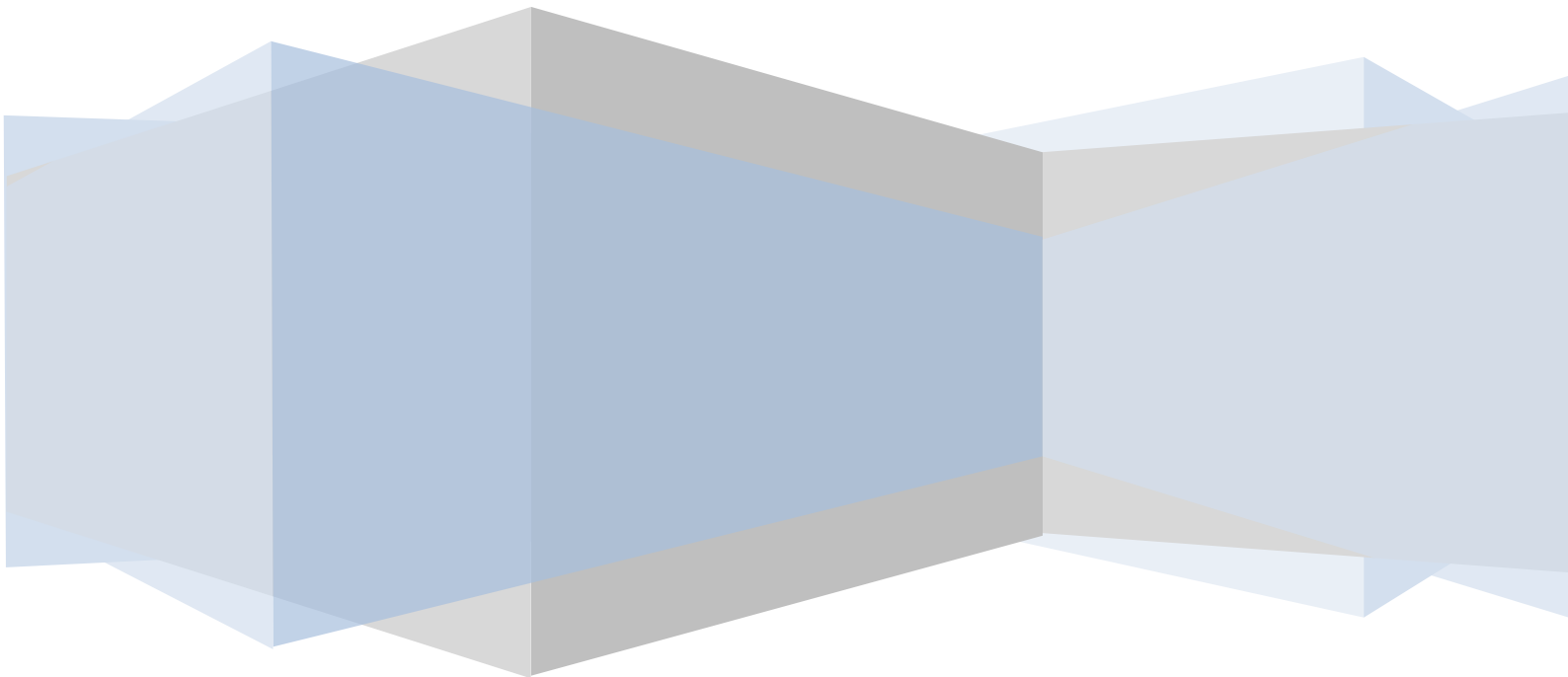
CHAPTER FOUR

Results & Discussion

4-1 Introduction

Epoxy resin was using a ratio (5:2) with hardener; the Aluminum and Copper powders were then added in proportions (5%, 15%, 25%, 35%, and 45%). Different shapes of samples were obtained, depending on ASTM for each test. This chapter includes five section, these parts are:

a. Diagnostics results and discussion for particle size (copper and aluminum powder) using SEM and AFM Techniques.

b. Results and discussion for Mechanical Properties.

c. Results and discussion for Thermal conductivity.

d. Results and discussion for Differential Scanning Calorimeter (DSC).

e. Results and discussion of electrical A.C. characteristics.

4-2 SEM and AFM Techniques:

SEM and AFM Techniques used to know Shapes and particle size for metal powders. Noticed the images of the Scanning Electron Microscope (SEM) with magnifying force between (569x-23171x) by select between (200µm- 5 µm), differences in particle sizes for Aluminum powder, Where it is observed that there are clusters of particles and that the form of particles semi-spherical and oval. However, the measurement of particle size for each grouping was difficult. As Shown in Figure (4-1). Therefore, the particle size and the percentage of its presence in the sample were measured through a test (AFM). It was performed to measure the average diameter for Aluminum powder. Table (4.1) and Figure

(4-2) showed the results for Aluminum powder with average diameter (305.33 nm).

While, Showed the images of the (SEM), differences in particle sizes for Copper powder, Where it is observed that there are clusters of particles and that the form of particles dendritic structure. However, the measurement of particle size for each grouping was difficult. As Shown in Figure (4-3).

Therefore, the particle size and the percentage of its presence in the sample were measured through a test (AFM). It was performed to measure the average diameter for Copper powder. Table (4.2) and Figure (4-4) showed the results for Copper powder with average diameter (240.91 nm).
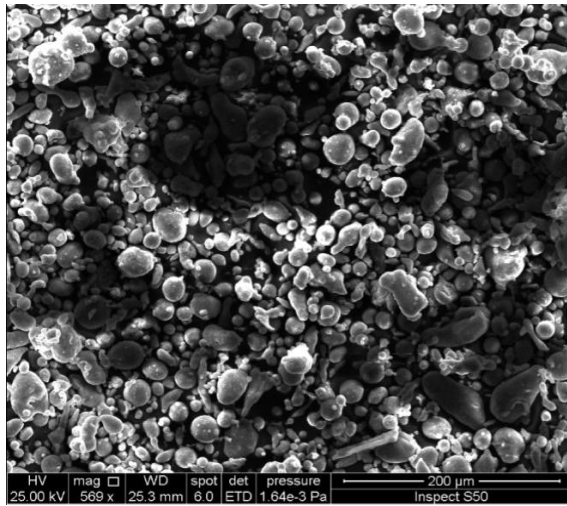
200µm
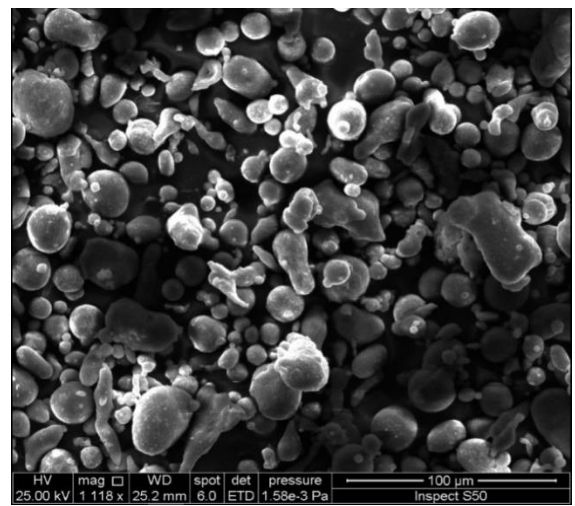
100 µm
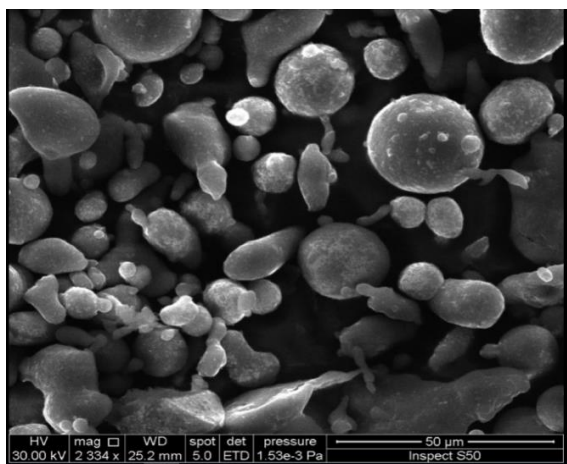
50µm
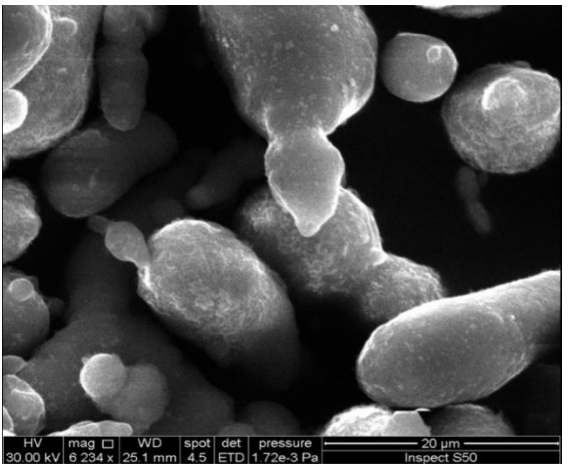
20µm
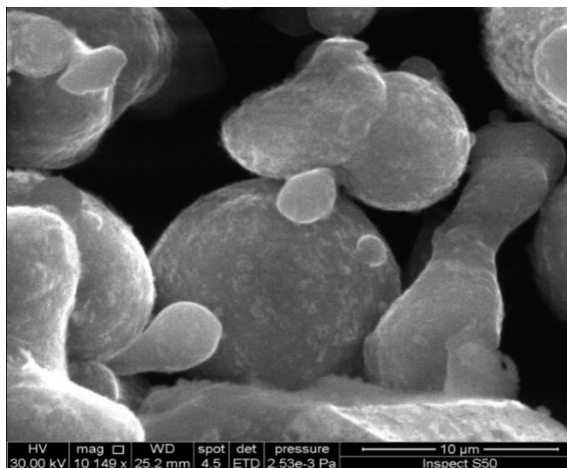
10µm
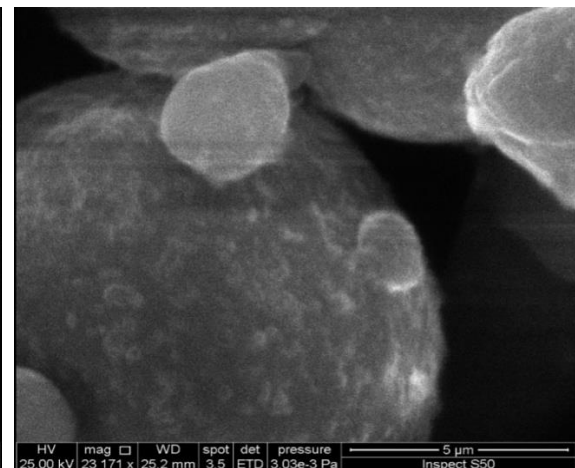
5µm

Figure (4-1): Shoued the SEM images of the Aluminum particles.

Table (4.1): Partical size Distribution and average diameter of Aluminum powder

Avg. Diameter:305.33 nm		<=10% Diameter:220.00 nm	
<=50% Diameter:280.00 nm		<=90% Diameter:420.00 nm	
Diameter (nm) <		Volume (%)	Cumulation (%)
220		3.97	3.97
240		11.11	15.08
260		18.25	33.33
280		12.70	46.03
300		11.90	57.94
320		7.14	65.08
340		9.52	74.60
360		4.76	79.37
380		5.56	84.92
400		2.38	87.30
420		1.59	88.89
440		2.38	91.27
460		4.76	96.03
480		2.38	98.41
500		1.59	100.00
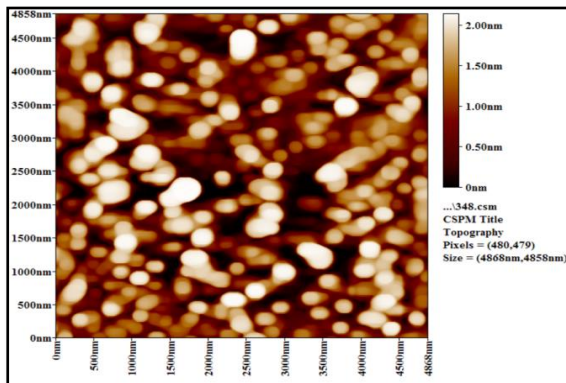
(a)
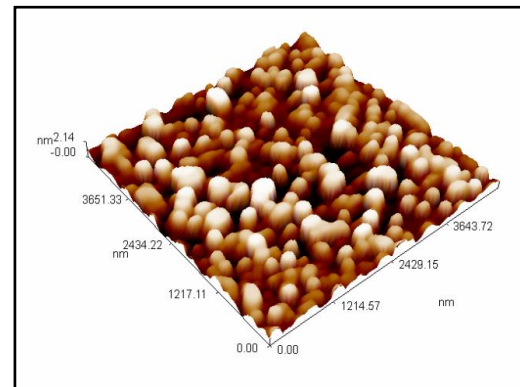
(b)
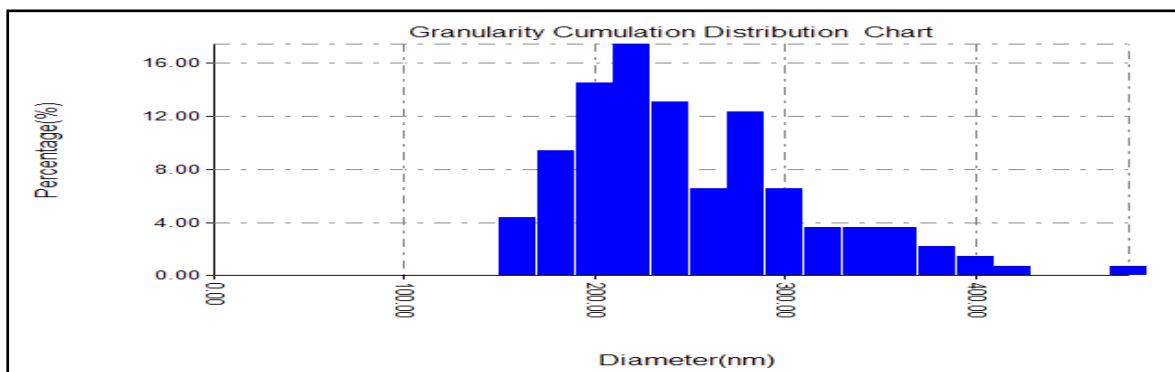
(c)

Figure (4-2): AFM image of Al powder and represents (a) 2D view of AFM image, (b) 3D view of AFM image, (c) Granularity cumulation chart for Al.
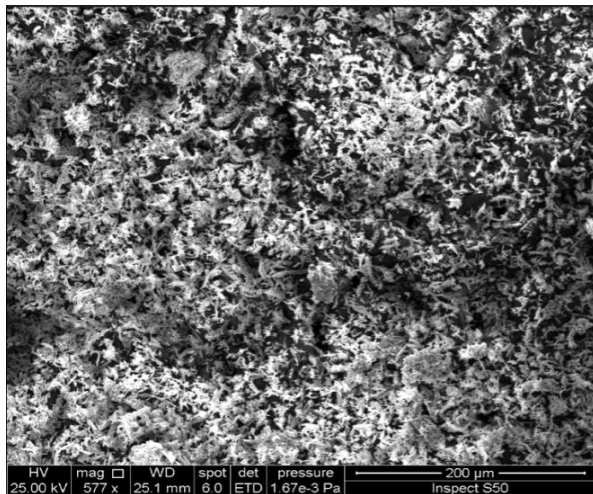
200µm   100 µm
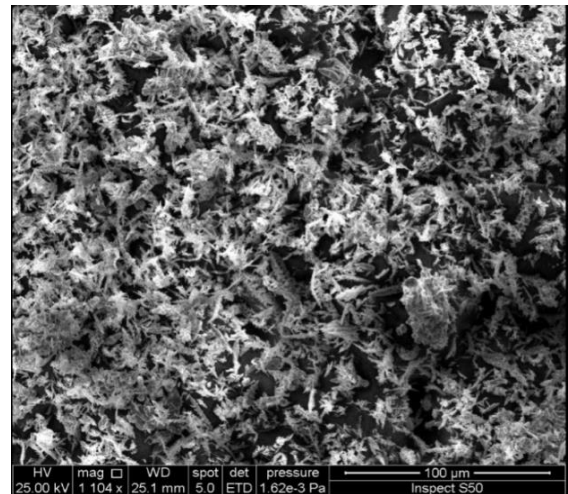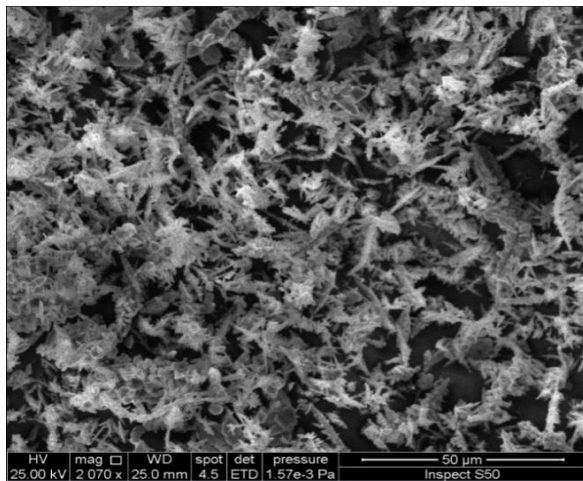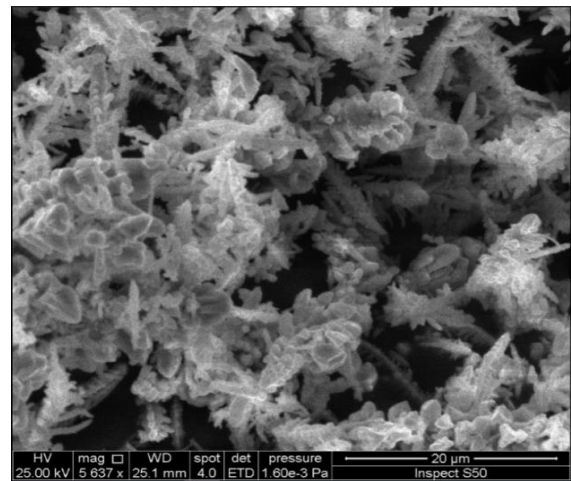
50µm   20µm
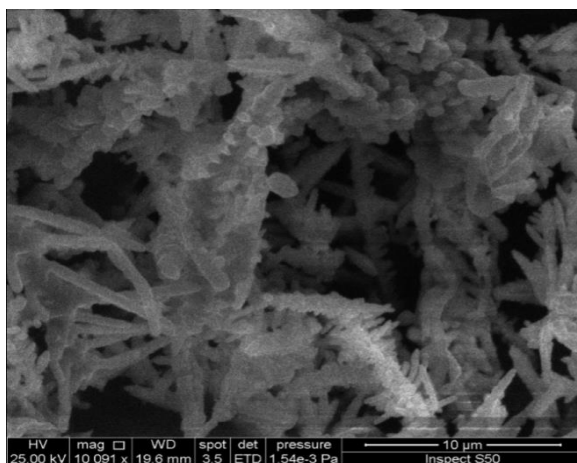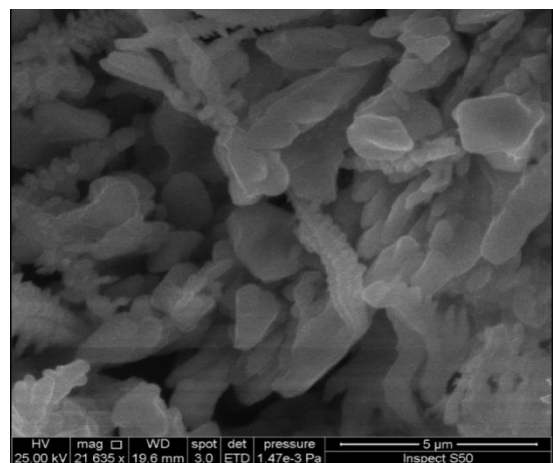
10µm   5µm

Figure (4-3): Shoued the SEM images of the Copper particles.

Table (4.2): Partical size Distribution and average diameter of Copper powder

Avg. Diameter:240.91 nm		<=10% Diameter:160.00 nm
<=50% Diameter:220.00 nm		<=90% Diameter:320.00 nm
Diameter (nm)<	Volume (%)	Cumulation (%)
160	4.35	4.35
180	9.42	13.77
200	14.49	28.26
220	17.39	45.65
240	13.04	58.70
260	6.52	65.22
280	12.32	77.54
300	6.52	84.06
320	3.62	87.68
340	3.62	91.30
360	3.62	94.93
380	2.17	97.10
400	1.45	98.55
420	0.72	99.28
480	0.72	100.00
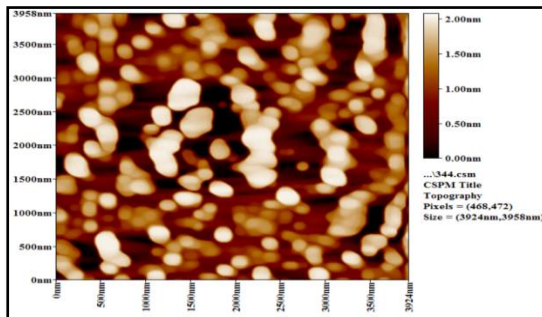
(a)
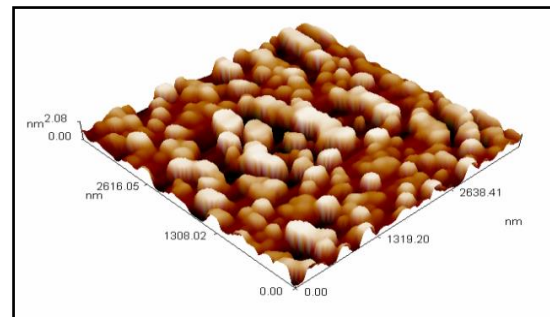
(b)
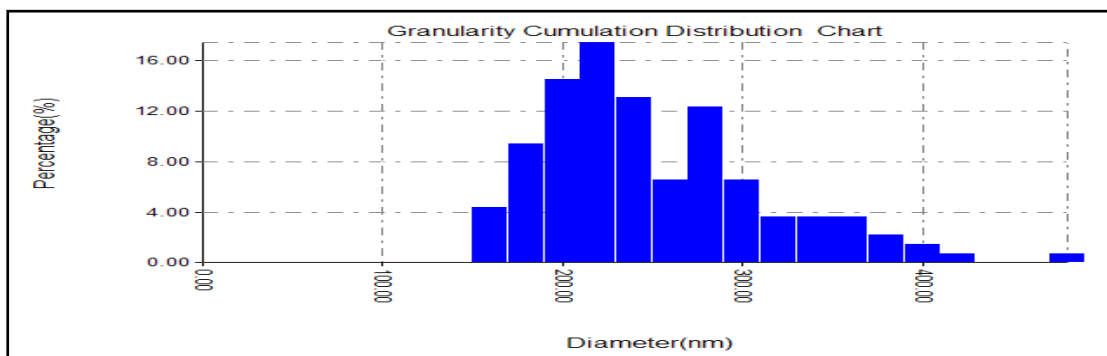
(c)

Figure (4-4): AFM image of Cu powder and represents (a) 2D view of AFM image, (b) 3D view of AFM image, (c) Granularity cumulation chart for Cu.

4-3 The Mechanical Properties

4-3-1 Bending Test

Bending stresses are important in structure tests because of variety of loading situations in service. It determines the behavior and properties of the structure. Many parameters should be concerned test data. Polymeric composites are susceptible to mechanical damages when they are subjected to efforts of tension, flexural, compression which can leads to material failure [85].The mechanical properties of the materials are affected by many factors including: particles type, particle size, volume fraction, and specimen thickness [86].

4-3-1-1 Bending Test of Epoxy Specimen

In bending test, the specimen effects by two mode of test. The convex side of the specimen is extended and the concave side is compressed with an unstrained neutral axis through the center (section 2-12-2). According to the experimental work (section 3-4-3), the load –deflection curve were convert to bending stress - deflection curve as seen in Figures (4-5), using equation(2-4). Also the modulus of elasticity was calculated using equation (2-5).

Tables (4.3) listed the values of flexural strength, modulus of elasticity, maximum fracture force and maximum deflection of Epoxy. Figure (4-5), show the bending stress - deflection curve for pure Epoxy. It was observed that the relationship was linear; increased deflection with increased stress, this behavior is agreement with reference [21, 24, and 87]. To explain this behavior, epoxy is the thermoset polymer, which has backbone chains, and because of the Epoxy has cross-linking between the backbone chains, the movement of these chains needed force to bending the specimen. So that if the specimen is more bended the backbone chains will resist and the stresses will increase [88].

Thus no yield point due to brittleness of Epoxy specimen. Stress constantly from top to bottom expanding craze to lead to crack starting from the bottom

surface of the sample and spread toward the upper surface of the sample, leading to the break as seen in. The reason is due to the fact that the polymeric chains consisting of the sample are intertwined, when you shed the stress on the sample will lead to the occurrence of landslides in the polymeric chains and move those chains in various directions until the occurrence of break-up [21][89].

Figure (4-5): Bending stress -deflection curve for pure epoxy.

Table (4.3): Values of the modulus of elasticity, maximum fracture force, maximum deflection and flexural strength of Epoxy.

Materials	E(MPa)	Maximum Fracture Force (N)	Maximum Deflection (mm)	Maximum Ɠf (MPa)
Epoxy	2.7880448	143.5	5.2	86.1

4-3-1-2 Bending Test for Epoxy/ Aluminum composites

During the flexural test for composites, there are three factors dominate the resulting flexural strength of a specimen; the flexural strength of the matrix, the adhesion between fillers and matrix and the adhesion between the Particles and the characterization of filling materials. This means that, the last two factors are effect on the original strength of the matrix, and these two factors would lead to distribute the applied force on the cross-sectional area of the composite

under test. According to the experimental work (section 3-4-3), the load – deflection curves were convert to bending stress - deflection curves as seen in Figure (4-6), using equation(2-4). Also the modulus of elasticity was calculat using equation (2-5).

Table (4.4): listed the values of flexural strength, modulus of elasticity, maximum fracture force and maximum deflection of Epoxy/ Aluminum composites. Observed form Figure (4-6), the behavior Epoxy/ Aluminum composites are different according to the weight percentage of filler, the curves include two regions, **linear** it's called elastic region and be under yield point, The linear region of the curves, shows that the applied stress on the specimen is distributed on the backbone of the polymer, because of the Epoxy has cross linking between the backbone chain restricts the movement of these chains under bending stresses [88]. **Non linear** it's called plastic region, the specimen is deformed, because of the concentration of the stresses at the lower region of the specimen (the convex side) where the specimen is extended, the stress will be constricted at the ends of crazes, which is grown to form the micro cracks, these micro cracks will be accumulated together to form the main crack, which pass through the specimen until the fracture occurs [42]. This behavior is agreement with reference [20].

Add aluminum powder particles which consists of different sizes to epoxy resins and mix them, these particles will spread between polymer cross linking chains, this spread will disrupt the process of moving the chains when the stresses on them. Was observed when ratios increased as shown in the Table (4.4) and Figure (4-7) that Young modulus Increases with increasing weight ratios. So the difference of the values of maximum fracture due to the presence of particles leads to the formation of the interphase region, the interphase region will allow for cracks to spread faster when they are present in a coherent manner.

Using equation (2-6),The bending stress - deflection curves were convert to Flexural stress –strain curves as seen in Figure (4-8), to calculate the Yield strength σy for composites, yield is obtained by drawing a line parallel to the initial portion of the Flexural stress –strain curve starting at strain (0.2%). A typical curve shown in Figure (4-9), the intersection line with the curve gives σ$_y$, The Yield Points for all Aluminum percentage increasing with increasing ratio fillers as seen in Table (4.5) and Figure (4-10).
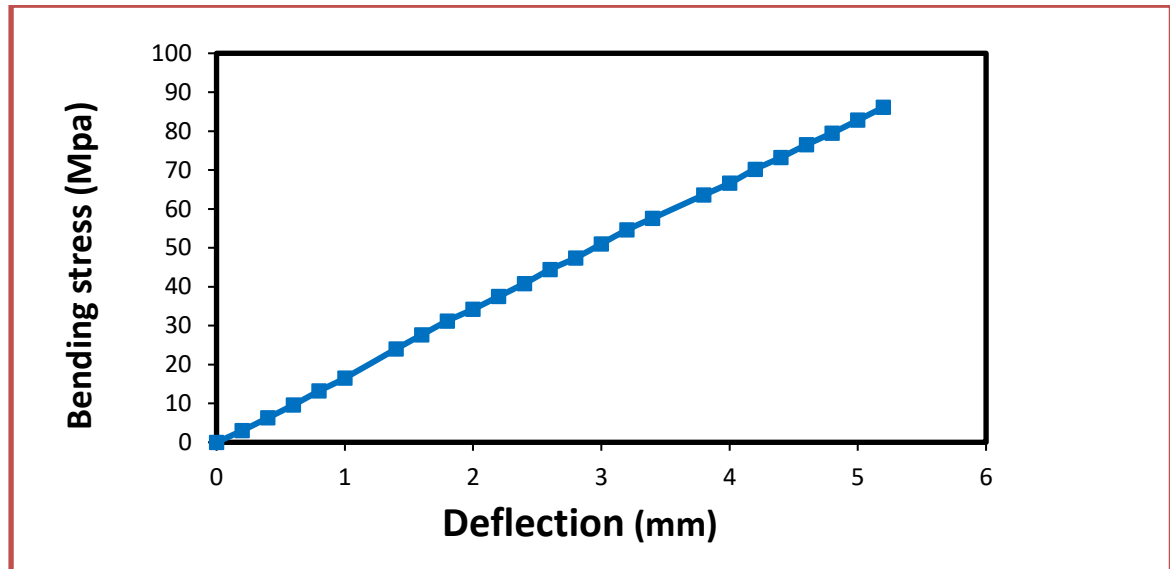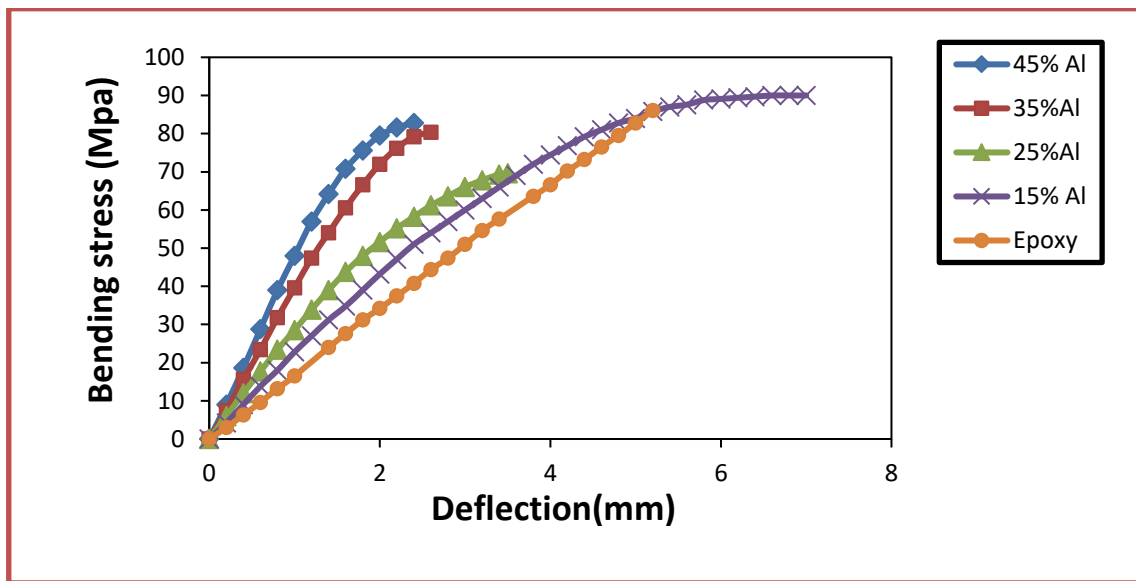

Figure (4-6): Bending stress – deflection for Ep/ Al composites.

Table (4.4): Values of the modulus of elasticity, maximum fracture force, maximum deflection and flexural strength of Epoxy/ Aluminum composites

Samples	E(MPa)	Maximum Fracture Force (N)	Maximum Deflection (mm)	Maximum Ꝺf (MPa)
100% EP	2.7880448	143.5	5.2	86.1
85% EP/ 15% Al	3.8596608	150	7	90
75% EP/ 25%Al	4.9005568	116	3.5	69.6
65% EP/ 35% Al	6.7376128	134	2.6	80.4
55%EP/ 45% Al	8.16384	138	2.4	82.8
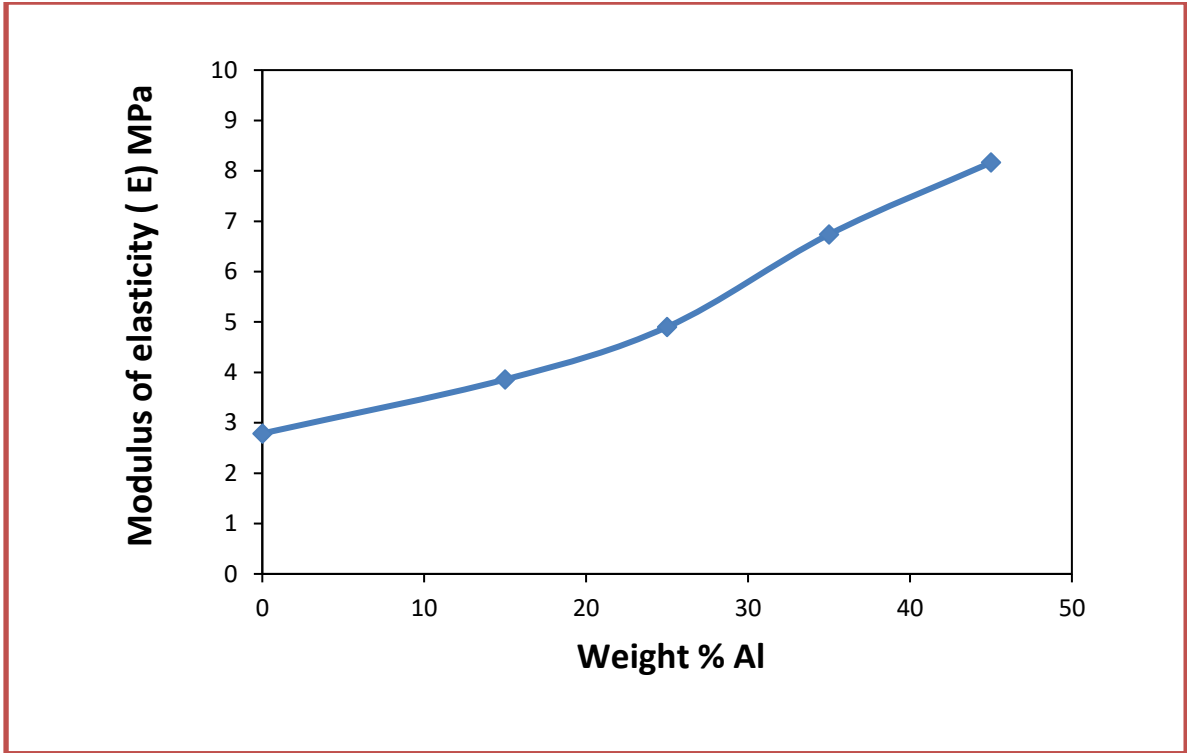
Figure (4-7): Modulus of elasticity Vs. Al weight percentage for Ep/ Al composites.
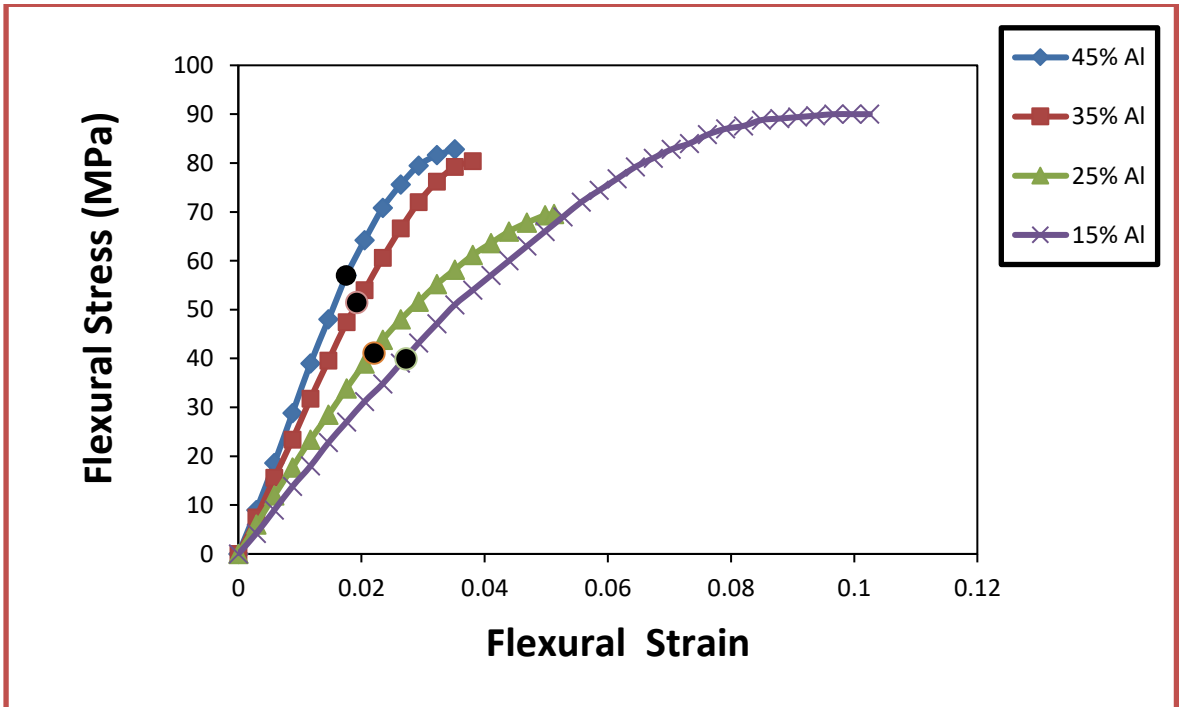
Figure (4-8): Flexural stress –strain for Epoxy/ Aluminum composites, ● Refers to yield point.
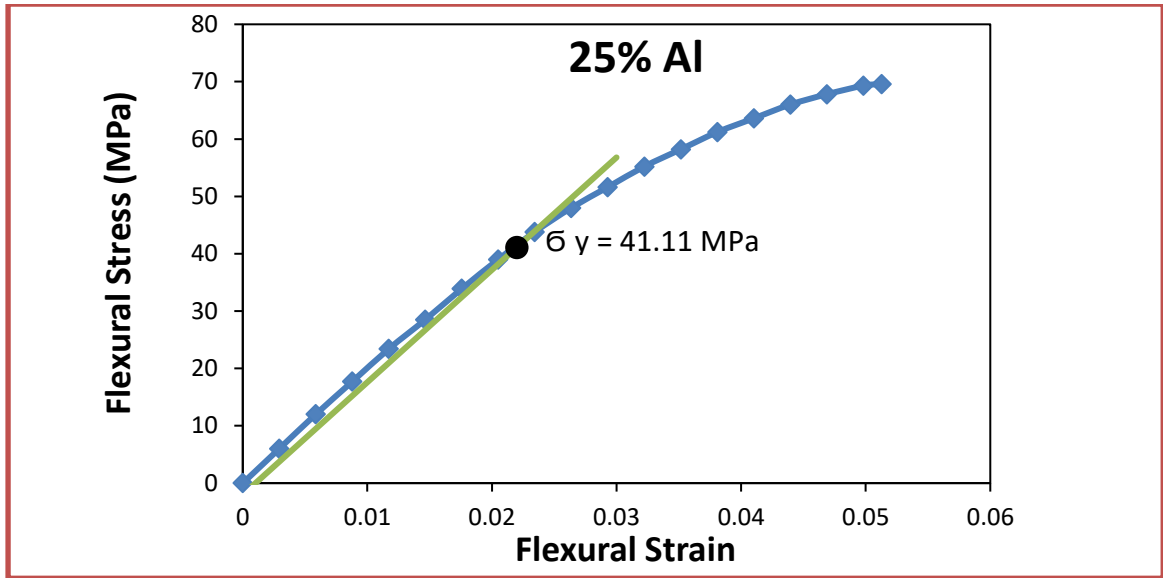
Figure (4-9): Flexural stress –strain for 75%EP/ 25%Al composite to determine the Yield point.

Table (4.5): Values of the Yield point of Epoxy/ Aluminum composites.

Samples	Yield Point Бy (MPa)
85% EP/ 15% Al	39.95
75% EP/ 25%Al	41.11
65% EP/ 35% Al	51.5
55%EP/ 45% Al	57.0
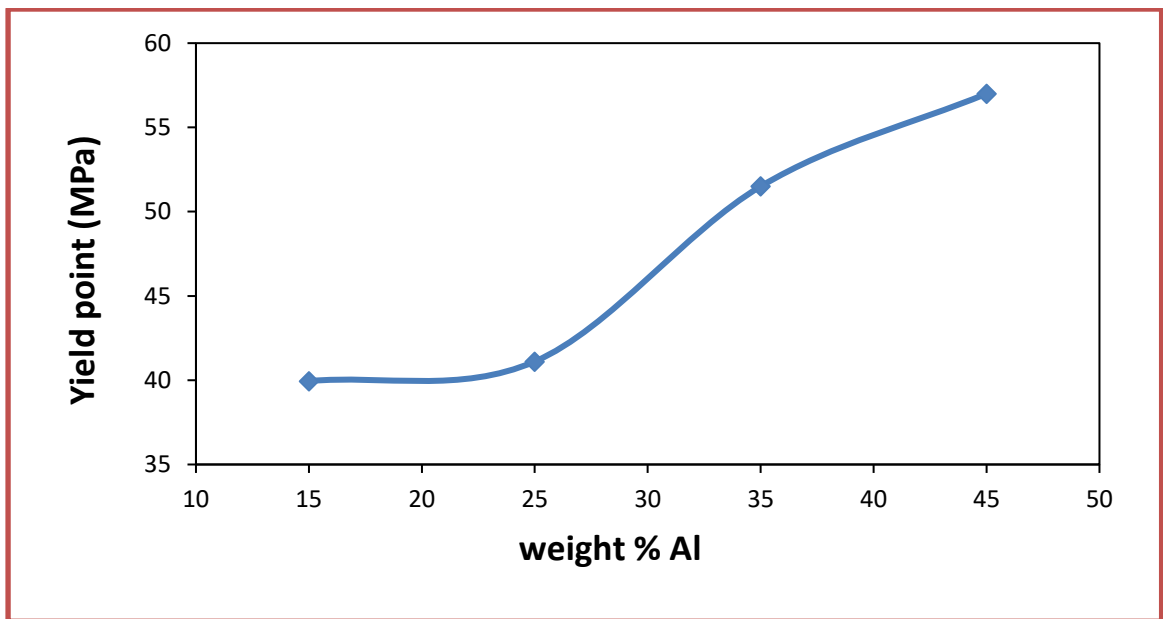
Figure (4-10): Yield point V.s Al weight percentage for Ep/Al composites.

4-3-1-3 Bending Test for Epoxy/ Copper composites

During the flexural test for composites, there are three factors dominate the resulting flexural strength of a specimen; the flexural strength of the matrix, the adhesion between fillers and matrix and the adhesion between the Particles and the characterization of filling materials. This means that, the last two factors are effect on the original strength of the matrix, and these two factors would lead to distribute the applied force on the cross-sectional area of the composite under test. According to the experimental work (section 3-4-3), the load – deflection curves were convert to bending stress - deflection curves as seen in Figure (4-11), using equation(2-4). Also the modulus of elasticity was calculated using equation (2-5).

Table (4.6): listed the values of flexural strength, modulus of elasticity, maximum fracture force and maximum deflection of Epoxy/ Copper composites. Observed form Figure (4-11), the behavior Epoxy/ Copper composites are different according to the weight percentage of filler, the curves include two regions, **linear** it's called elastic region and be under yield point, The linear region of the curves, shows that the applied stress on the specimen is distributed on the backbone of the polymer, because of the Epoxy has cross linking between the backbone chain restricts the movement of these chains under bending stresses [88]. **Non linear** it's called plastic region, the specimen is deformed, because of the concentration of the stresses at the lower region of the specimen (the convex side) where the specimen is extended, the stress will be constricted at the ends of crazes, which is grown to form the micro cracks, these micro cracks will be accumulated together to form the main crack, which pass through the specimen until the fracture occurs [42]. This behavior is agreement with reference [89 and 90].

Add Copper powder particles which consists of particles dendritic structure to epoxy resins and mix them, these particles will spread between polymer cross

linking chains, this spread will disrupt the process of moving the chains when the stresses on them. Was observed when ratios increased as shown in the Table (4.7) and Figure (4-12) that Young modulus Increases with increasing weight ratios. Either the difference the values maximum for fracture this is because the presence of particles leads to the formation of the interphase region, the interphase region will allow for cracks to spread faster when they are present in a coherent manner.

Using equation (2-6),The bending stress - deflection curves were convert to Flexural stress –strain curves as seen in Figure (4-13), to calculate the Yield strength σy for composites, yield is obtained by drawing a line parallel to the initial portion of the Flexural stress –strain curve starting at strain 0.2%. The intersection line with the curve gives σ_y, The Yield Points for all Copper percentage increasing with increasing ratio fillers as seen in Table (4.7) and Figure (4-14).
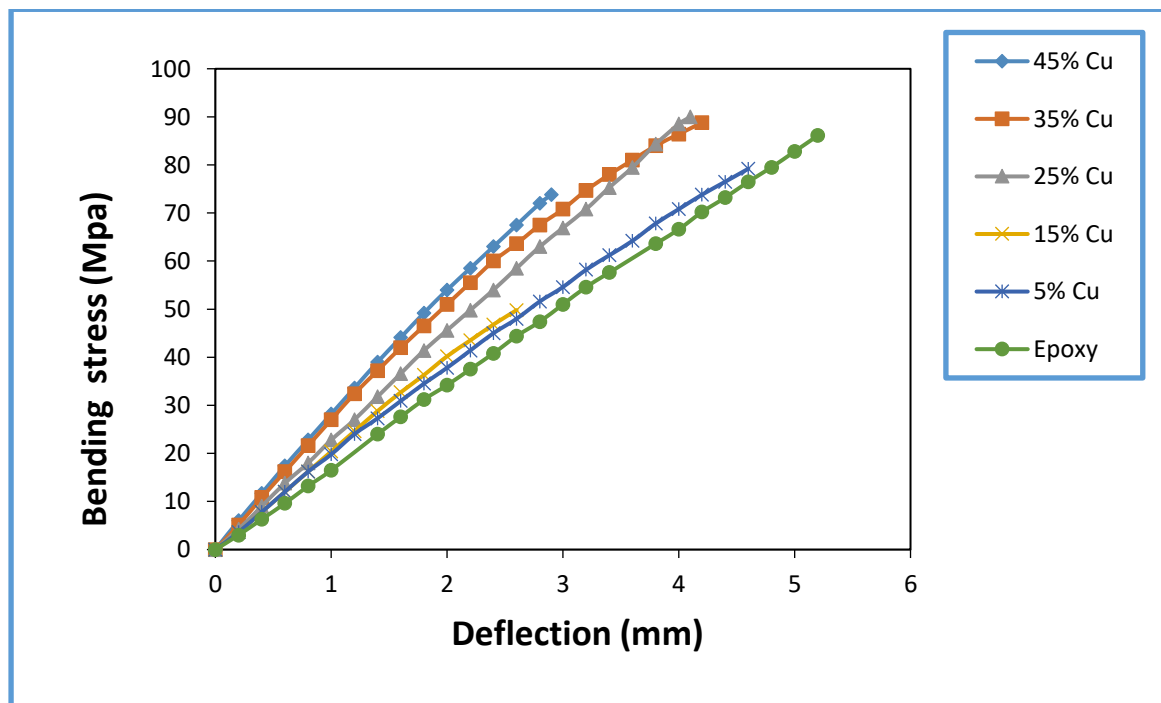
Figure (4-11): Bending stress – deflection for Ep/ Cu composites.

Table (4.6): Values of the modulus of elasticity, maximum fracture force, maximum deflection and flexural strength of Epoxy/ Copper composites

Samples	E(MPa)	Maximum Fracture Force (N)	Maximum Deflection (mm)	Maximum Ɠf (MPa)
100% EP	2.7880448	143.5	5.2	86.1
95% EP/ 5% Cu	3.4039808	131	4.6	79.2
85% EP/ 15% Cu	3.4658304	82	2.6	49.8
75% EP/ 25% Cu	3.8596608	150	4.1	90
65% EP/ 35% Cu	4.6052352	148	4.2	88.8
55% EP/ 45% Cu	4.8330752	123	2.9	73.8
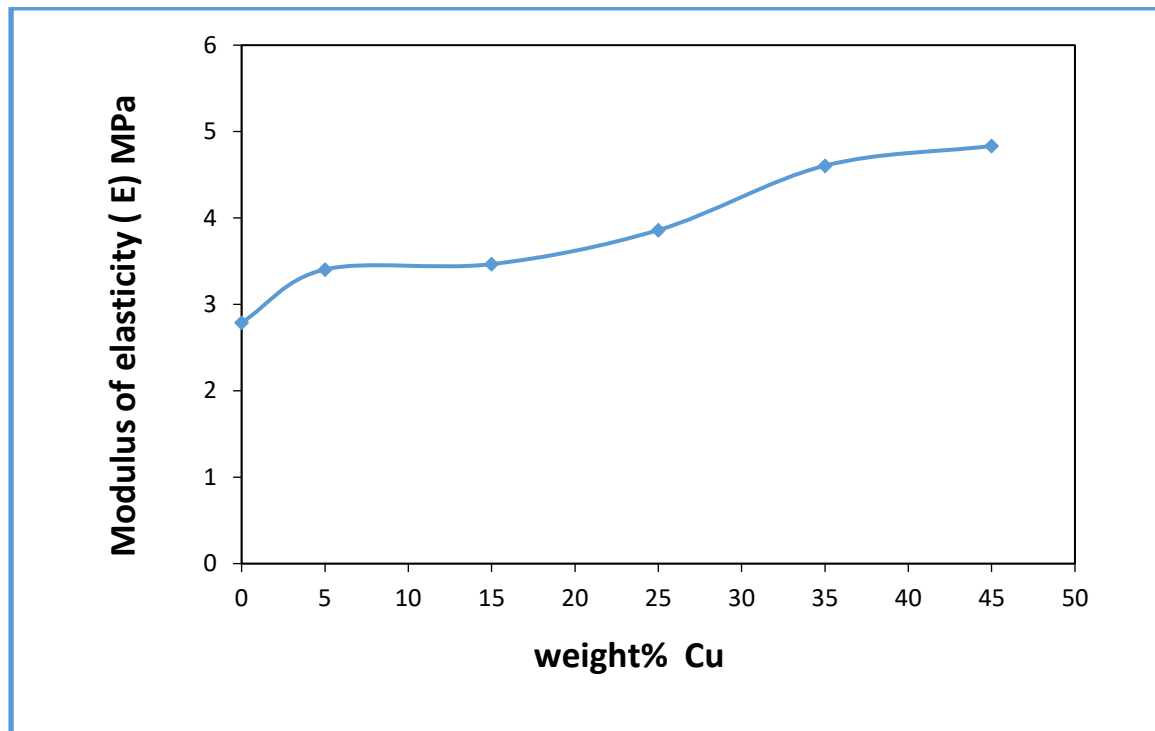
Figure (4-12): Modulus of elasticity Vs. Cu weight percentage for Ep/ Cu composites.
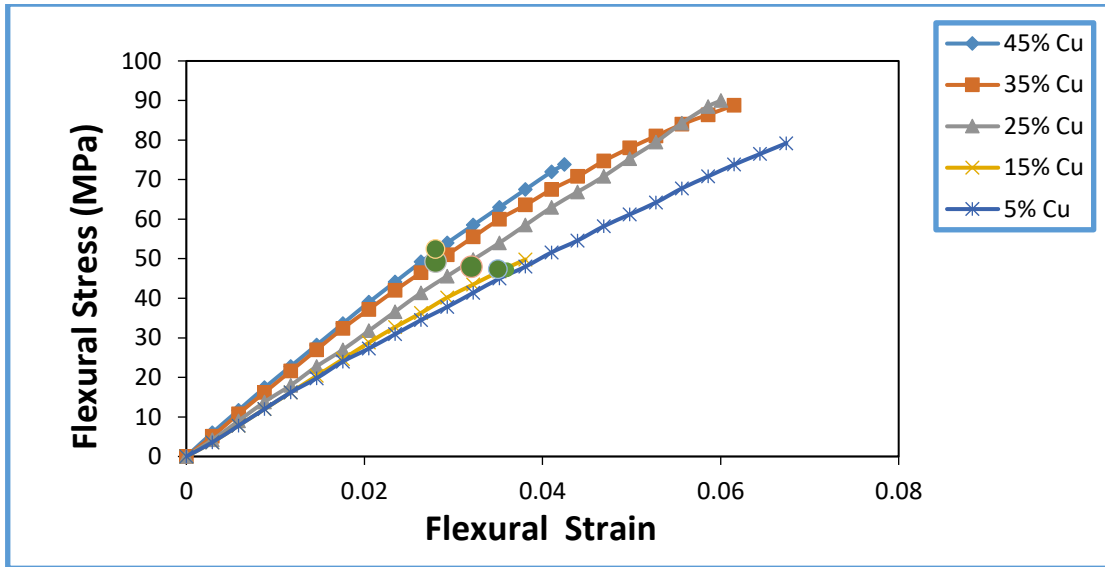
Figure (4-13): Flexural stress – Flexural strain for Epoxy/ Copper composites, ⬤ Refers to yield point.

Table (4.7): Values of the Yield point of Epoxy/ Copper composites.

Samples	Yield Point Ϭy (MPa)
95% EP/ 5% Cu	47.1826
85% EP/ 15% Cu	47.4
75% EP/ 25% Cu	48
65% EP/ 35% Cu	49.3088
55% EP/ 45% Cu	52.4872
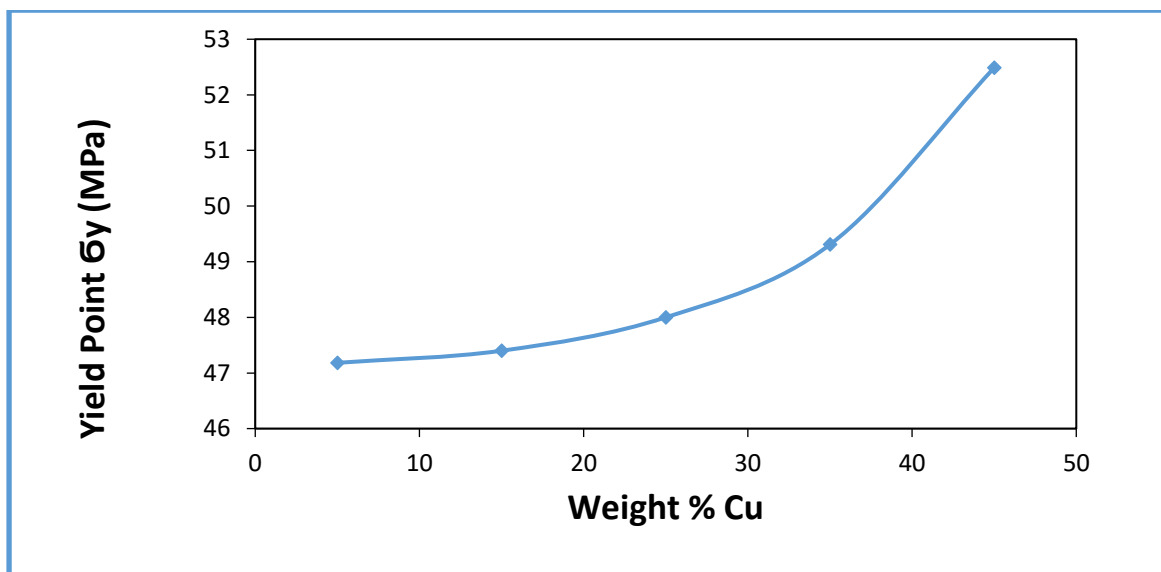
Figure (4-14): Yield point V.s Cu weight percentage for Ep/ Cu composites.

4-3-2 Hardness Test

4-3-2-1 Hardness Test for Epoxy/ Aluminum composites

Figure (4-15) and Table (4-8) shows the variation values for Shore D Hardness of Epoxy resin and its composites. It has been found that the hardness values increase with increasing of Aluminum powder ratio in the Epoxy composites, it was observed that there was an increasing in hardness values as Aluminum particles increase in Epoxy matrix until reach to (15%) Al and then become semi- stable as the Aluminum particles increase in the Epoxy composite, This behavior is agreement with reference [20], to explain this behavior, at low ratio, the particles of Al will distribute between the chain for epoxy, so that the indenter of device will touch the surface of specimen which contain deferent and small sizes of particles beside of epoxy. The increase Al powder, the Al particles will lined and clustered in all the volume of specimen, so that the indenter of device will touch the surface of clustered Al at the surface and the values of hardness will increase.
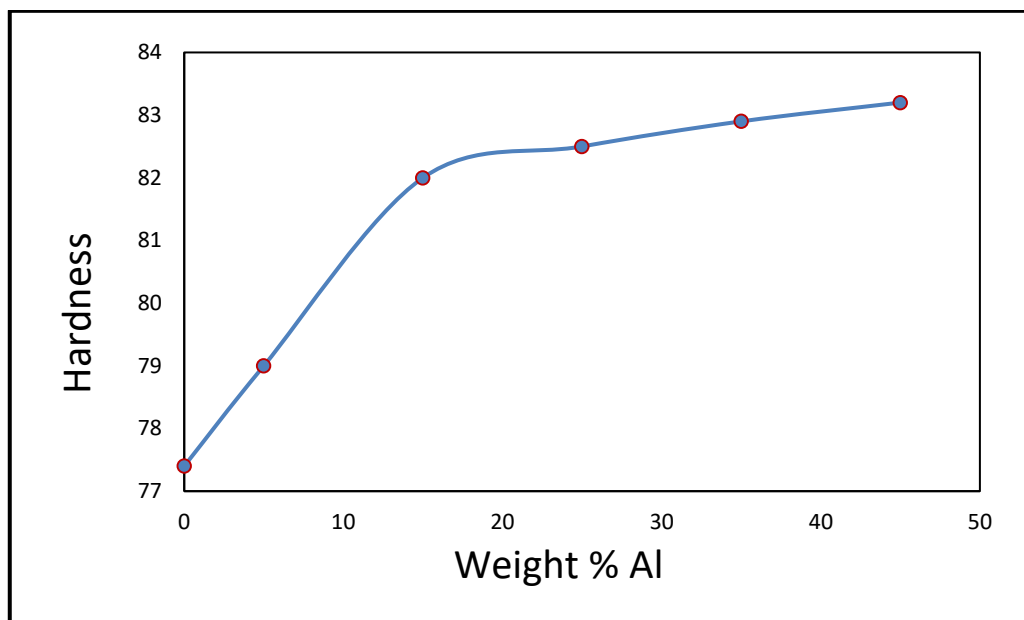
Figure (4-15): Hardness V.s Al weight percentage for Ep/ Al composites.

Table (4-8): Values of the Shore D Hardness for EP/ Al composites.

Samples	Shore D Hardness
100% EP	77.4
95% EP/ 5% Al	79
85% EP/ 15% Al	82
75% EP/ 25% Al	82.5
65% EP/ 35% Al	82.9
55% EP/ 45% Al	83.2

4-3-2-2 Hardness Test for Epoxy/ Copper composites

Figure (4-16) and Table (4-9) shows the variation values for Shore D Hardness of Epoxy resin and its composites. It has been found that the hardness values increase with the increasing of Copper powder ratio in the Epoxy composites, as from this figure it was observed that there was an increasing in hardness values as Copper particles increase in Epoxy matrix. This behavior is agreement with reference [20], to explain this behavior, the particles of Copper will distribute between the chains for epoxy, so that the indenter of device will touch the surface of specimen which contain deferent and small sizes of particles beside of epoxy. The increase Cu powder, the Cu particles will lined and clustered in all the volume of specimen, so that the indenter of device will touch the surface of clustered Cu at the surface and the values of hardness will increase.
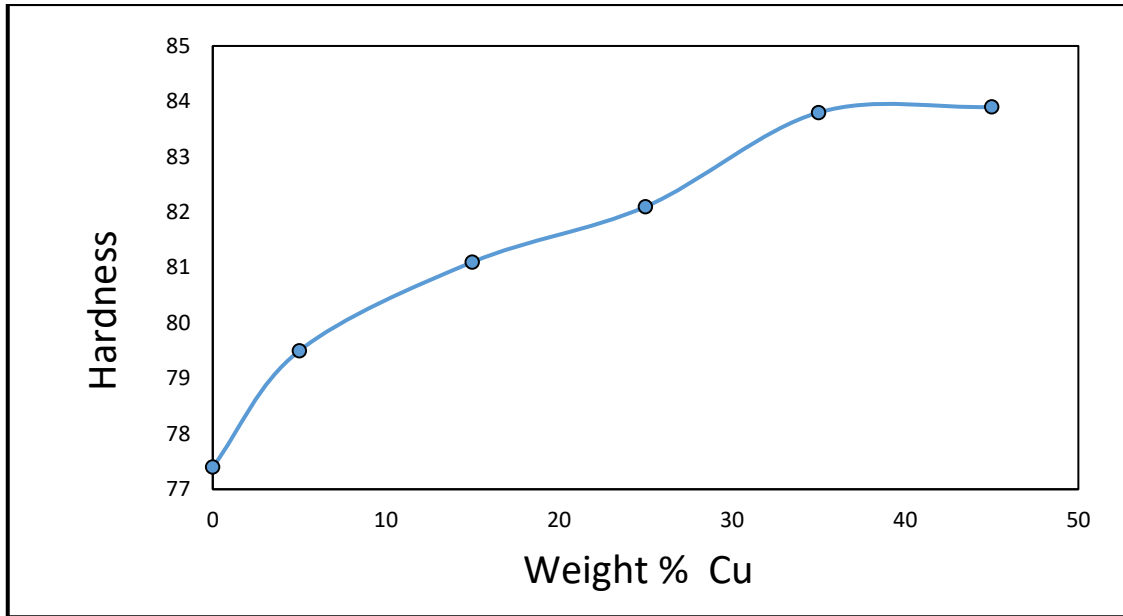
Figure (4-16): Hardness V.s Cu weight percentage for Ep/ Cu composites.

Table (4-9): Values of the Shore D Hardness for EP/ Cu composites.

Samples	Shore D Hardness
100% EP	77.4
95% EP/ 5% Cu	79.5
85% EP/ 15% Cu	81.1
75% EP/ 25% Cu	82.1
65% EP/ 35% Cu	83.8
55% EP/ 45% Cu	83.9

4-4 Thermal conductivity

4-4-1 Thermal conductivity for Epoxy/ Aluminum composites

Thermal conductivity of Epoxy resin and EP/Al composites are shown in the Figure (4-17). The results appear that Thermal conductivity (k_{th}) values increased with increasing wt% of reinforcement, the max value (1.459549 W/m. K) at (45%) for EP/Al composites as shown in Table (4-10), this table includes the weight percentage of composite and the thermal conductivity of the

composite. It is clear that (k_{th}) for EP/Al composites increase with increasing wt% of Al particles, that there is no interaction between them. This behavior is agreement with reference [13- 16-17 and 92].

Aluminum has thermal conductivity and its value (247 W/m. K) [93]. Add to epoxy led to increase the thermal conductivity Compare with Epoxy due to Aluminum particles will spread random according to manufacturing process. The presence of these particles plays important role in the transition heat process.

When the sample surface acquires the heat will moving this energy from the higher temperature zone to the low region heat, the heat transfer mechanism in the composite that depend on heat transfer by phonons and electrons. Upon arrival of a quantity of heat to the polymer chains and molecules made up a bunch of phonons which in turning will travel or absorb by Aluminum particles, transporting energy by electrons to the surrounding area leading to heat transfer from the higher temperature zone to the low region heat. Thermal conductivity for composites increase with increasing weight percentage of filler, The consequences show that the thermal conductivity increase for all composites .additionally it's affected by the type and weight percentage of additive, this end result is in a good agreement with general theory of the thermal conductivity of composites, which has been predicted that the addition of a second phase with thermal conductivity different than that of the matrix can be effect on the thermal conductivity of the resulting composite [94].

Also, heat is transferred as elastic wave, and because of the existence of an interphase (between the matrix and additives) the transfer's motion of these waves are restrained. The transfer of thermal energy as elastic wave is still complex and difficult process since there is disconnection in structure and

transference from one phase to another, i.e. the wave loses part of its energy at the interphase region between the matrix and the reinforcement materials [84].

Figure (4-17): Thermal conductivity V.s Al weight percentage for Ep/ Al composites.

Table (4-10): Values of the Thermal conductivity for EP/Al composites.

Samples	k_{th} (w/m. k)
100% EP	0.671688
95% EP/ 5% Al	0.732399
85% EP/ 15% Al	0.857353
75% EP/ 25% Al	0.976934
65% EP/ 35% Al	1.104591
55% EP/ 45% Al	1.459549

4-4-2 Thermal conductivity for Epoxy/ Copper composites

Thermal conductivity (k_{th}) of Epoxy resin and EP/Cu composites are shown in the Figure (4-18). The results explain that (k_{th}) values of increased with increasing wt% of reinforcement, with max. value (1.5027 W/m. K) at (45%) for EP/Cu composite as shown in Table (4-11). It is clear that (k_{th}) for EP/ Cu composites increase with increasing weight percentage of Cu particles,

that there is no interaction between them. This behavior is agreement with reference [13-16 and 23].

The results show that the thermal conductivity increase for all composites, also it is affected by the weight percentage of additive; this result is in a good agreement with general theory of the thermal conductivity of composites. In polymeric materials, heat is transferred as elastic wave, and because of the existence of an interphase (between the matrix and additives) the transfer's motion of these waves are restricted. The transfer of thermal energy as elastic wave is still complex and difficult process since there is disconnection in structure and transference from one phase to another, i.e. the wave loses part of its energy at the interface region between the matrix and the reinforcement materials [94]. The increase in (k_{th}) is due to the fact that copper is a good heat conductor and its value of thermal conductivity (398 W/m. K) [95]. Add to epoxy led to increase the thermal conductivity Compare with Epoxy due to copper particles randomly according to manufacturing process, having these particles play important role in the process of Thermal conductivity.
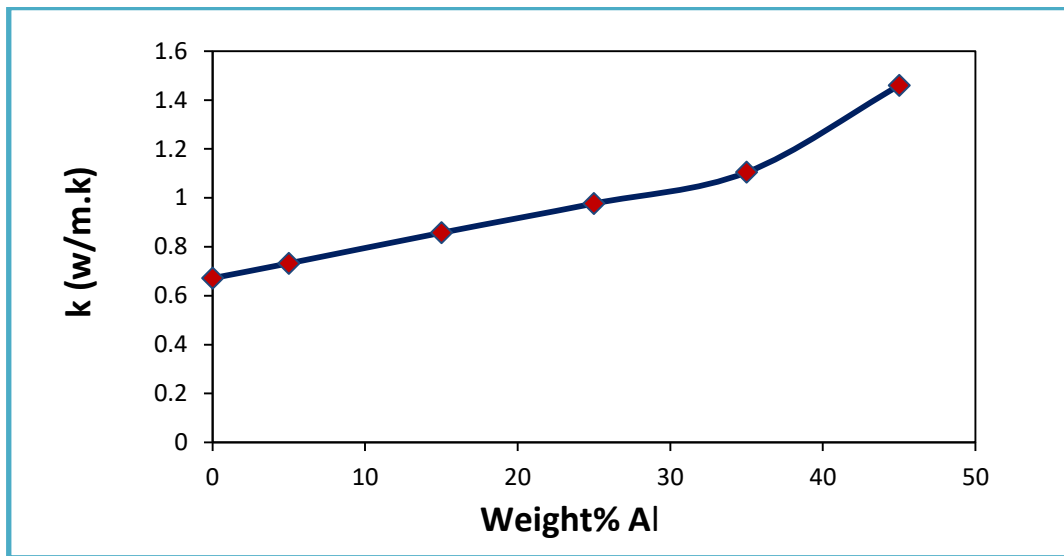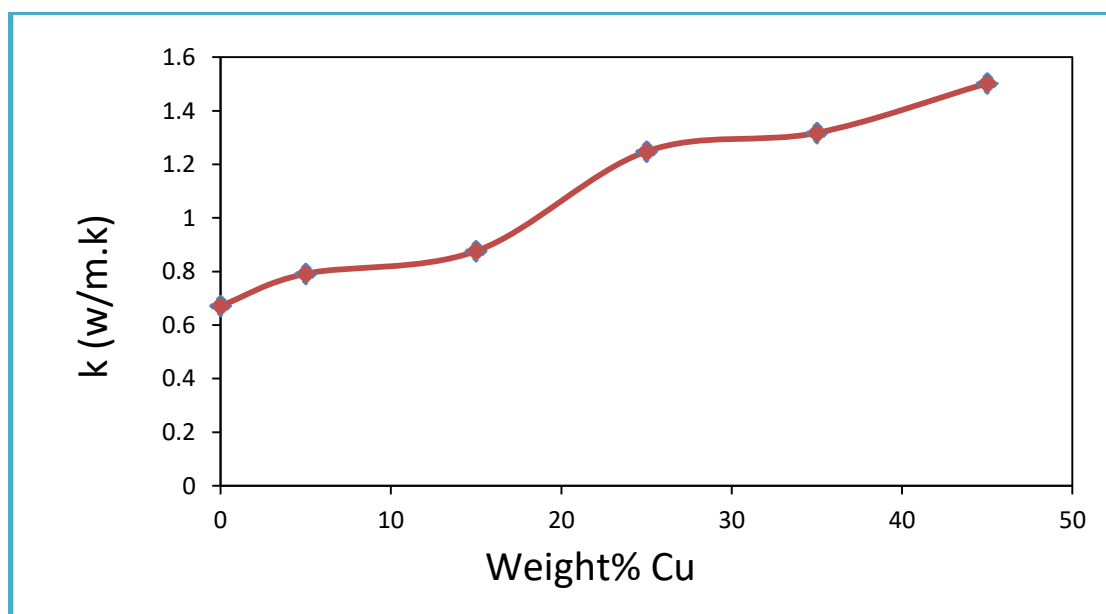

Figure (4-18): Thermal conductivity V.s Cu weight percentage for Ep/ Cu composites.

Table (4-11): Values of the Thermal conductivity for EP/Cu composites.

Samples	k_{th} (w/m. k)
100% EP	0.671688
95% EP/ 5% Cu	0.79167
85% EP/ 15% Cu	0.876408
75% EP/ 25% Cu	1.248396
65% EP/ 35% Cu	1.317728
55% EP/ 45% Cu	1.502775

4-5 Differential Scanning Calorimeter (DSC)

4-5-1 Differential Scanning Calorimeter for Epoxy/ Aluminum composites

During the Figure (4-19), Heat Flow – Temperature curves for Epoxy/ Aluminum composites, Glass transition temperature (Tg) of the specimen were delineates from the tangents of (DSC) spectra as a function of temperature. Glass-transition Temperature of Epoxy resin and EP/Al composites are shown in the Figure (4-20). The results appear that (Tg) values for increase with increasing weight percentage from reinforcement, the maximum value (60.002 ºC) at (45%) filler weight percentage for EP/Al composites as shown in Table (4-12). To explain this behavior, the particles of Aluminum will distribute between the crosslink chains for epoxy, which reduce the mobility of epoxy chains and filling free space between epoxy chains. Upon heating through the (Tg), the amorphous solid polymer transforms from a rigid to a rubbery state. the value of the (Tg) will depend on molecular characteristics that affect chain stiffness, Increasing the molecular weight also tends to raise the (Tg), when the molecular weight is very high, this is due to a decrease in free volume due to a decrease in the concentration of the ends of the polymer chains because free volume depends on the concentration of the ends of the chains. Where the number of chain ends decreases with the molecular weight of the polymer

increases. Also, the glass transition temperature increases with increasing polymer polarity and increasing the density of the cohesive energy.

Figure (4-19): Heat Flow V.s Temperature for Ep/ Al composites.
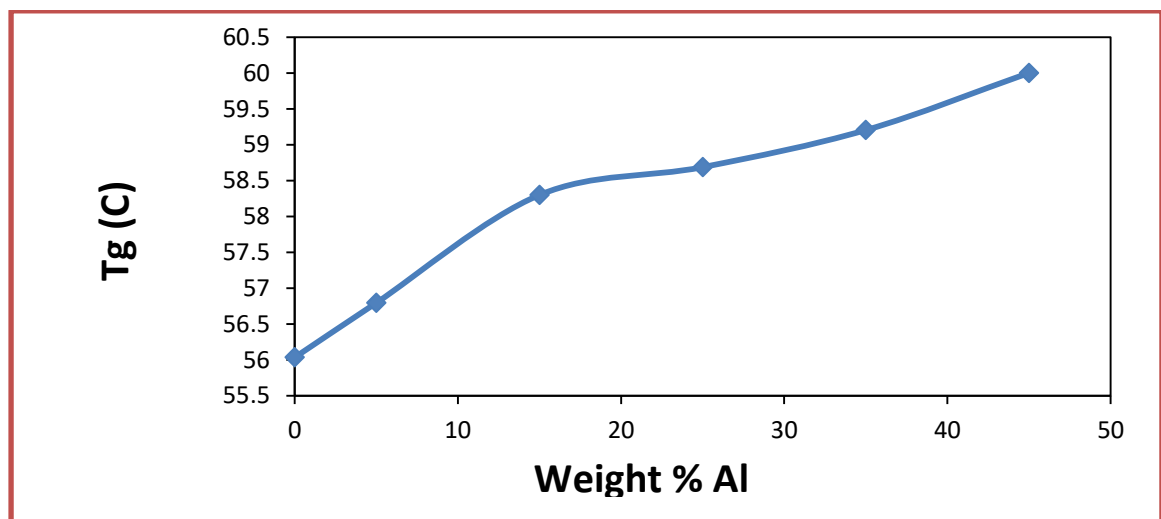
Figure (4-20): Tg V.s Al weight percentage for Ep/ Al composites.

Table (4-12): Values of the Tg (^{o} C) for EP/Al composites.

Samples	Tg (^{o}C)
100% EP	56.034
95% EP/ 5% Al	56.795
85% EP/ 15% Al	58.3
75% EP/ 25% Al	58.688
65% EP/ 35% Al	59.205
55% EP/ 45% Al	60.002

4-5-2 Differential Scanning Calorimeter for Epoxy/ Copper composites

The Figure (4-21) showed the Heat Flow – Temperature curves for Epoxy/ Copper composites, Glass transition temperature (Tg) of the specimen were delineates from the tangents of (DSC) spectra as a function of temperature. (Tg) of Epoxy resin and EP/Cu composites are shown in the Figure (4-22). The (Tg) values are increased with increasing weight percentage of copper except the ratios (25%) and (45%), the maximum value (58.949 °C) at (35%) Cu of composites as shown in Table (4-13), to explain this behavior, the particles of Copper will distribute between the crosslink chains for epoxy, which reduce the mobility of epoxy chains and filling free volume between epoxy chains. Upon heating through the (Tg), the amorphous solid polymer transforms from a rigid to a rubbery state. The value of the (Tg) will depend on molecular weight characteristics that affect chain stiffness. Increasing the molecular weight also tends to raise the (Tg), when the molecular weight is very high, this is due to a decrease in free volume due to a decrease in the movement of the ends of the polymer chains because free volume depends on the concentration of the ends of the chains. Where the number of chain ends movement decreases with the molecular weight of the polymer increases. Also, the glass transition temperature increases with increasing polymer polarity and increasing the density of the cohesive energy. The decrease in the degree of glass transition temperature, due to the increased degree of branching in polymers and this is because the increase in the degree of branching leads to Increase the number of chains ends and increase free volume. These behaviors agree with reference [96 and 97].
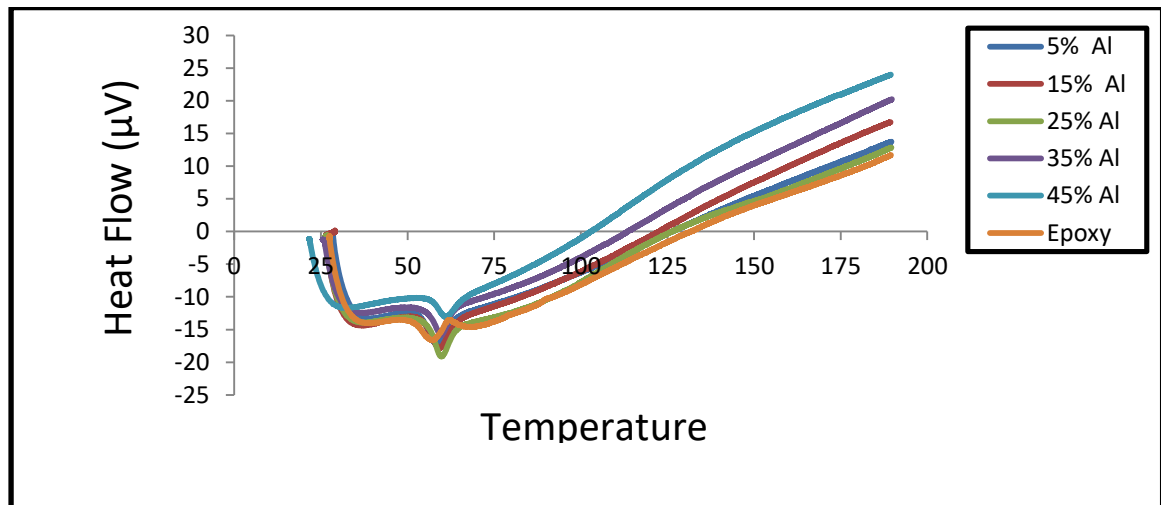
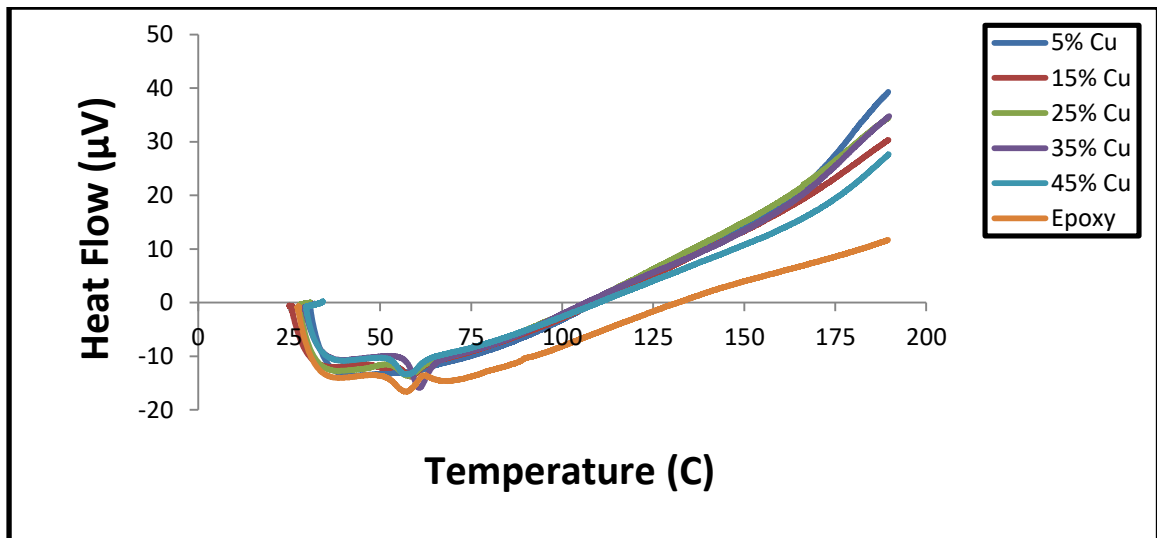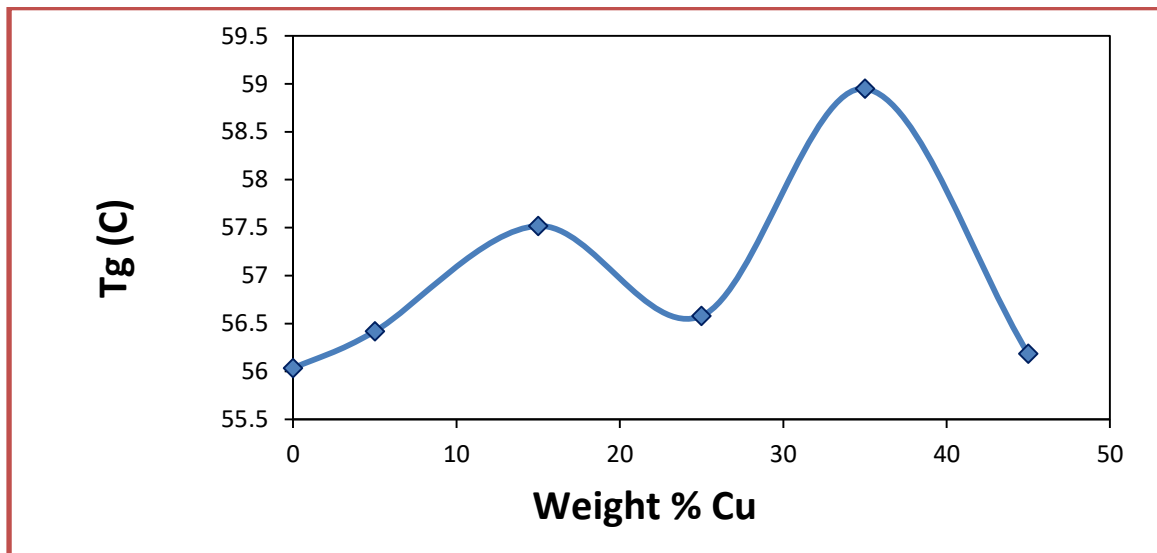Figure (4-21): Heat Flow V.s Temperature for Ep/ Cu composites.

Figure (4-22): Tg V.s Cu weight percentage for Ep/ Cu composites.

Table (4-13): Values of the Tg (º C) for EP/Cu composites.

Samples	Tg (º C)
100% EP	56.034
95% EP/ 5% Cu	56.419
85% EP/ 15% Cu	57.518
75% EP/ 25% Cu	56.58
65% EP/ 35% Cu	58.949
55% EP/ 45% Cu	56.184

4-6 Electrical A.C. characteristics

4-6-1 Electrical A.C. characteristics for Epoxy/ Aluminum composites

The difference $\sigma_{a.c}$ (w) with frequency (50 Hz) to (15MHz) for Epoxy and EP/Al composites (with similarly the same thickness) at room temperature, Figure (4-23) explains the frequency dependence for the Epoxy and EP/Al composites. It is clear from this figure that $\sigma_{a.c}$ (w) increases with increasing frequency. The same behavior of the frequency dependence of $\sigma_{a.c}$ (w) was obtained for all EP/Al composites. It was shown that the a.c conductivity increases when increasing Al concentration. This behavior is agreement with reference [14] This can be attributed to hoping conduction of mobile charge carriers (ions) over barrier between two sites, which is similar to that observed in amorphous semiconductors and glasses [98-99]. Dielectric analysis measures the electric properties of a material as a function of frequency.

Figure (4-24) shows the dielectric constant $\acute{\epsilon}$ depended of frequency at different concentration for EP/Al composites. It is clear from this figure that dielectric constant $\acute{\epsilon}$ decreases with frequency. The decrease of $\acute{\epsilon}$ frequency can be attributed to the fact that at low frequencies $\acute{\epsilon}$ for polar EP/Al composites are explained by the contribution of multi component of polarizibility, deformational (electronic and ionic) and relaxation (orientational and interfacial) polarization.

First, electronic polarization arises from the displacement of the valence electrons relative to the positive nucleus. This type of polarization takes place at frequencies up to (10^{16} Hz). Second type is ionic polarization, occurs due to the displacement of negative and positive ions with respect to each other. The maximum frequency of ionic polarization is (10^{13} Hz). Third; dipolar

polarization occurs if the material contains molecules, with permanent electric dipole moment that can change orientation into the direction of the applied electric field. The dipolar polarization takes place at frequencies up to (10^{10} Hz). The final one is the space charge polarization which occurs due to impedance mobile charge carriers by interfaces. Space charge polarization typically occurs at frequency range from (1) to (10^3 Hz). The total polarization of the dielectric material can be given as the sum of these four types of polarization [100]. The obtained results in the present research may be referred to the total polarization. When the frequency is increased, the orientation polarization decreases since it takes more time than electronic and ionic polarization. This decreases the value of dielectric constant $\acute{\epsilon}$ reaching a constant value at higher frequency correspondingly to interfacial polarization.

It is clear from Figure (4-25) that $\acute{\epsilon}$ decreases with increasing frequency through the studied range. The decrease of $\acute{\epsilon}$ with frequency may be attributed to the fact that the migration of ions is the main source of the dielectric loss $\breve{\epsilon}$ at low frequencies. Accordingly, $\breve{\epsilon}$ at low and moderate frequency is characterized by high values of $\breve{\epsilon}$ due to the contribution of ion jump and conduction loss of ion migration, in addition to the ion polarization. But, at higher frequency values the ion vibrations may be the only source of dielectric loss so $\breve{\epsilon}$ decreases at higher values of frequency. The obtained results of $\breve{\epsilon}$ can be analyzed according to the theory of hopping of charge carriers over a potential barrier between charged defects [101] [102].

It is clear from Figure (4-26) that tanδ (dissipation factor) decreases with increasing frequency through the studied range at room temperature. The same behavior of the tanδ dependence of frequency was obtained for all (EP/Al) composites. tanδ in composites material adopted on the number of charge carriers and the frequency of the imposed electric field in the composites. In

general, a tanδ value for composites with higher concentrations of Al was attributed to interfacial polarization. Also, the tanδ showed increased with a decreased in frequency at room temperature. In low frequency range, when the electrical dipoles are an able to follow the variation of the electric field [14].

It is clear from Figure (4-27) that Capacitance decreases with increasing frequency through the studied range at room temperature. The same behavior of the Capacitance dependence of frequency was obtain for all EP/Al composites. It is seen that the capacitance decreases in the low frequency range and attains a constant value in the high frequency range, this is the usual behavior observed in all EP/Al composites. The decrease of capacitance with increasing frequency is attributed to the increasing inability of the dipoles to orient themselves in a rapidly varying applied field [103].

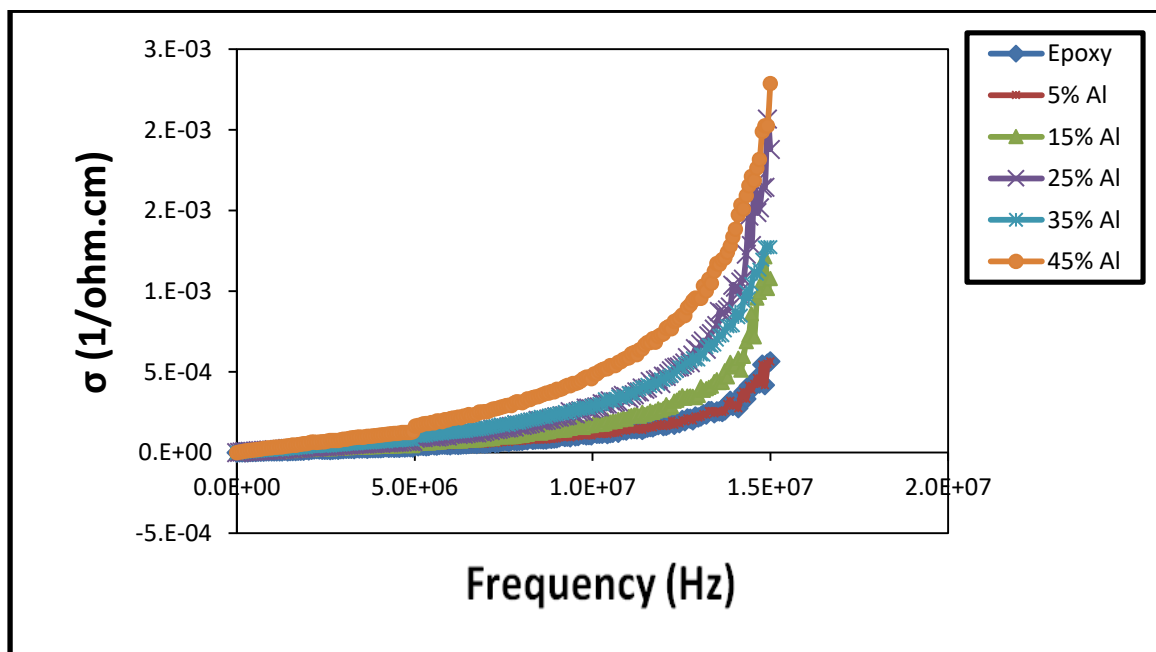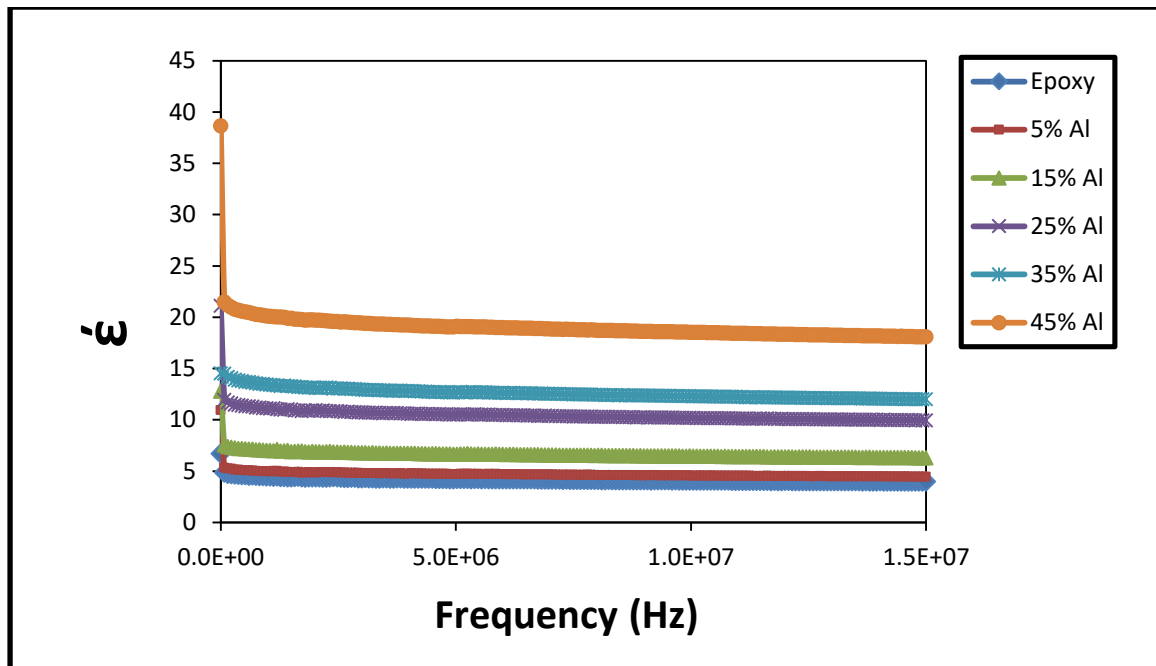Figure (4-23): Conductivity V.s frequency for Epoxy and EP/Al composites.

Figure (4-24): Real permittivity V.s frequency for Epoxy and EP/Al composites.

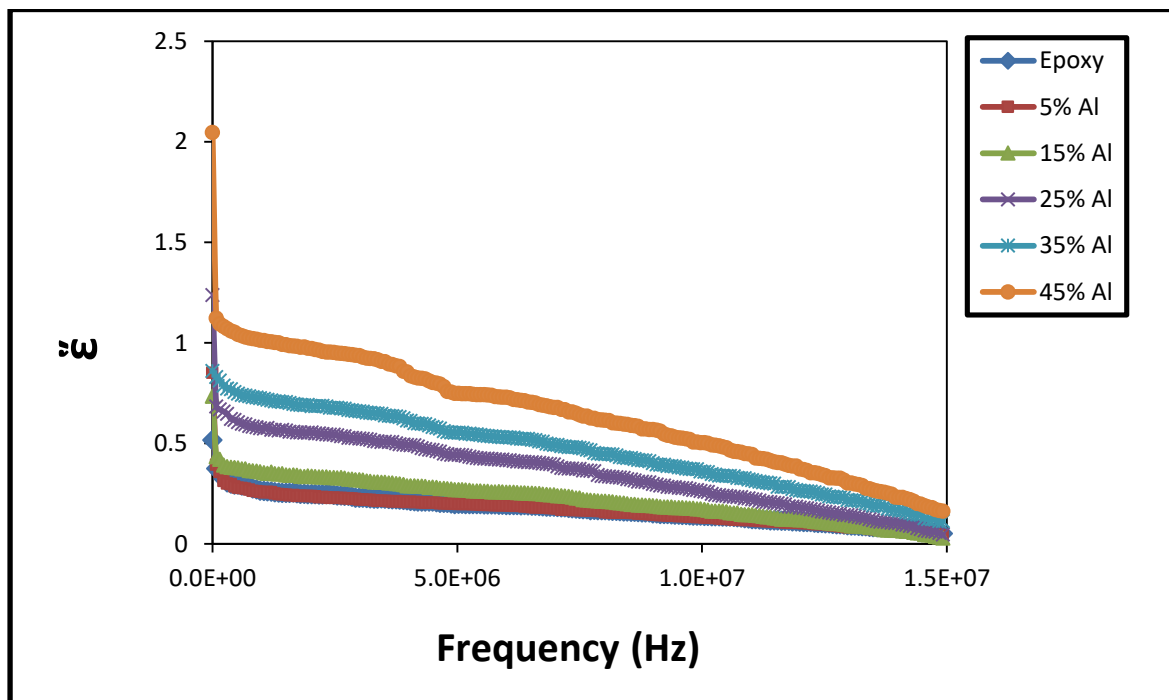

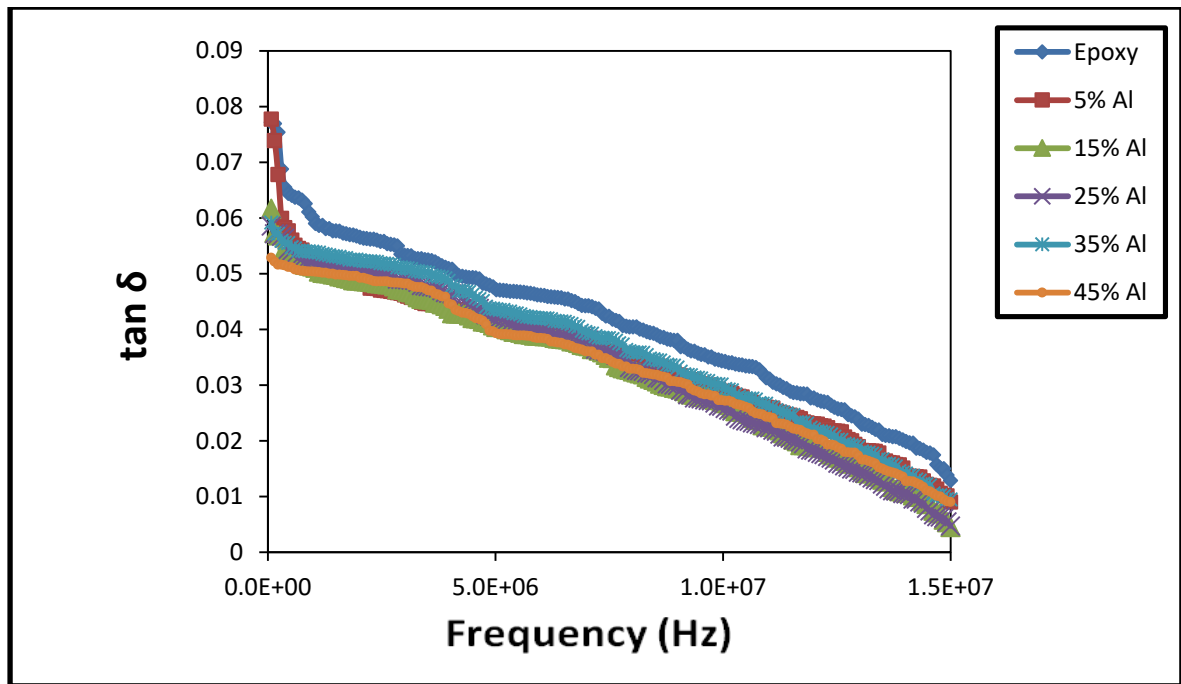Figure (4-25): Variation of $\breve{\varepsilon}$ V.s frequency for Epoxy and EP/Al composites.

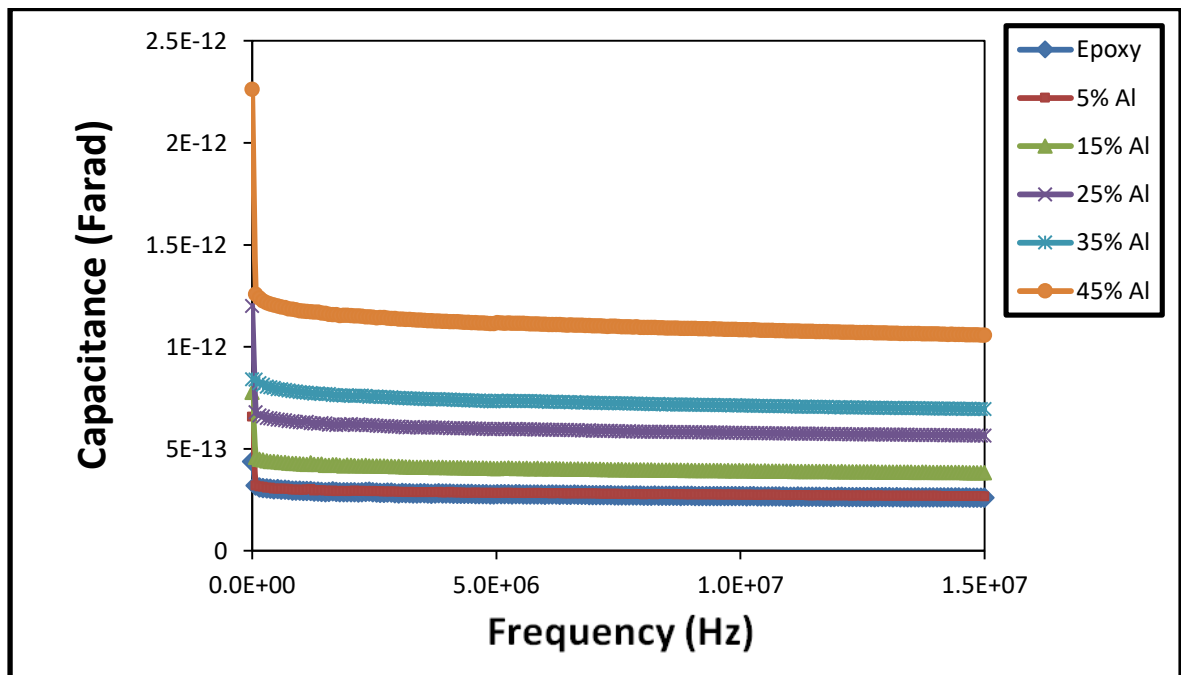Figure (4-26): Tan δ value V.s frequency for Epoxy and EP/Al composites.

Figure (4-27): capacitance V.s frequency for Epoxy and EP/Al composites.

4-6-2 Electrical A.C. characteristics for Epoxy/ Copper composites

The difference of $\sigma_{a.c}$ (w) with frequency in the range (50Hz) to (15MHz) for Epoxy and EP/Cu composites (with similarly the same thickness) at room temperature, Figure (4-28) explains the frequency dependence for the Epoxy and EP/ Cu composites. It is clear from this figure that $\sigma_{a.c}$ (w) increases with increasing frequency. The same behavior of the frequency dependence of $\sigma_{a.c}$ (w) was obtained for all EP/ Cu composites. It was shown that the a.c conductivity increases when increasing Cu concentration. This can be attributed to hoping conduction of mobile charge carriers (ions) over barrier between two sites, which is similar to that observed in amorphous semiconductors and glasses [97-98]. Dielectric analysis measures the electric properties of a material as a function of frequency.

Figure (4-29) shows the dielectric constant $\acute{\varepsilon}$ depended of frequency at different concentration for EP/ Cu composites. It is clear from this figure that dielectric constant $\acute{\varepsilon}$ decreases with frequency. The decrease of $\acute{\varepsilon}$ frequency can be attributed to the fact that at low frequencies $\acute{\varepsilon}$ for polar EP/ Cu composites are explained by the contribution of multi component of polarizibility, deformational (electronic and ionic) and relaxation (orientational and interfacial) polarization. The obtained results in the present research may be referred to the total polarization. As explain at section (2-14-3). When the frequency is increased, the orientation polarization decreases since it takes more time than electronic and ionic polarization. This decreases the value of dielectric constant $\acute{\varepsilon}$ reaching a constant value at higher frequency correspondingly to interfacial polarization.

As shown in Figure (4-30), when increasing the frequency, the $\breve{\varepsilon}$ decreases, and the main source of the $\breve{\varepsilon}$ at low frequency, attributed to the migration of

ions, high values for $\acute{\varepsilon}$ at low and moderate frequency, due to the influence, of the polarization, conduction loss for ion migration and ion jump. But, at higher frequency values the ion vibrations may be the only source of dielectric loss so $\acute{\varepsilon}$ decreases at higher values of frequency. The obtained results of $\acute{\varepsilon}$ can be analyzed according to the theory of hopping of charge carriers over a potential barrier between charged defects [101-102].

It is clear from Figure (4-31) that tanδ (dissipation factor) decreases with increasing frequency at room temperature. The same behavior of the tanδ dependence of frequency was obtained for all EP/ Cu composites. tanδ in composites material adopted on the No. carriers and the frequency of the imposed electric field in the composites. In general, a tanδ value for composites with higher concentrations of Cupper was due to polarization. It was showed at room temperature tanδ increased with a decreased in frequency. At low frequency, the dipoles are an able to follow the variation of the electric field [14].

It is clear from Figure (4-32) that Capacitance decreases with increasing frequency through the studied range at room temperature. The same behavior of the Capacitance dependence of frequency was obtained for all EP/ Cu composites. It is seen that the capacitance decreases in the low frequency range and attains a constant value in the high frequency range, this is the usual behavior observed in all EP/Cu composites. The decrease of capacitance with increasing frequency is attributed to the increasing inability of the dipoles to orient themselves in a rapidly varying applied field [103].

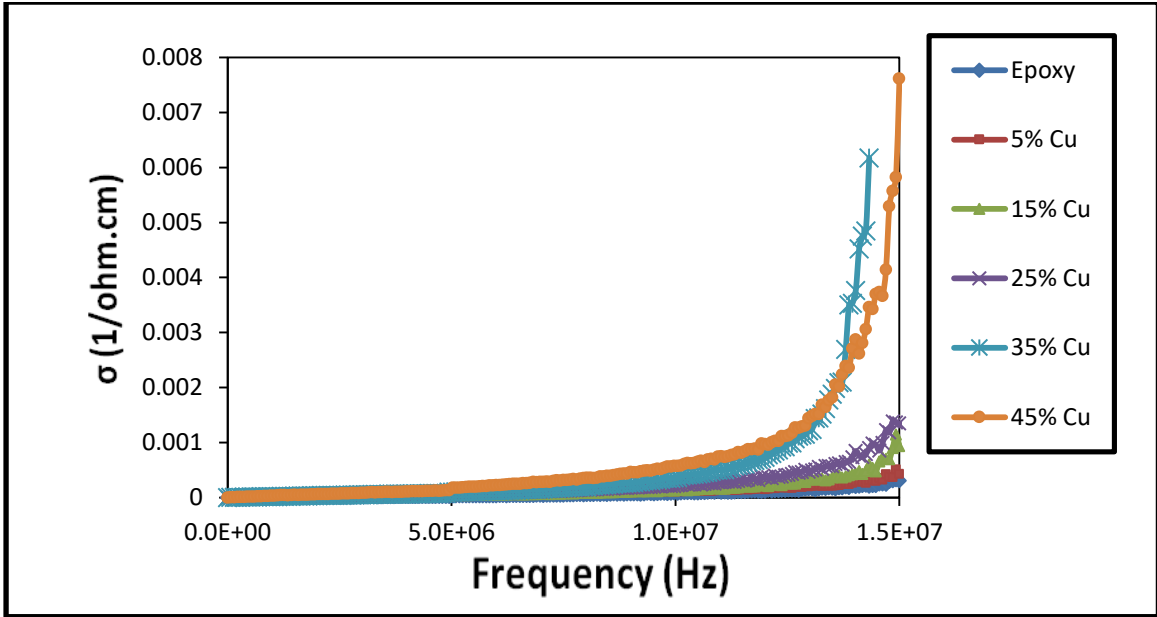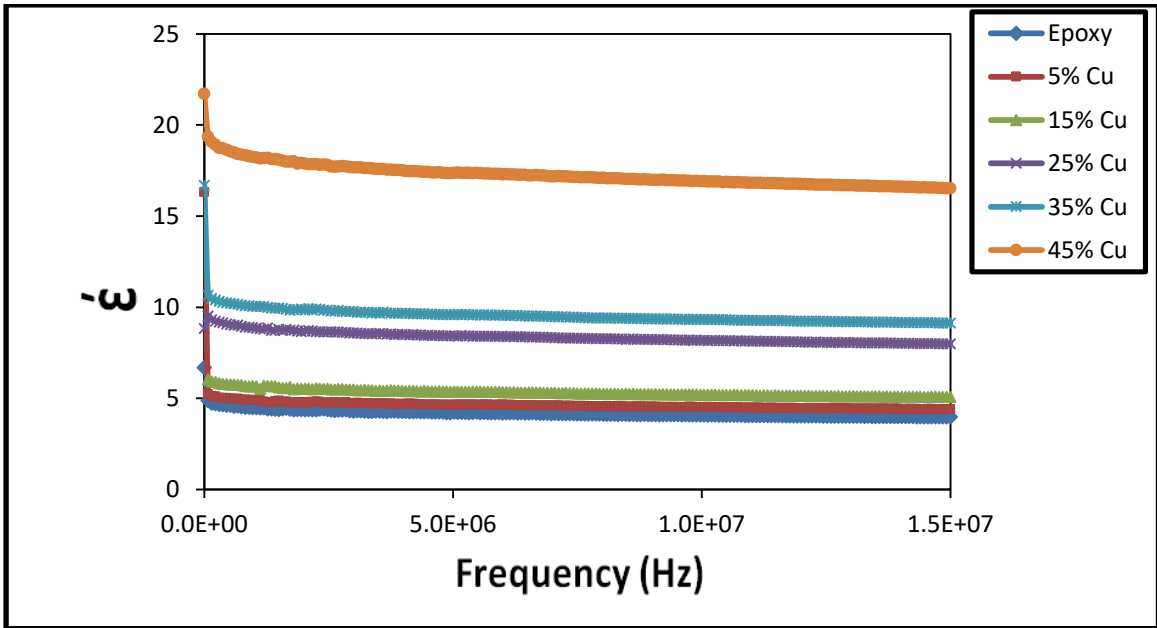Figure (4-28): Conductivity V.s frequency for Epoxy and EP/Cu composites.

Figure (4-29): Real permittivity V.s frequency for Epoxy and EP/Cu composites.
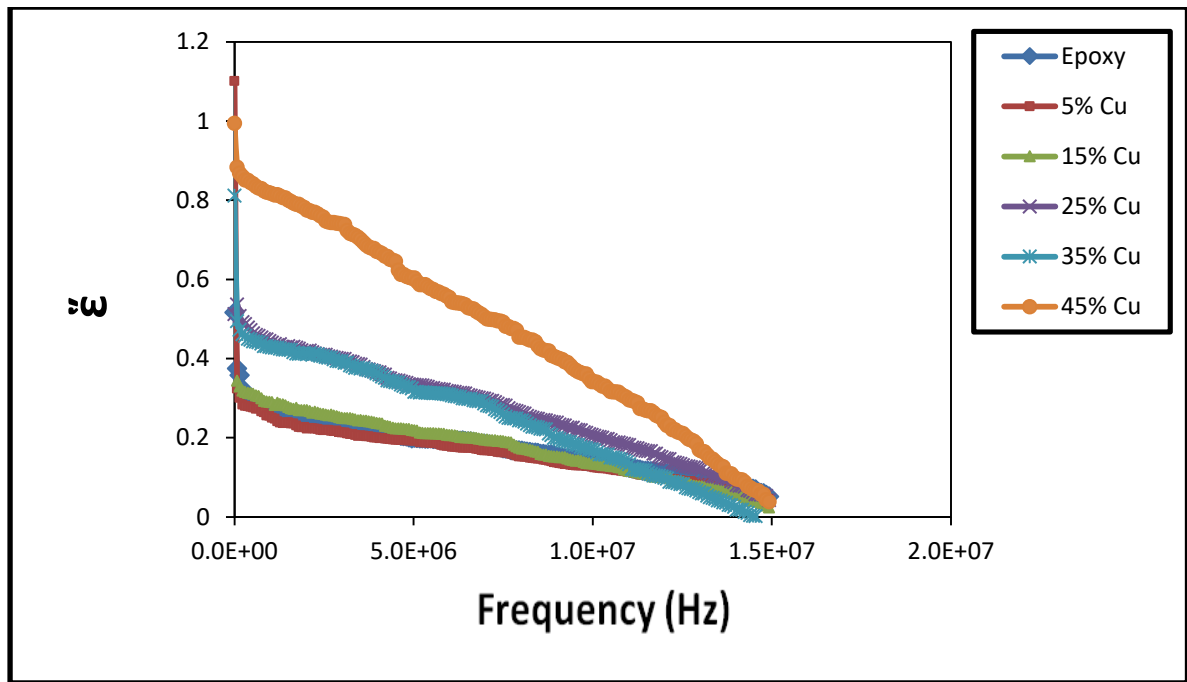
Figure (4-30): Variation of ɛ̌ V.s frequency for Epoxy and EP/Cu composites.
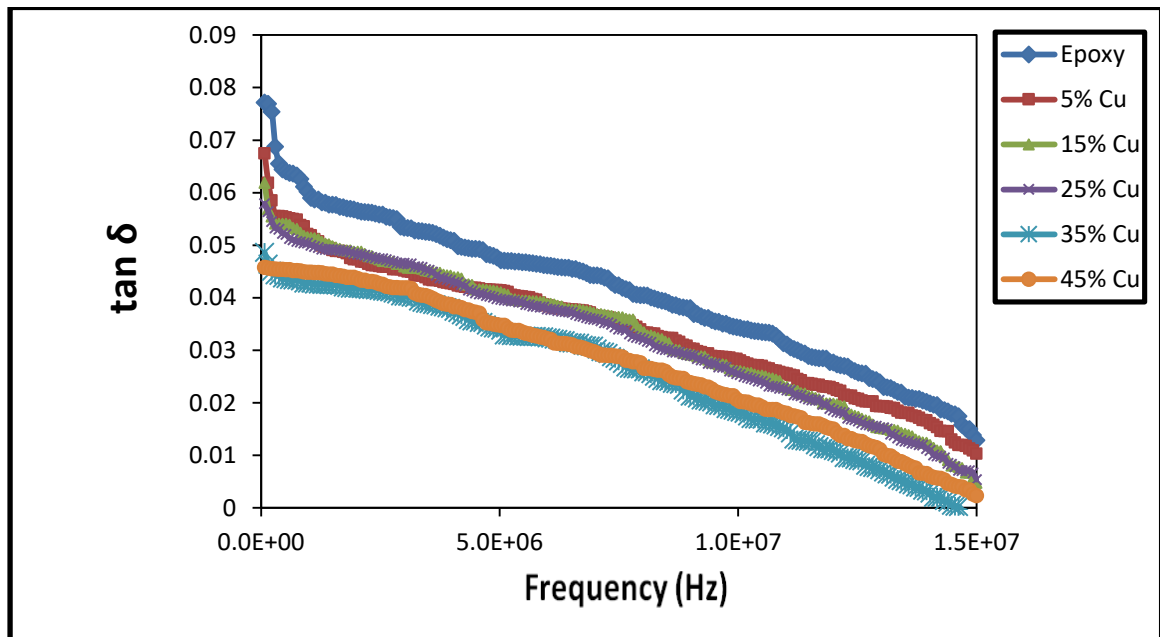
Figure (4-31): Tan δ value V.s frequency for Epoxy and EP/Cu composites.
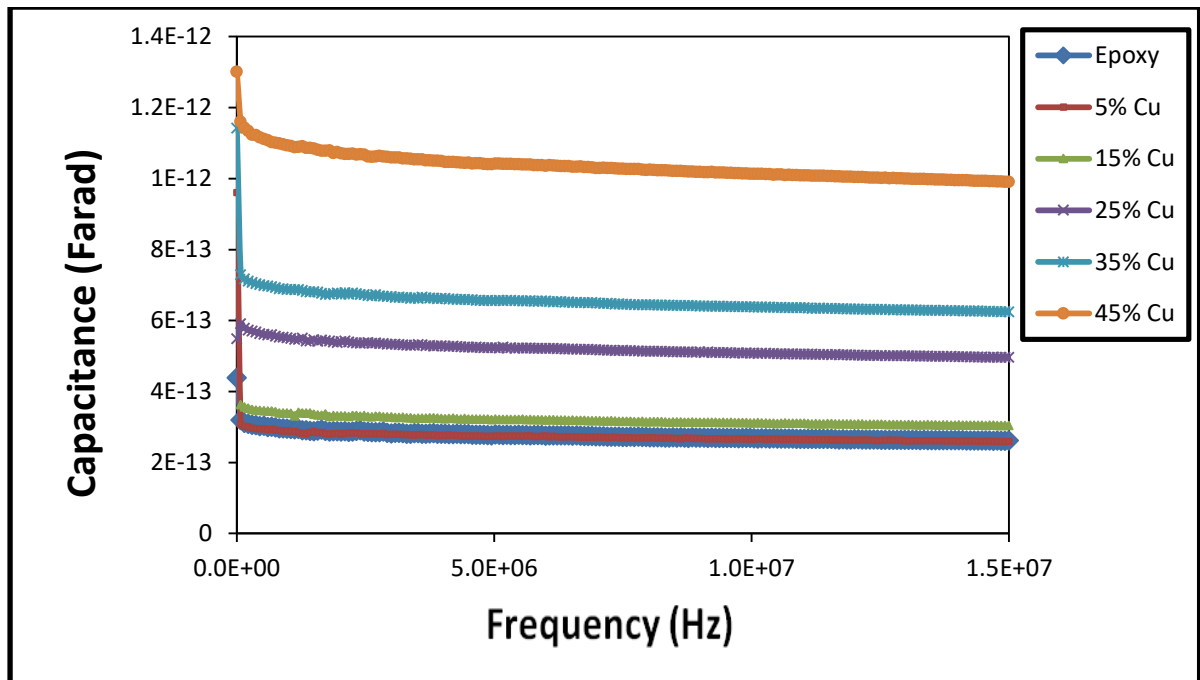
Figure (4-32): Capacitance V.s frequency for Epoxy and EP/Cu composites.

CHAPTER FIVE

Conclusion and Future works
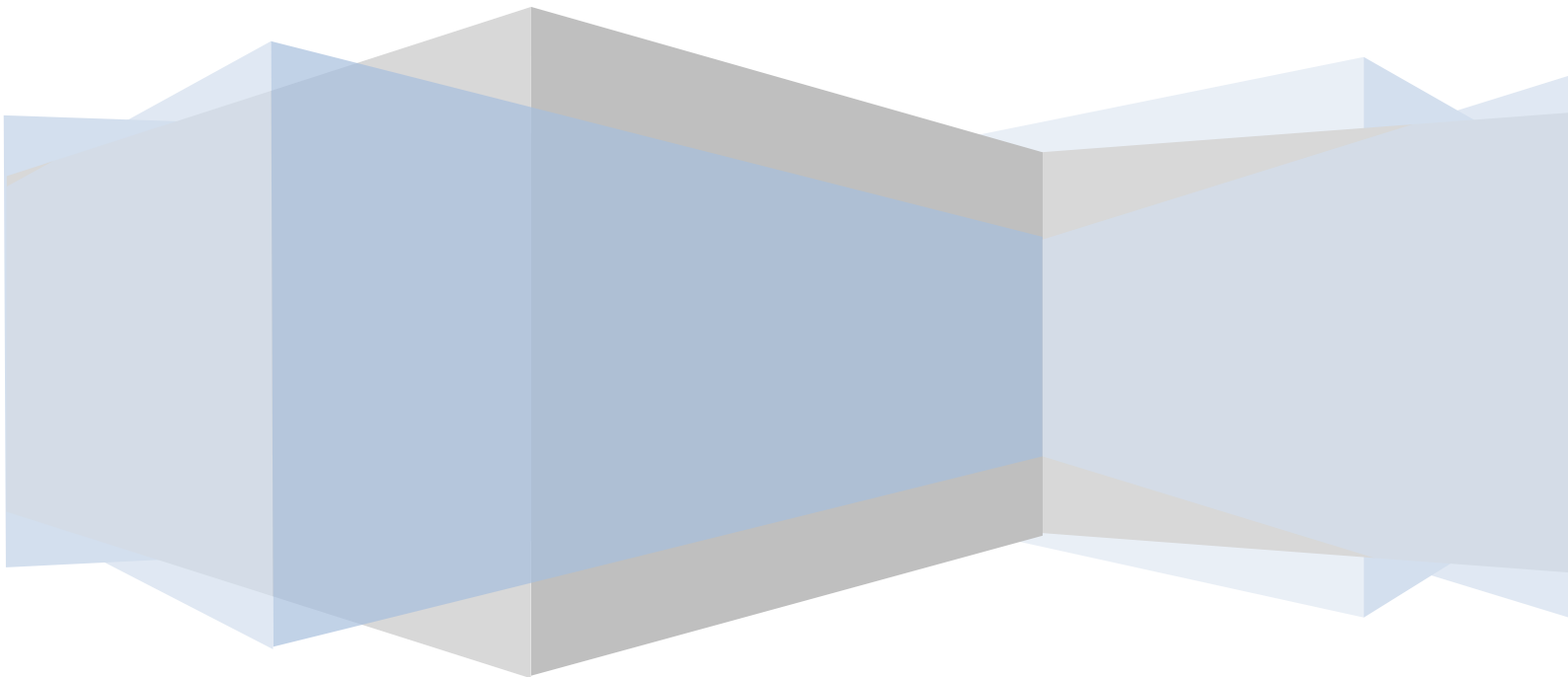
CHAPTER FIVE

Conclusion and Future Work

5-1 Conclusion

The following Conclusions obtained from experimental work:

1. Flexural modulus (E) increase with increasing Aluminum percentage and it has maximum values of (8.1638MPa) for (55%EP+45%Al), which increased by (192.8%) compared to epoxy. While Flexural modulus (E) increase with increasing Copper percentage and it has maximum values of (4.8330752 MPa) for (55%EP+45%Cu), which increased by (73.35%) compared to epoxy.

2. Flexural strength, Fracture Force and Deflection decreases with increasing of the Aluminum percentages compared with pure Epoxy, except the ratio (85%EP+15%Al). Flexural strength has maximum value of (90MPa) for the (85%EP+15%Al), which increased by (4.53%) compared to epoxy. While Flexural strength and Fracture Force decreases with increasing of the Copper percentages compared with pure Epoxy, except the ratio (75%EP+25%Cu) and (65%EP+35%Cu). Flexural strength has maximum value of (90MPa) for the ratio (75%EP+25%Cu), which increased by (4.53%) compared to epoxy. Deflection decreases with increasing of the Copper percentages compared with pure Epoxy.

3. Yield Points for all Aluminum and Copper percentages increasing with increasing ratio fillers.

4. Increase hardness with Aluminum concentration increase, and it has maximum values of (83.2) for (55%EP+45%Al), which increased by (7.5%) compared to epoxy. While Increase hardness with Copper concentration increase, and it has maximum values of (83.9) for (55%EP+45%Cu), which increased by (8.4%) compared to epoxy.

5. Increase thermal conductivity for (EP/Al) composites with increasing addition rates of fillers, where high value (1.459549 (W/m. K)) at (55%EP+45%Al), which increased by (117.3%) compared with epoxy. And Thermal conductivity for (EP/Cu) composites were increase with increasing wt% from fillers, (1.502775 (W/m. K)) is max value at (55%EP+45%Cu), which increased by (123.73%) compared with epoxy.

6. The Glass transition temperature (T_g) of (EP/Al) composites were increased with increasing concentration of Al powder, the maximum value (60.002°C) at (55%EP+45%Al), Which increased by (7.081%) compared with epoxy. While the maximum Glass transition temperature (T_g) of (EP/ Cu) composites were the value (58.949°C) at (65%EP+35%Cu), which increased by (5.2%) compared with epoxy.

7. Electrical properties for (EP/Al) composites, conductivity $\sigma_{a.c}$ (w) increases with increasing frequency, conductivity increases with increasing Al concentration, and it has maximum value of (0.002288179 (ohm.cm)$^{-1}$) at frequency (15MHz) for (55%EP+45%Al). We note at increasing frequency, decreases all from έ, ἕ, dissipation factor tanδ and capacitance. While Electrical properties for (EP/Cu) composites, conductivity $\sigma_{a.c}$ (w) increases with increasing frequency, conductivity increases with increasing Cu concentration, and it has maximum value of (0.007614337 (ohm.cm)$^{-1}$) at frequency (15MHz) for (55%EP+45%Cu). The έ, ἕ, dissipation factor tanδ and Capacitance decreases with increasing frequency.

8. Information's of measurements for mechanical and thermal properties could be beneficial in applications automotive. As for information's of measurements for electrical properties could be beneficial as Capacitors to energy storage in the laser systems.

5-2 Future work

1. Study other mechanical properties for (EP/Cu) and (EP/Al) composites such as creep, impact, and wear.

2. Study environmental effect (moisture and UV effect) for (EP/Al) and (EP/Cu) composites.

3. Mixing (Cu% +Al%) with Epoxy and Study the mechanical properties and Thermal conductivity.

4. Estimation the lifetime of using (TGA) temperature (EP/Cu) and (EP/Al) composites.

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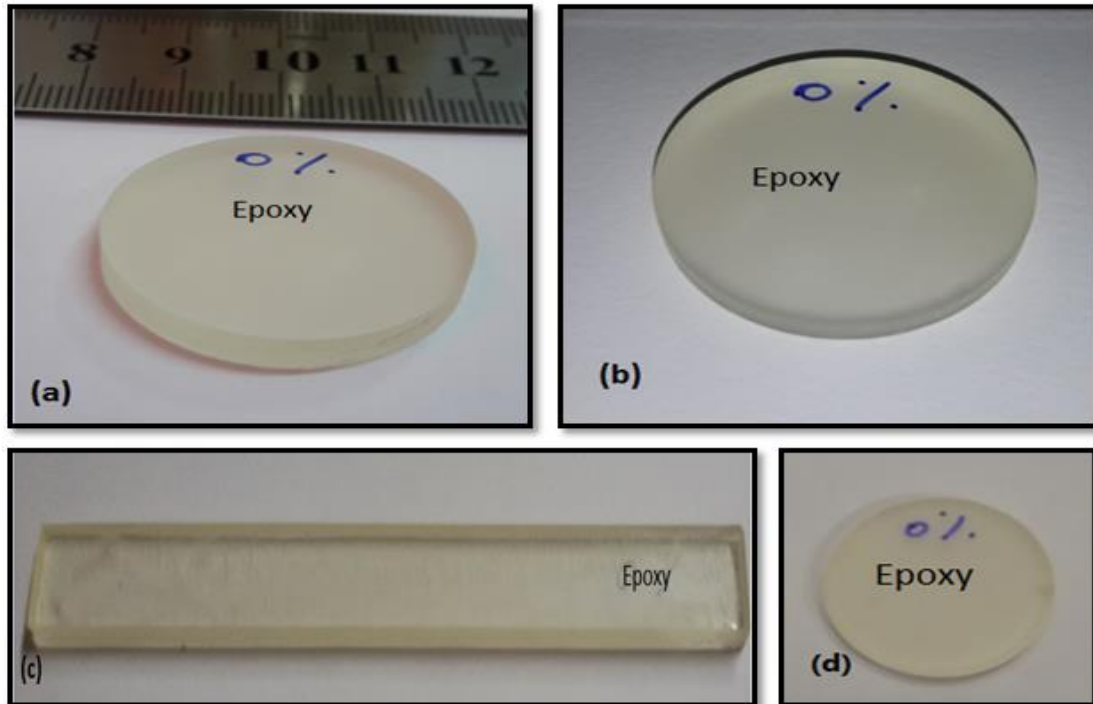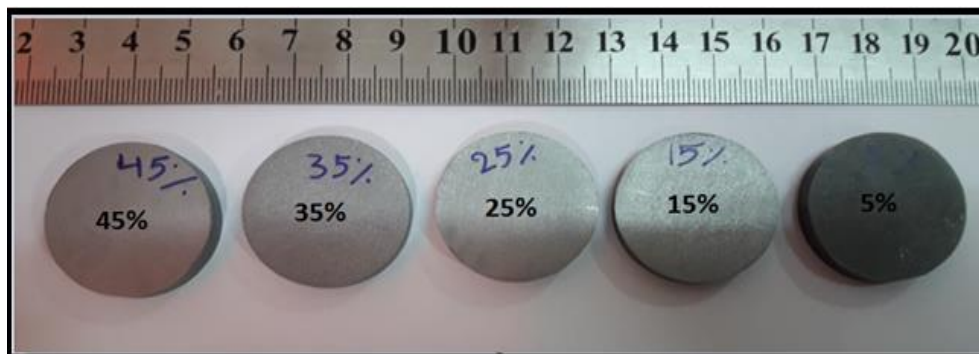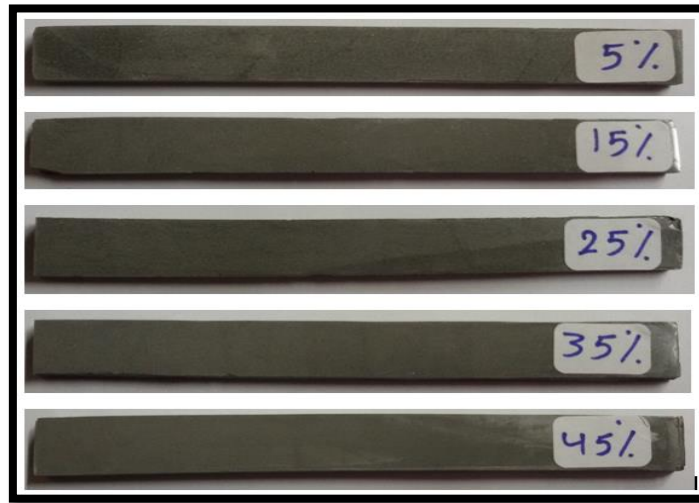
Appendix

Figure show the samples of epoxy resins. (a) Photograph of Shore D Hardness Test Specimen. (b) Photograph of Thermal conductivity Test Specimen. (c) Photograph of Flexure Test Specimen. (d) Photograph of Electrical Test.
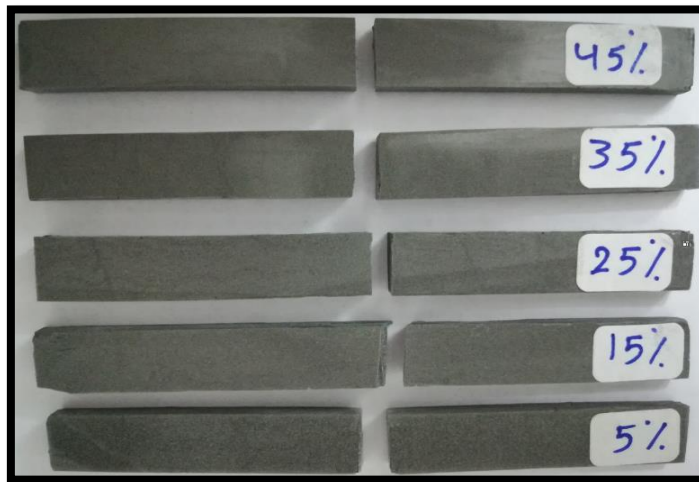

Photograph of Shore D Hardness Test Specimens for Epoxy/ Aluminum composites.

Photograph of Flexure Test Specimens for Epoxy/ Aluminum composites before testing.

Photograph of Flexure Test Specimens for Ep/ Al composites after testing.

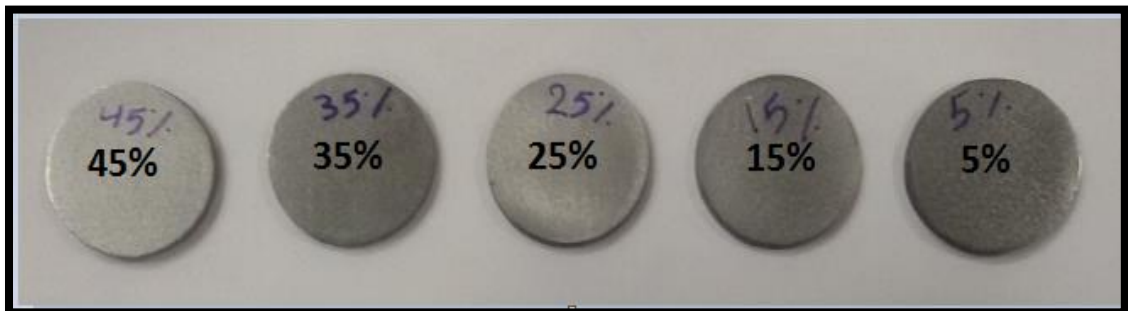Figure Photograph of Thermal conductivity Test Specimens for Epoxy/ Aluminum composites.
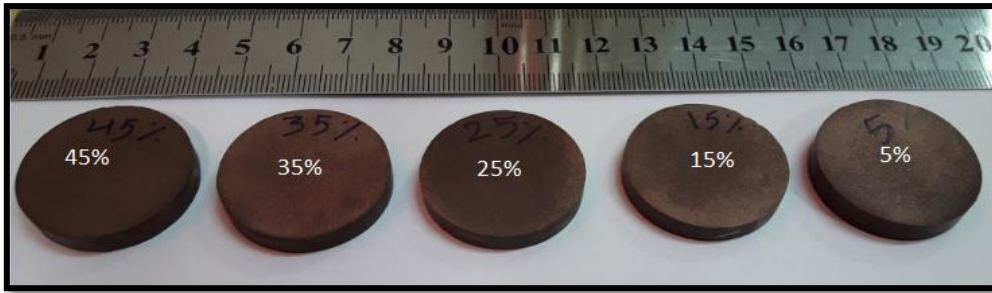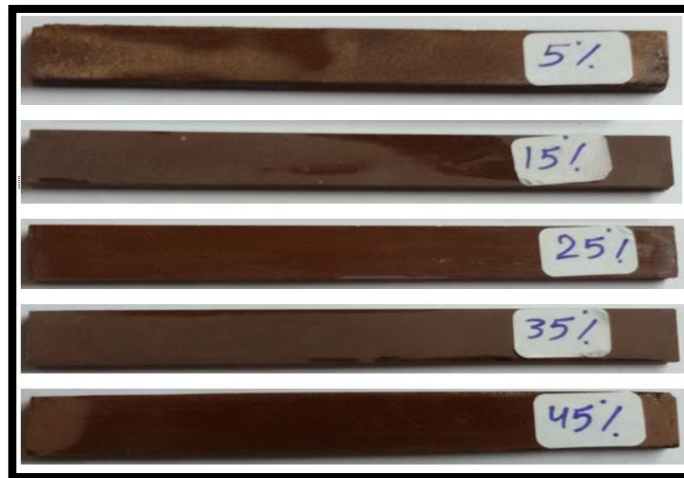

Figure Photograph of Electrical Test Specimens for Epoxy/ Aluminum composites.
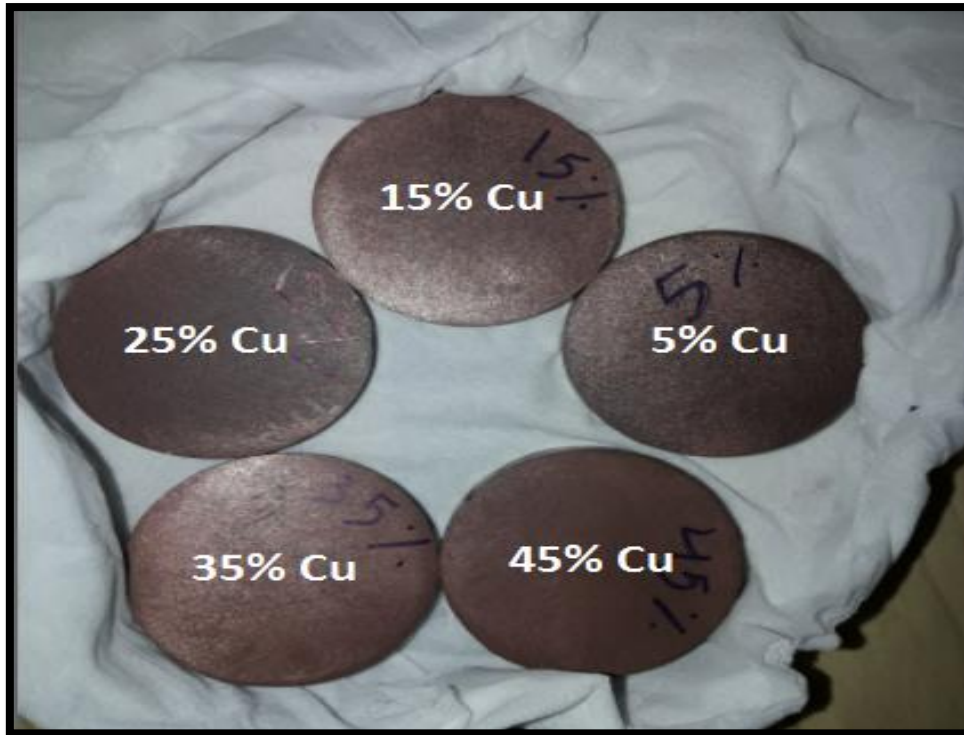
Photograph of Shore D Hardness Test Specimens for Epoxy/ Copper composites.

Photograph of Flexure Test Specimens for Epoxy/ Copper composites before testing.

Photograph of Flexure Test Specimens for Ep/ Cu composites after testing.

Photograph of Thermal conductivity Test Specimens for Epoxy/ Copper composites.

Photograph of Electrical Test Specimens for Epoxy/ Copper composites.

الخلاصة

متراكبات الايبوكسي /المنيوم ومتراكبات الايبوكسي /النحاس حضرت بخلط نسب وزنية مختلفة من مسحوق الالمنيوم والنحاس، لفحص ودراسة خصائصها الفيزيائية (قوة الانحناء، معامل الانحناء، أعظم قوة للكسر، أعظم انحراف، نقطة الخضوع، الصلادة، التوصيل الحراري، درجة الانتقال الزجاجي، التوصيلية الكهربائية، ثابت العزل، ظل زاوية الفقد والسعة).

اظهرت النتائج التي تم الحصول عليها من فحص الانحناء ذي النقاط الثلاثة ان معامل المرونة لمتراكبات الايبوكسي/ المنيوم والايبوكسي/ نحاس يزداد بزيادة تركيز المساحيق المعدنية. قوة الانحناء وأعظم قوة للكسر وأعظم انحراف لمتراكبات الايبوكسي/ المنيوم تتناقص مع زيادة تركيز مسحوق الالمنيوم مقارنة مع الايبوكسي النقي ماعدا النسبة 15% من مسحوق الالمنيوم. بينما قوة الانحناء واعظم قوة للكسر تتناقص مع زيادة تركيز مسحوق النحاس مقارنة مع الايبوكسي النقي ماعدا النسبتان 25% و 35%. الانحراف تتناقص مع زيادة تركيز مسحوق النحاس مقارنة مع الايبوكسي. نقاط الخضوع لجميع نسب الالمنيوم والنحاس زادت مع زيادة تركيز المساحيق المعدنية. ازدادت الصلادة مع زيادة نسب التدعيم بالمساحيق المعدنية.

التوصيلية الحرارية لجميع متراكبات الايبوكسي/المنيوم و الايبوكسي / نحاس ازدادت مع زيادة تركيز المساحيق المعدنية. اظهرت النتائج التي تم الحصول عليها من الماسح التفاضلي المسعري الحراري ان درجة حرارة الانتقال الزجاجي لجميع متراكبات الايبوكسي/المنيوم ازدادت مع زيادة تركيز مسحوق الالمنيوم، اما لمتراكبات الايبوكسي / نحاس بلغت اعلى قيمة لها عند نسبة التدعيم 35% من مسحوق النحاس. المعلومات التي تم الحصول عليها من القياسات للخصائص الميكانيكية والحرارية تكون مفيدة في تطبيقات السيارات.

أظهرت الصفات الكهربائية لمتراكبات الايبوكسي/المنيوم والايبوكسي / نحاس ان التوصيلية الكهربائية تزداد مع زيادة التردد، وتزداد التوصيلية مع زيادة تركيز مسحوق الالمنيوم والنحاس، ثابت العزل والخسارة وظل الفقد والسعة تنخفض مع زيادة التردد. وعلية يمكن استخدامها كمتسعات خزن طاقة في أنظمة الليزر.

تأثير مسحوق الالمنيوم والنحاس على الخصائص الفيزيائية لمتراكبات الايبوكسي

رسالة تقدمت بها

رحاب نصر فاضل

بكالوريوس في علوم الفيزياء

كلية التربية للعلوم الصرفة ـ ابن الهيثم/جامعة بغداد

(2012)

مقدمة إلى مجلس كلية التربية للعلوم الصرفة ـ ابن الهيثم/ جامعة بغداد

وهي جزء من متطلبات نيل شهادة الماجستير

علوم في الفيزياء

بأشراف

أ. د. كريم علي جاسم

2018م   1438هـ