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ELECTRICAL AND MECHANICAL PROPERTIES OF MWCNT FILLED CONDUCTIVE ADHESIVES ON LEAD FREE SURFACE FINISHED PCB's.

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ABSTRACT OF THESIS

ELECTRICAL AND MECHANICAL PROPERTIES OF MWCNT FILLED CONDUCTIVE ADHESIVES ON LEAD FREE SURFACE FINISHED PCB's.

Electrically conductive adhesives (ECA) are an alternative to tin/lead solders for attaching Surface Mount Devices (SMD) in electronic assemblies. ECAs are mixtures of a polymer binder (for adhesion) and conductive filler (for electrical conductivity). They bring more conductivity, higher strength, less weight and longer durability than metal alloys. ECAs can offer numerous advantages such as fewer processing steps, lower processing temperature and fine pitch capability. Multi walled carbon nanotubes (MWCNT) were used as conductive fillers in this research because of their novel electronic and mechanical properties.

The high aspect ratio of the nanotubes makes it possible to percolate at low loadings to obtain good electrical and mechanical properties. Replacing the metal filler with CNTs in the adhesive made the ECA light weight, corrosion resistant, reduced processing temperature, lead free, electrically conductive and high mechanical strength. The MWCNTs at different loadings were mixed with epoxy and epoxy: heloxy to form a composite mixture. Different loadings, additives and mixing methods were used to obtain good electrical and mechanical properties and pot life. Pressure dispensing, screen and stencil printing were the processing techniques used for making the samples. The volume resistivity, contact resistance, die shear and lap shear tests were conducted on different surface finished Printed Circuit Boards (PCB) like silver, tin and Electro less Nickel Immersion Gold (ENIG). The results are summarized and compared with traditional methods.

KEYWORDS: Carbon nanotubes(CNTs), Electrically conductive adhesives(ECAs),Multi walled carbon nanotubes (MWCNTs), Printed Circuit Boards (PCB),epoxy, heloxy.

Keerthi Varma Mantena

7/17/2009

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THESIS

Keerthi Varma Mantena

The Graduate School

University of Kentucky

2009

ELECTRICAL AND MECHANICAL PROPERTIES OF MWCNT FILLED CONDUCTIVE ADHESIVES ON LEAD FREE SURFACE FINISHED PCB's.

THESIS

A thesis submitted in partial fulfillment of the requirements for the degree of "Master of Science in Electrical Engineering in the College of Engineering" at the

University of Kentucky

By

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Lexington, Kentucky

Director: Dr. Janet K Lumpp, Professor

Electrical and Computer Engineering

Lexington, Kentucky

2008

DEDICATION

To My Parents and Family

ACKNOWLEDGEMENTS

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Chapter 1 Introduction

Lead based solder materials have been in use for the interconnection of electronic components on printed circuit boards (PCBs) for a long time now. As the environmental awareness in increasing, there is a great deal of pressure on the industry to eliminate or try to reduce the usage of lead. The policies and legislations have been proposed worldwide to ban and reduce the use of lead in solders through RoHS, and the United States is also taking the restrictions seriously. There has been a great effort with in the industry to develop lead-free and environment friendly solder materials to replace the lead-based solders.

An alternative to the lead based solder are the lead free electrically conductive adhesives (ECAs). ECAs consist of polymer binder which provides mechanical strength, and conductive fillers, that offer electrical conduction. ECAs provide environment friendly solution for interconnection in electronic applications. ECAs also have some potential advantages over the conventional solder techniques which include low temperature processing , finer pitch and simple processing [1][2]. From these the ECAs are considered as the next generation interconnection materials for electronic packaging. This interconnection technology is relatively new when compared to other technologies so it does have some limitations and drawbacks. The reliability issues that include limited impact resistance [3], increased contact resistance and weak mechanical strength in some climatic conditions [4] [5] are some of the major drawbacks that are preventing ECAs from replacing the solder in certain electrical applications. It is necessary to understand the mechanisms underlying these problems, and to improve the electrical properties of the conductive adhesives before ECAs can be widely used as solder replacement.

Integrated chip packaging is the first level packaging which provides mechanical strength, environment protection, heat dissipation and electrical connections for the semiconductor devices. The second level packaging is system level packaging which provides interconnection of the devices on the system level board as well mechanical support and heat dissipation for the system. The important package interconnection technologies presently in the market are wire bonding, flip chip bonding and tape automated bonding [6].

The IC packaging processes make individual electrical components ready to be handled; Printed Wiring Assembly processes integrate different electrical components into fully functional units. The two major PWB assembly technologies are through-hole assembly and surface mount assembly. In through assembly, leads of the components are inserted into the conducting plated through holes and soldered on the back side of the PWB. Where as in surface mount assemble the components can be placed on either side of the board and soldered. Due to the fact that surface mount assembly utilizes both sides of the board and size of surface mount components is usually smaller than comparable through-hole components, surface mount assembly has grown in popularity [6].

In this thesis, we focused on the electrical and mechanical properties of carbon nanotube adhesives on the commercially available surface finishes in the market. We also modified the adhesive by using a heloxy, which is a viscosity modifier. We tested this composite in five different variations on all the three surface finishes gold, silver and tin. Replacing the MWCNTs with metal fillers have the advantages of being lead free, resistant to corrosion, light weight and good mechanical strength.

Chapter 2 Background

2.1 Conductive adhesives

Electrically conductive adhesive (ECA) technology is one of the lead free alternatives to solder technology. ECAs consist of a polymer binder and conductive filler. The polymer based adhesives are widely used in the industry. They are used as insulators due to their excellent dielectric properties. Conductive filler particles are mixed into the polymer adhesive to provide electrical continuity. Metal filled isotropic conductive adhesives were commercially available in the market even before RoHS. ECAs can be classified as thermoplastic or thermosetting, based on their physical properties upon curing. The two main classifications of ECAs are isotropic conductive adhesive (ICA) and anisotropic conductive adhesive (ACA).

ICAs can be either thermoplastic or thermosetting. Thermoplastics have a long linear molecular structure which gives them the ability to melt even after curing when heated to a particular melting point without altering their intrinsic properties[b]. They are based on conductive filler particles mixed in a solvent and dispersed in a polymer matrix. Thermoplastic adhesives have unlimited shelf life at room temperature. Thermoplastic adhesives are rework able [7]. One main disadvantage is it has a longer curing time when compared to most of thermosetting adhesives [8].

Thermosets have a three dimensional polymer structure. Thermosets undergo chemical reactions and form chemical cross links between the polymer chains. Due to these they resist deformation even at very high temperatures. Most of the thermosets do not require solvents. Due to the reduced use of solvents, there is a minimum possibility of formation of bubbles and voids due to emissions during curing. The main drawbacks of thermosets are limited shelf life and poor rework ability.

ECAs can offer numerous advantages such as fewer processing steps which reduce processing cost, lower processing temperature which makes the use of heat-sensitive and low cost substrate possible, and fine pitch capability. The main limitations of the ICA technology are lower conductivity, unstable contact resistance with non-noble metal finished components, and poor mechanical impact performance [9].

2.1.1 Isotropic and Anisotropic Conductive Adhesives

Isotropic conductive adhesive

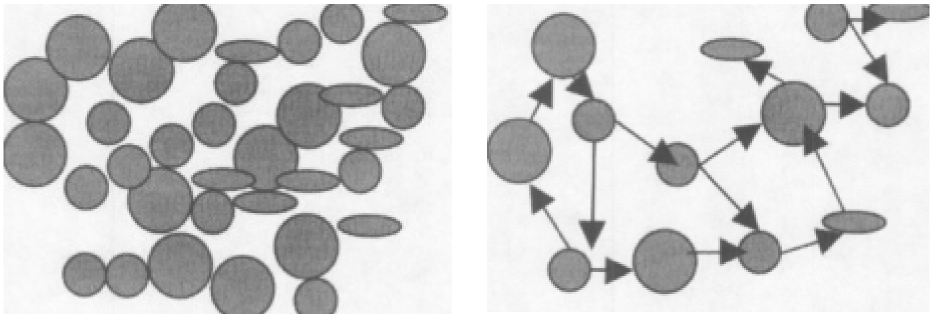
Isotropic conductive adhesives consist of polymer resin such as epoxy and conductive fillers particles such as silver flakes. Most of the commercial ICAs are based on thermosetting resins [10]. The most common binders are thermoset epoxies due to their properties, such as excellent adhesive strength, good chemical and corrosion resistances and low cost, while thermoplastics are usually added to allow softening and rework under moderate heat [10]. Most commonly used conductive fillers are silver, gold, nickel, copper and carbon in various sizes and shapes. The conductive fillers provide the composite with electrical conductivity through contact between the conductive particles. Silver flakes are the most commonly used conductive fillers because of the high conductivity, simple process and the maximum contact with flakes. Nickel- and copper-based conductive adhesives generally do not have good resistance stability, because both nickel and copper are easily oxidized. Even with antioxidants, copper-based conductive adhesives show an increase in bulk resistivity after aging, especially under high-temperature and -humidity conditions. The conductive fillers in the ICA composite make contact with each other and form a conductive path to provide electrical conductivity. To ensure good electrical conductivity the volume fraction loading of the conductive filler particles must be higher than the percolation threshold. The conductive filler provides the electrical conductivity and the polymer resin gives the mechanical strength between the component and the substrate. An increase in loading will result in better electrical conductivity; on the other end it decreases the mechanical strength of the polymer resin. In a typical ICA the volume fraction loading conductive fillers is about 25% to 30% [11]. For silver the corresponding weight percent is 78% to 82%.

Anisotropic conductive adhesive

Anisotropic conductive adhesives are pastes or films of thermoplastics. The volume fraction of filler in ACAs is 0.5% to 5% which is far below the percolation threshold, thus there is no continuous electrically conductive path in ACAs before bonding. The component terminations are pressed against the corresponding pads on the substrate in order to compress the conductive particles trapped between the component terminations and substrate pads in order to form the electrically conductive path during bonding. ACAs require high heat and pressure during the bonding process. After bonding the ACAs become electrically conductive in one direction and remain insulating the other direction. The use of ECAs is a no-clean process, since there is no flux involved in the assembly process, The ECAs work well with all types of board finishes [12].

2.1.2 Percolation and Conductivity in ICA

There are two conductive pathways for isotropic conductive adhesives as shown in Figure 1. One is genuine conduction, caused by particle-to-particle contact within the polymer matrix. The other is percolation, which involves electron transport brought about by quantum-mechanical electron tunneling between particles close enough to allow dielectric breakdown of the matrix [12]. The conductivity of isotropic conductive adhesives depends on uniform dispersion of the filler particles to form a conductive path in the polymer matrix. Most of the traditional metal filled conductive adhesives require 25 to 30% volume fraction to overcome the percolation threshold and ensure electrical conductivity [11]. However for less than 1% volume fraction of carbon black in the resin can cause electrical conduction. The largest reduction in volume resistivity is observed at0.25 wt% which means the percolation threshold is less than 0.25 wt% [20].



1(a) Particle-to-particle   1(b) electron tunneling

Fig 1: Conduction Mechanism in conductive adhesives [12]

2.2 Carbon Nanotubes

Carbon nanotubes are added as conductive fillers in the ECA. High aspect ratio, large surface area multiwall carbon nanotubes (MWCNTs) have the potential as ICA filler particles to reach the percolation threshold with small volume fraction loading compared to silver filled adhesives. The MWCNT used in the present study have aspect ratios of 2500:1.

Carbon nanotubes are molecular-scale tubes of graphitic carbon with outstanding properties. They are among the stiffest and strongest fibers known, and have remarkable electronic properties and many other unique characteristics. They are chemically stable, mechanically very storing and conduct electricity. For this reason they open up new perspectives for various applications, such as nanotransistors in circuits, field emission displays, artificial muscles or added reinforcements in alloys. They exhibit extraordinary strength and unique electrical properties and are efficient conductors of heat [13].

2.2.1 Geometry of Carbon nanotubes

An ideal nanotube is a hexagonal network of carbon atoms that has been rolled up to make a seamless cylinder. Just nanometer across, the cylinders can be tens of microns long, and each end is "capped" with half of a fullerene molecule. Single-wall nanotubes have a fundamental cylindrical structure, and these form the building blocks of both multi-wall nanotubes and the ordered arrays of single-wall nanotubes called ropes. The atoms in CNT are arranged in hexagons, the same arrangement as in graphite. In graphite the hexagons lie in flat sheets on top of each other while in a nanotube, the sheet is rolled into a tube [14].

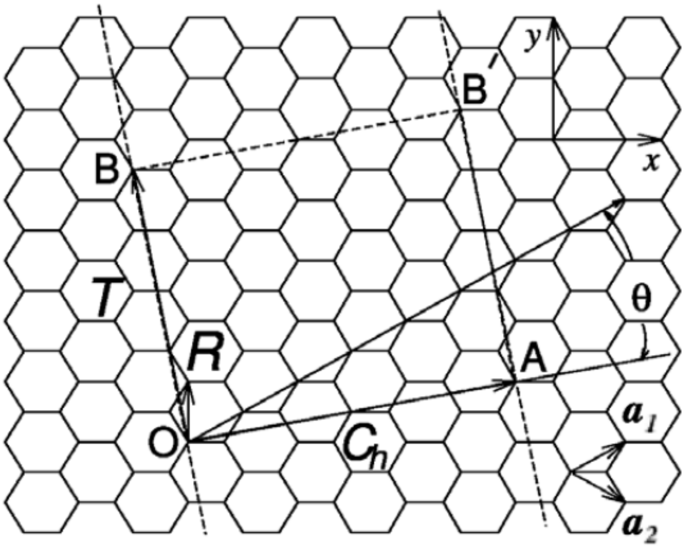


Figure 2: Unrolled hexagonal lattice of a carbon nanotube [14].

.There are three unique geometries of carbon nanotubes. They are also referred to as flavors. The three flavors are armchair, zigzag, and chiral [e.g. zigzag (n, 0); armchair (n, n); and chiral (n, m)]. These flavors can be classified by how the carbon sheet is wrapped into a tube as shown in the below figure 3.

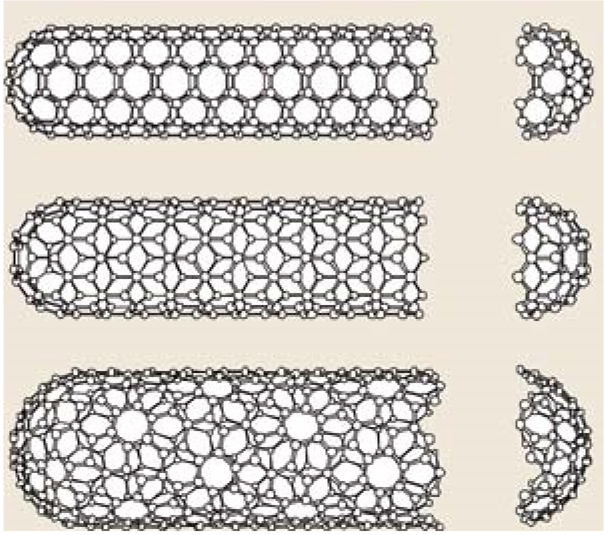


Figure 3: Three distinctive arrangements of carbon in SWNTs [14].

Shown in Figure 3 is a (5, 5) armchair nanotube (top), a (9, 0) zigzag nanotube (middle) and a (10, 5) chiral nanotube. The diameter of the nanotubes depends on the values of *n* and *m* [14].

There are two main types of nanotubes, single walled nanotubes (SWNTs) which are one layer of carbon hexagons and multiwalled nanotubes (MWCNTs) wherein one single tube is encased inside several larger tubes. The arrangement of the single walled and multi walled carbon nanotubes are shown in Figure 4.

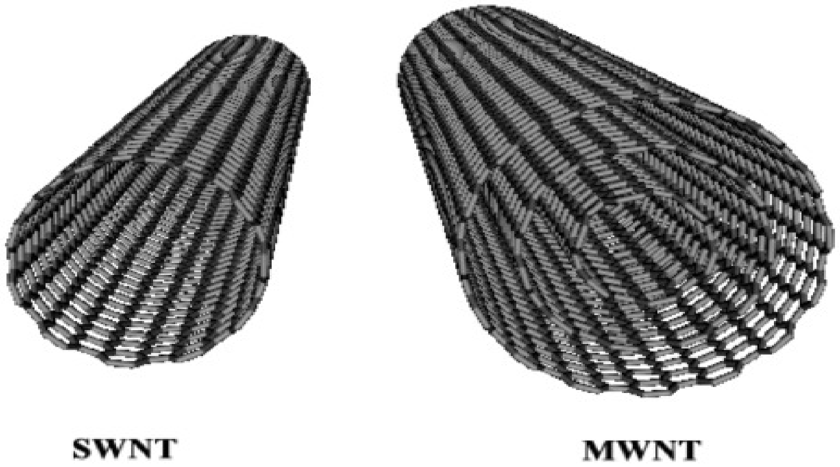


Fig 4: Single walled and Multi walled Carbon Nanotubes.

2.2.2 Single walled nanotubes (SWCNTs)

These can be considered as long graphite sheets. Nanotubes generally have a length to diameter ratio of 1000 so they can be approximated as one dimensional structure. Single walled nanotubes are a very important variety of carbon nanotube because they exhibit important electric properties that are not shared by the multi walled carbon nanotube (MWCNT) variants. Single walled nanotubes are the mostly likely candidate for miniaturizing electronics past the micro electromechanical scale that is currently the basis of modern electronics [13].

The most basic building block of these systems is the electric wire, and SWNTs can be excellent conductors. One useful applications of SWNTs is in the development of first intramolecular field effect transistors (FETs). Single walled nanotubes are still very expensive to produce, and the development of more affordable synthesis techniques is vital to the future of carbon nanotube technology [13].

2.2.3 Multi walled Carbon Nanotubes (MWCNTs)

Multi walled nanotubes consist of multiple layers of graphite rolled in on them to form a tube shape. These can be considered as a collection of concentric SWNTs with different diameters, lengths and properties. Multi walled nanotubes consist of multiple layers of graphite rolled in on them to form a tube shape. The distance between each layer is approximately 0.34 nm, slightly larger than the interlayer distance of graphite sheets and the layers are coupled to each other through van der Waals forces [13].

High aspect ratio, large surface area multiwall carbon nanotubes (MWCNTs) have the potential as ICA filler particles to reach the percolation threshold with small volume fraction loading. The MWCNT used in the present study have aspect ratios of 2500:1. MWCNT are electrically conductive independent of synthesis method and have extremely high strength to weight ratio offering the potential to improve the stiffness of the polymer matrix. The unique mechanical and electronic properties of multiwall nanotubes are proving to be a rich source of new physics and could also lead to new applications in materials and devices.

2.2.4 Properties of Carbon nanotubes

Multiwall carbon nanotubes (MWCNT) are metallic, whereas single wall carbon nanotubes (SWCNT) may be metallic or semiconducting depending on the chirality of the grapheme walls. The measured electrical conductivity of metallic carbon nanotubes is in the order of 10^4 S/cm [15]. The thermal conductivity of carbon nanotubes at room temperature can be as high as 6600 W/mK [16]. Carbon nanotubes have a Young's modulus of approximately 1 TPa and a maximum tensile strength of nearly 30 GPa [17]. The density of MWCNT is 2.6 g/cm3 and the density of SWCNT range from 1.33 g/cm3 to 1.40 g/cm3 depending on the chirality [18]. Carbon nanotubes are the strongest and stiffest materials on earth, in terms of tensile strength and elastic modulus respectively.

Blending the extreme properties of carbon nanotubes into polymer matrix composites has tremendous potential for increasing strength, strength to weight ratio, thermal conductivity and electrical conductivity of the bulk polymer.

2.3 Different Surface Finishes of Printed Circuit Boards (PCBs)

RoHS directive impacts not only the solder alloys that one is permitted to use, but also many other aspects of the electronics assembly process, including the selection and compatibility of board fabrication materials. The selection of an appropriate surface finish plays an important role in printed circuit board manufacturing to ensure solder ability, reliability and high yield assembly of through whole and surface mount products. Surface finish is applied to copper pads and exposed traces for protection during storage and transportation from the time of board manufactured to the time the circuit is assembled. Therefore, the surface finish must maintain the solder ability of the board and protect the metal areas from contamination and oxidation, thus increasing its shelf-life. PCB Surface Finishes vary in price, availability, shelf life, reliability and assembly processing. While each finish has its own benefits, in most cases, the process, product or environment will dictate the surface finish that is best suited for the application. All Printed Circuit Boards (PCBs) have copper finishes on their surface. If the copper finish is left unprotected the copper will oxidize and deteriorate, there are various protective finishes available. The most prevalent are; Hot Air Solder Leveling (HASL), Organic Solder Preservative (OSP), Electroless-Nickel Immersion Gold (ENIG), Immersion Silver and Immersion Tin. We carried out the tests on Immersion Silver, Immersion Tin and Electro less-Nickel Immersion Gold [19].

2.3.1 Electroless Nickel Immersion Gold

ENIG coatings have been used with great success on many boards despite the high per unit cost. It has a flat surface and excellent solder ability. The main drawback is that the electroless nickel layer is brittle and has been found to break up during mechanical stress. This effect is known in the industry as 'black pad' or 'mudflat cracking'.

The advantage of ENIG is excellent solder ability, coplanar - flat surface, excellent shelf life and withstands multiple reflows. The disadvantages are higher cost, 'black pad' issue, manufacture process uses cyanide and other hazardous chemicals [19].

2.3.2 Immersion Tin

Immersion tin is a newer alternative surface finish, with many similar characteristics to its silver counterpart. However, there are major health and safety issues to consider. It is mainly used in Europe and Asia whilst its use in the US is restricted due to the concern over the thiourea used in tin solution (a suspected carcinogen).

The advantages of immersion tin are good solder ability, flat surface area and relatively low cost. The disadvantages are health and safety concerns and limited number of heat cycles [19].

2.3.3 Immersion Silver

Immersion silver is a relatively recent addition to the PCB finish. Its main use has been in Asia and is gaining popularity in North America. Europe now has an emerging market. During the soldering process the silver layer dissolves into the solder joint leaving a tin/lead/silver alloy on the copper which provides very reliable solder joints for Ball Grid Array (BGA) packages. The contrasting color makes it easy to inspect, as opposed to OSP. It has also been a drop in replacement for HASL for soldering operations. There is although an underlying issue over silver 'migration'.

The advantages of immersion silver are good solder ability, coplanar - flat surface, and 'drop in' replacement for HASL. The disadvantages are insufficient data as it is recently added to the market.

Chapter 3 MWCNT Epoxy and Epoxy/Heloxy Composite Preparation

3.1 Materials

The epoxy resin and viscosity modifier used for the experiments were diglycidyl ether bisphenol-F (Epon 862, Shell Chemical, USA) and neopentyl glycol diglycidyl ether (Miller-Stephenson Chemical, USA), respectively. The curing agent used is methyl hexahydro phthalic anhydride (MHHPA, Miller-Stephenson Chemical, USA). Cyanoethyl-2-ethyl-4 methylimidazole is used as a catalyst (2E4MZ-CN, Shikoku Chemical, Japan). Multiwall carbon nanotubes grown by chemical vapor deposition were obtained from University of Kentucky Center for Applied Energy (CAER). As grown MWCNT have an average length of 50 μm and diameter of 20 nm. Shatter milled MWCNT were processed at CAER by grinding for 10 minutes in a shatter mill.

3.2 MWCNT-Epoxy composite preparation

The resin to curing agent to catalyst ratio is 100:85:0.5 for each batch of conductive adhesive. Each component in a 20 gram batch is added by weight to a polyethylene container and a Thinky AR-250 planetary mixer blends the adhesive. The planetary mixer generates large continuous centripetal forces (up to 400G) by compound motion of the revolving centrifuge and the counter-rotation of the material container. The high shear mixing action achieves uniform distribution and removes air bubbles from the mixture. No blending tool is required and each batch is mixed in a new clean container to prevent cross contamination. First, the resin, curing agent and catalyst are added to the sample container and then the desired weight percent loading of MWCNT are added. Each mixing cycle includes five minutes at 800 rpm rotation and 2000 rpm revolution rates followed by a three minute defoaming cycle at 60 rpm rotation and 2200 rpm revolution rates. Batches are stored in a refrigerator at 40°C. At room temperature, the mixtures have a pot life of approximately 72 hours. Formulations are allowed to come to room temperature and remixed prior to sample fabrication using a 3 minute mix and 2 minute defoam cycle. Pads are stencil printed onto the ceramic substrates using an Aremco 3230B screen printer. All samples are held at 80°C for 4 hours to evaporate the solvents in the epoxy and then cured at 150°C for 45 minutes.

3.3 SMWCNT-Epoxy composite preparation:

Shatter milled MWCNT were processed at CAER by grinding for 10 minutes in a shatter mill. The Nanotubes thus formed were shorter than the as grown nanotubes. The SEM picture of the shatter milled MWCNTs are as shown in the Figure 5. The length of the SMWCNTs is approximately halved when compared to the original MWCNT.

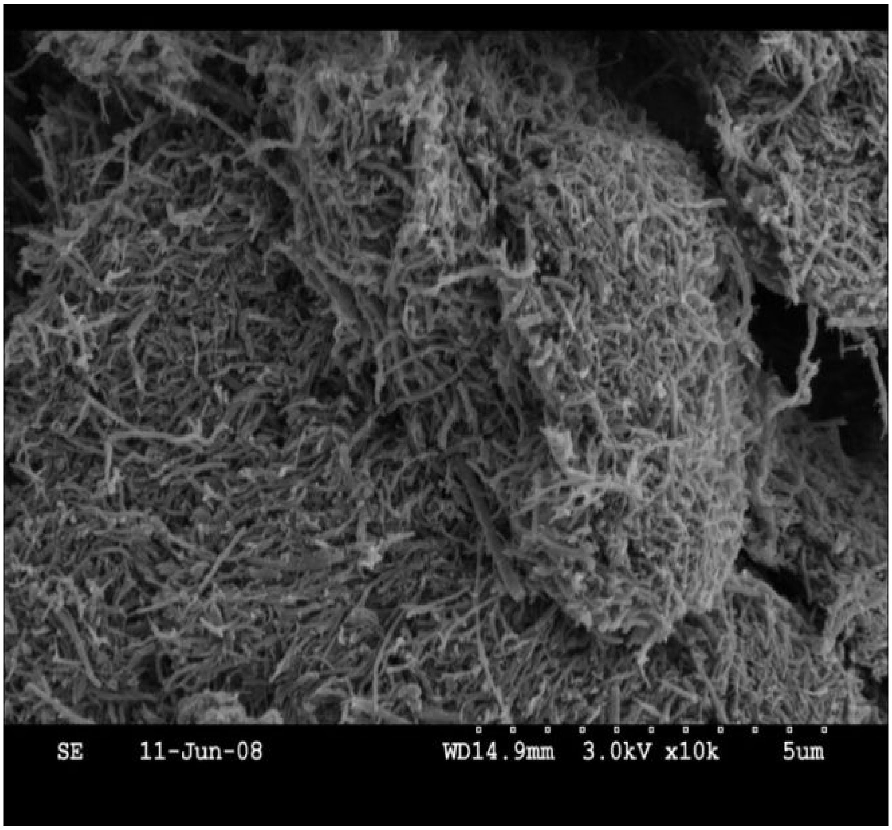


Figure 5: Scanned Electron Microscopic image of a shatter milled MWCNT.

The preparation of this composition is same as that of the MWCNT- epoxy composite preparation.

3.4 MWCNT-epoxy: Heloxy composite preparation

While the epoxy resin has a room temperature viscosity of 25-45 P, the Heloxy viscosity modifier has a viscosity of 13-18 cP. The resin portion of the formulation was varied to include Heloxy modifier at epoxy to Heloxy ratios of 30:70, 40:60, 50:50, 60:40 and 70:30. First, the epoxy and Heloxy modifier are taken in required proportions and are mixed with the curing agent and catalyst in desired weight ratio in a clean container. Then the total weight of the mixture is measured and then the corresponding weight of MWCNTs for desired %loading is calculated and CNTs are added to this mixture. Now the total weight along with the container is determined and that weight is compensated in the Thinky mixer. Thus 8wt%, 12wt%, 14wt% MWCNT mixtures are prepared with varying epoxy-Heloxy compositions as 2:3, 3:7, 1:1, 3:2,7:3 for each wt % except for 14 wt% which was limited only to 2:3, 1:1, 3:2 ratio of epoxy-Heloxy.

Chapter 4 Experimental Setup

4.1 Volume Resistivity Measurement

The electrical conductivity of a conductive adhesive is often evaluated as the volume resistivity (ohm-cm) of the material. For bulk material, volume resistivity can be measured by the method shown in Figure 21. In Figure 21, the bulk material is placed between the two electrode plates. A digital ohmmeter is connected to the two electrode plates and reading is taken once the display stabilized. Volume resistivity can be calculated using the following formula:

$$R_{vol} = (R \times l \times w) / t$$

Where

R_{vol} = volume resistivity (ohm-cm)

R = measured resistance (ohm)

l = length of the electrode plate (cm)

w = width of the electrode plate (cm)

t = thickness of the conductive adhesive (cm)

To measure the volume resistivity, the mixed epoxy/MWCNT composites can be either injection molded to make in bar shape and use the method discussed above or printed as a film and use four-point probe method. In our experiment, MWCNT/epoxy composites were stencil-printed onto aluminum oxide substrates (4 inches × 4 inches, CoorsTek, USA) to form uniform film samples. On the stencil pattern were six 1 cm × 1cm Figure 6 square openings and two substrates were printed for each epoxy/MWCNT loading, thereby ten squares for each loading were printed. Samples were held at 80 °C for 4 hours to out gas and then cured at 150 °C for 30 minutes. The volume resistivity of each sample was calculated by multiplying the sheet resistance by the average thickness of the epoxy/MWCNT composite layer.

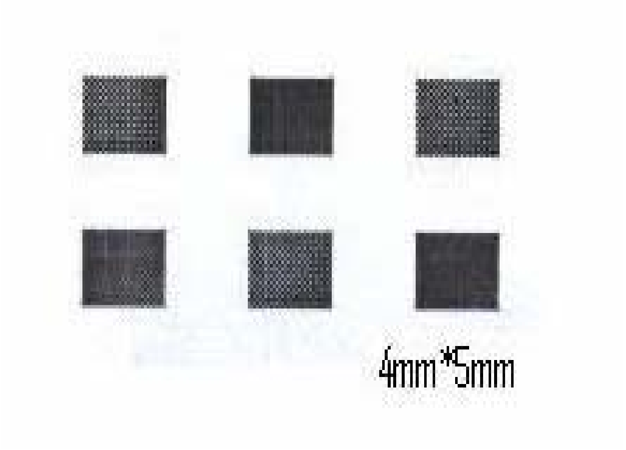


Figure 6: Volume Resistivity Pattern

A four-point probe tester (Signatone, USA) was used to record the voltage drop with the injected current. The sheet resistance (Rs) is proportional to the ratio of the voltage, V to the current, I. The schematic of the four point probe is shown in Figure 7.

$$R_s = C.V/I$$

$$C=4.53 \text{ (for infinite film)}$$

A stylus profilometer (Alpha-Step 500, Tencor, USA) was used to scan each square in four directions to achieve the average thickness, t. Figure 22 illustrates the volume resistivity measurement sample and arrows indicate scan directions for stylus profilometer thickness measurements. The sheet resistance was converted to volume resistivity, ρv by multiplying by thickness, t (in cm). Volume resistivity for each of the ten squares was calculated and averaged to determine the volume resistivity for each loading.

$$\rho_{v} = 4.53 \times (V/I) \times t$$

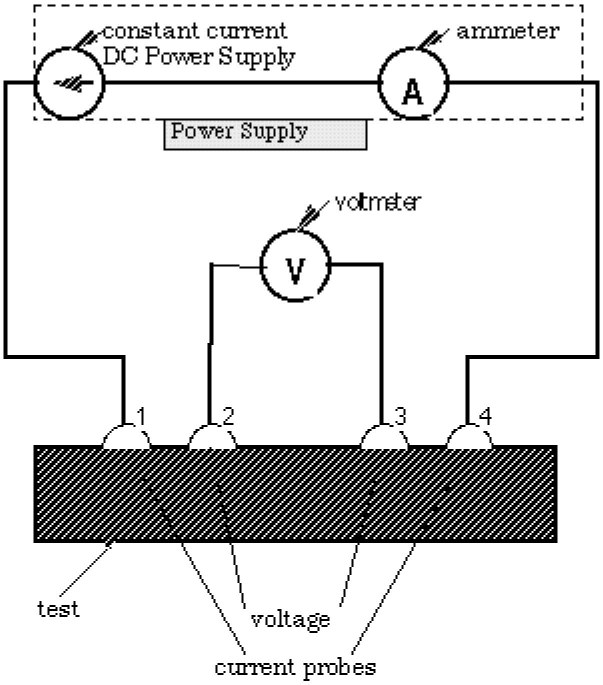


Figure 7: Schematic of four point probe

4.2 Contact Resistance Measurement

To measure the contact resistance of MWCNT filled ICAs, initially the adhesive is stencil printed using a stencil printer on to the pads of chosen printed circuit board and later,1206 size zero ohm resistors are mounted on the pads using a pick and place machine which uses a vacuum tip to hold the resistors. These resistors are mounted in daisy chain patterns. There are 10 zero ohm resistors on each daisy chain and 4 such daisy chains are fabricated onto the test board [22]. During the curing process, certain amount weight was placed on the sample for applying pressure. The weight enlarges the contact area while the height between the pads is reduced so that more contact points between pads and MWCNT are achieved there by reducing the contact resistance. The sample is placed in a heated oven for 240 minutes at a temperature of 85 ^{o}C and then the temperature is adjusted to 150^{0}C and sample is cured for 40 minutes. Then the oven is turned off and sample is taken out for resistance measurement. The schematic of the Contact resistance pattern is shown in the Figure 8.

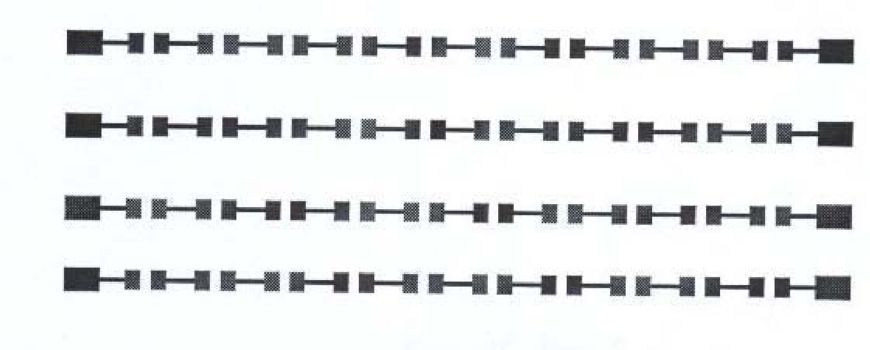


Figure 8: Contact resistance Measurement test board.

A digital multimeter RSR MAS830 is used to measure the resistance for each daisy chain and the resistance for each daisy chain is calculated and divided by 20 to obtain the average contact resistance for each set of 10 resistors. Hence the reported contact resistance is calculated by averaging the resistance values for 4 daisy chains. Samples on different surface finishes (silver, gold, tin) are prepared and each with different %MWCNT loadings such as 2 wt% MWCNT to 6 wt% MWCNT and different mixtures of epoxy and different epoxy and heloxy (E: H) ratios.

4.3 Lap Shear Strength Measurement

To evaluate the mechanical degradation of the polymer matrix caused by adding MWCNT, a lap shear test was conducted by using an Instron 4442 tensile tester (Instron, USA). The test sample was prepared by attaching two copper clad printed circuit board tabs using the epoxy/MWCNT composite. The lap area was 0.25 inch × 0.25 inch. Figure 9 illustrates the test sample. The amount of adhesive dispensed on the contact area was controlled by using a pressure dispenser. The tensile tester grips the 1×1 inch tabs and pulls until the composite attached lap area fails. The break force was recorded and divided by the lap area to calculate the lap shear strength (psi). Five samples were prepared to determine the average lap shear strength for each loading of MWCNT. Tabs were milled from bare printed circuit boards using a MITS FP-21T milling machine. To prepare the samples, tabs were attached by MWCNT/epoxy composite and MWCNT/epoxy/heloxy composite [23].

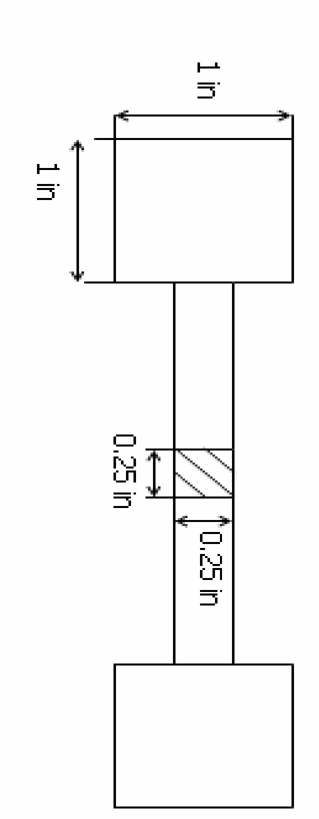


Figure 9: Lap shear test sample

4.4 Die shear Test Measurement

Die shear testing is usually used to determine the adhesion strength between the semiconductor die and the package substrate. In this test, the whole assembly is tightly fastened, force parallel to the substrate is gradually applied on the edge of the die, the break force is measured for each die-substrate assembly and the type of failure is determined by visual examination of the residual. Mil-Std-883 Method 2019 [21] is the most widely-used die shear test standard. To conform to this test standard, the following requirements for the test equipment must be fulfilled. First, the tool contacting the die should uniformly distribute the force to the full length of an edge of die. Second, the contact tool must be perpendicular the die attach plan. Third, a rotational substrate holding fixture is needed to ensure the die edge and contact tool are parallel. Forth, a binocular microscope (10X min magnification) and lighting system is needed to observe the die and contact tool interface while the test is being performed. The direction of the applied force must be perpendicular to the die edge and parallel to the substrate plane. The contact tool must only contact with the die edge and avoid contacting the die attach material or the substrate. There are three die separation modes: the die cracks and die residual remains, die separates from the die attach material and die attach material separates from the substrate. Schematic of the die shear test board with 32 pads is shown in Figure 11 below.

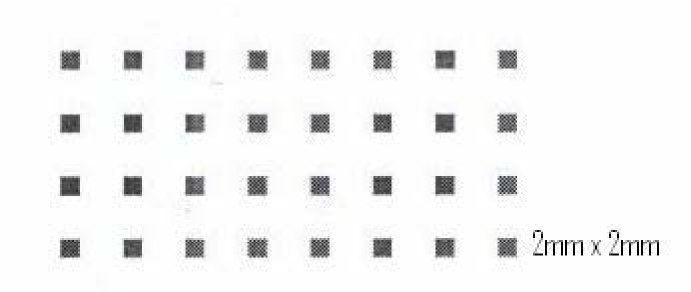


Figure 10: Schematic of die shear test sample

MWCNT filled conductive adhesive is stencil printed according to the pattern shown in Figure. A manual pick and place machine is used to place the silicon die on the metal pads and the assembly is cured. The silicon die size is 0.06 inch × 0.05 inch = 0.003 inch^2. Testing for the Die shear samples were performed at the NASA jet propulsion laboratory.

4.5 Aging Experiments

The aging experiments were conducted on the volume resistivity samples. The samples were tested in order to see how time and temperature effect the volume resistivity of the samples. The samples were tested after 24 hours at 85°C in an oven and 72 hours at 85°C in an oven.

All the experiments were conducted on the 12% MWCNT loading across all the epoxy : heloxy compositions.

Chapter 5 Results and Discussion

5.1 Volume resistivity Results

5.1.1 MWCNT Loadings with Epoxy composite vs. Volume Resistivity

The volume resistivity was measured for varying weight percentages of MWCNT loadings in MWCNT/Epoxy composition. Table 1 shows the measured volume resistivity of the epoxy/MWCNTs composites with different loadings. As the weight percentage of MWCNTs was increased, it was observed that the resistivity decreased. As it can be seen in Figure 12. It was observed that the viscosity of the epoxy/MWCNT composite increases with the increase in loadings. And above 12 wt% the composite becomes too viscous to process by screen printing, stencil printing or pressure dispensing.

From the Figure 12 it can be observed that the volume resistivity decreases with the increase in the MWCNT loading. I was also observed that the value does not change much from 8% to 12% loadings, which means the processability is limited above 8%. Increasing the weight percentage of MWCNT reduces the resitivity as expected. Contact between nanotubes form conductive paths through the composite structure. As more nanotubes are added the number of conductive paths increases. The variance also decreases with increased loading indicating that the resistivity measurement becomes more reliable with increased loading. In those formulations an upper limit of approximately 12 wt% MWCNT was observed, beyond which the mixtures were too viscous to process by printing or dispensing.

5.1.2 SMWCNT Loadings with Epoxy composite vs. Volume Resistivity

The SMWCNTs were shatter milled in University of Kentucky Center for Applied Energy (CAER). They are grinded in shatter mill for 10 minutes and the length is almost reduced to half of the original length.

Volume resistivity of the shatter milled multiwall nanotubes was also observed. The volume resistivity was measured at higher load percentages of SMWCNTs. The high resistivity at the lower percentages of SMWCNT could be due to the lower aspect ratio making them less likely to contact each other to form an electrical path. Higher loadings are possible due to the lower viscosity and increased process ability of the SMWCNT. But it was observed that the resistivity was much higher than MWCNT/epoxy composite.

Table 1: Measured Volume resistivity for epoxy/MWCNT composites for Different MWCNT loadings

Loading (Wt %)	Volume resistivity	Standard deviation
2%	16.79	4.34
3%	11.73	4.94
4%	8.82	1.46
5%	4.61	0.88
6%	4.46	0.82
7%	3.37	0.61
8%	2.72	0.38
12%	2.62	0.32

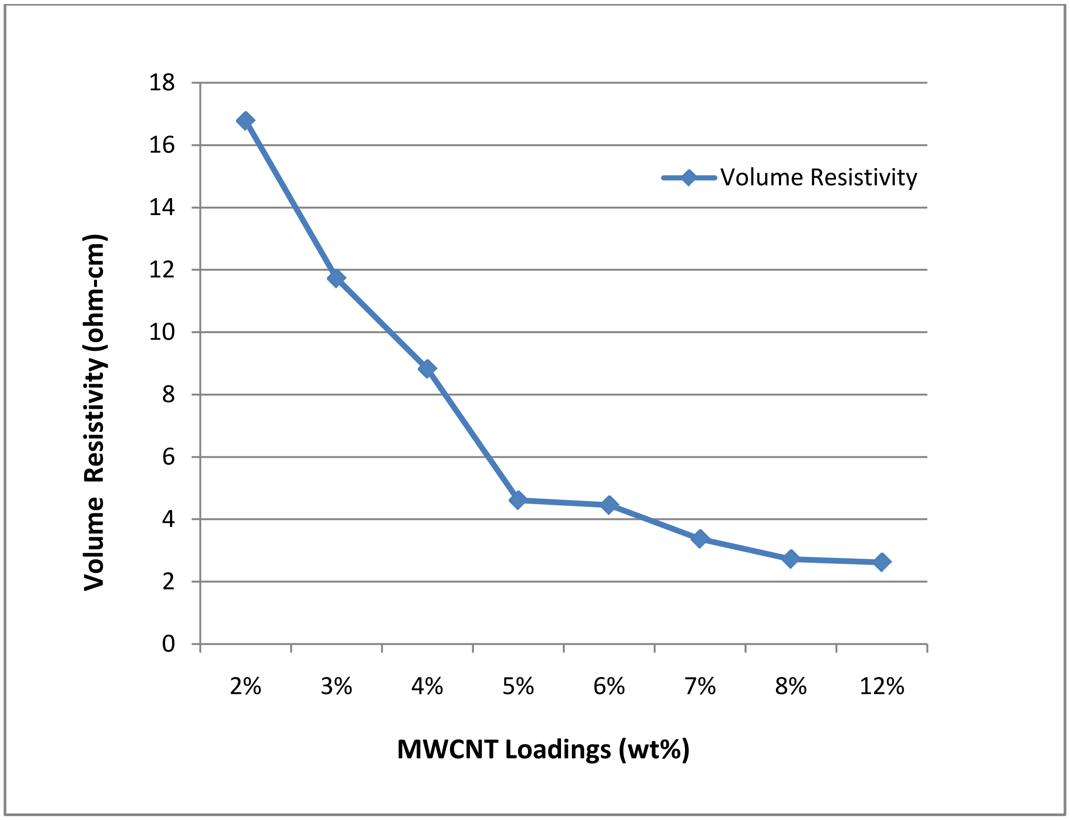


Figure 11: Average Volume resistivity vs. weight percentage of MWCNT in Epoxy Mixtures.

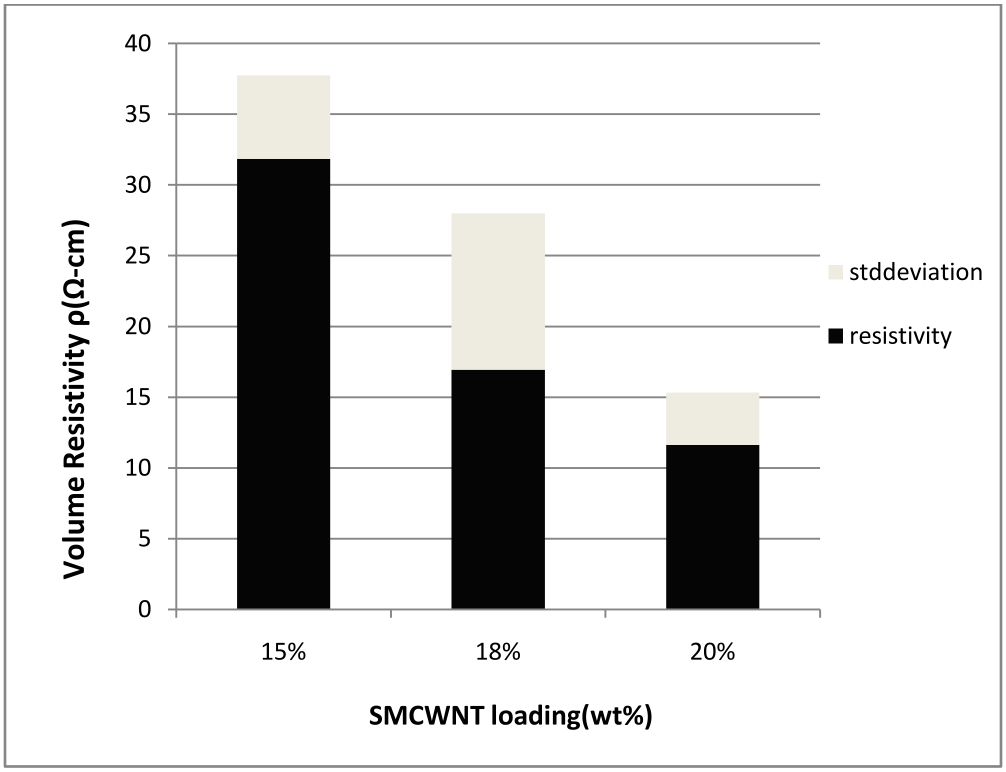


Figure 12: Average volume resistivity vs. weight percentage of SMWCNTs in Epoxy mixture.

5.1.3 MWCNT Loadings with Epoxy/Heloxy composite vs. Volume Resistivity

The volume resistivity was also measured by varying Epoxy-Heloxy composition. The heloxy is a viscosity modifier which when added decreases the viscosity of the composite. We then varied the weight proposition of the epoxy heloxy from 40:60, 50:50, and 60:40 for 14% loadings of MWCNTs. The 70:30 and 30:70 was limited to 12% MWCNTs. The variation of the resistivity for different weight percentages and the proportion of Epoxy-Heloxy composition are plotted as shown in the Figure 14. The 1:1 composition demonstrates a decreasing resistivity with increasing MWCNT loading, while the 2:3 and 3:2 ratios show minimum resistivities at 12 wt%.

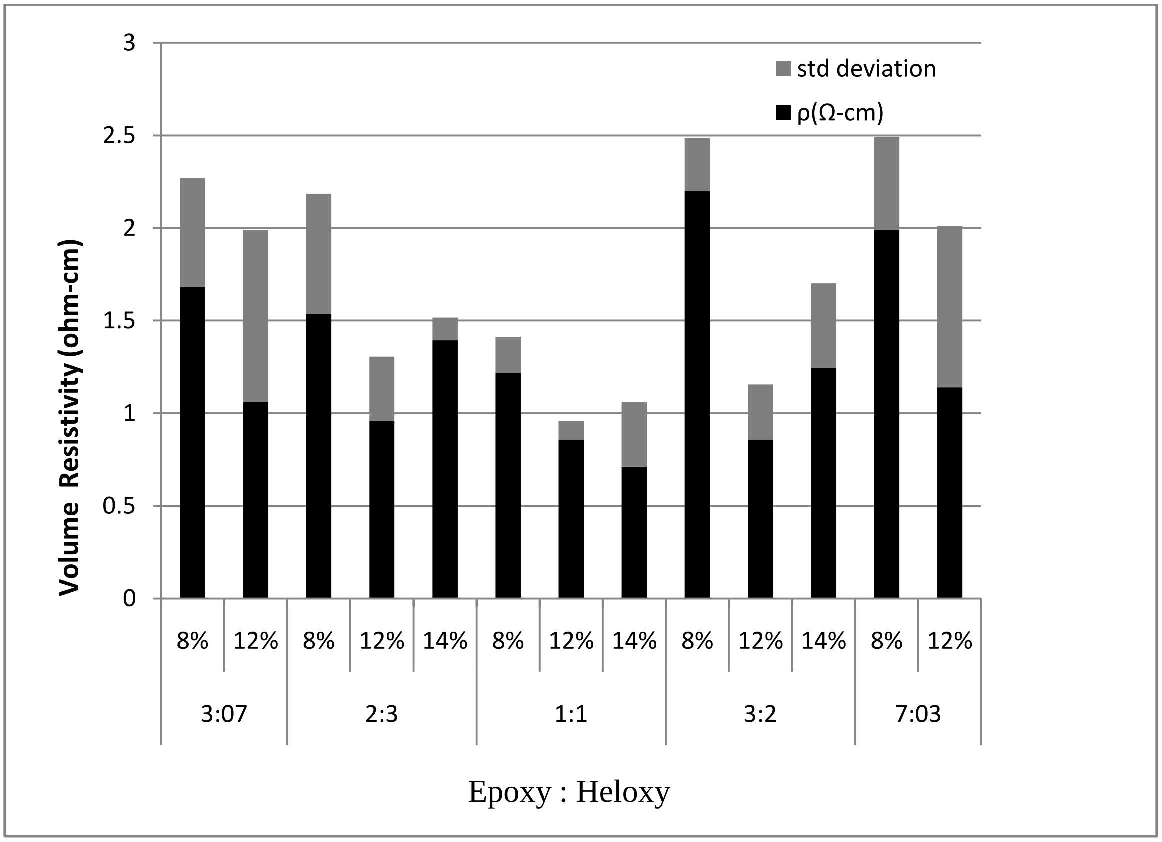


Figure 13: Average volume resistivity versus Epoxy to Heloxy ratio and weight percentage of MWCNT in Epoxy-Heloxy mixtures.

5.2 Contact Resistance Results and Discussion

5.2.1 Contact resistance results for MWCNT epoxy composite

Contact resistance measurement tests were conducted with 50um long MWCNT. The following table shows the experimental results for contact resistance measurements for various loadings on three different metals likely tin, gold and silver. This set of results is taken immediately after curing the sample. The results obtained from the experiments are shown in Table 2.

Although the contact resistance daisy chains allow us to average 80 contact points per test board, the results are often dominated by a few high resistance joints. The resulting data includes large standard deviations from various sample combinations. All three surface finishes silver, tin and gold displayed the extreme variance leading us to focus on tin coated boards as the least expensive test circuits. Unlike the volume resistivity graphs, the data presented in Figure 15 for average contact resistance does not show a clear trend as a function of loading wt%.

One explanation for the variability in the contact resistance data may be the absence of flux. Lead-tin and lead free solders contain thermally activated flux compounds to remove native oxides from the circuit board pads and component leads at temperatures below the melting point of the solder. The MWCNT filled adhesives do not contain a fluxing agent and rely primarily on pressure applied during curing to form electrical contacts between the component leads, nanotubes and circuit board pads. Conversely, MWCNT filled adhesives do not produce flux residues and therefore eliminate the need for cleaning and reduce long term corrosion of printed circuit assemblies.

It is observed that applying pressure on zero ohm resistors during curing dramatically improves conductivity. It is also observed that the contact resistance is variable with % loading and surface finishes. All the values are high for use as a solder replacement. In general, the obtained values from figure 15 indicate that contact resistance for gold is highest and for silver is least in the order Rag > Rsn > RAu.

A shift in contact resistance is also observed for each of these samples after room temperature storage illustrating that contact resistance increases with aging time. This may be because of galvanic corrosion of metal after moisture penetrated into cured polymer matrix and condensed at the interface between ECA and metal surface. Galvanic corrosion happens only under wet conditions with the presence of an electrolyte solution. Moisture absorbed by the cured ECA diffuses into the interface between the ECA and the non noble metal and forms an electrolyte solution. Metal oxide formation caused by galvanic corrosion of the non noble metal at the interface is responsible for the contact resistance increase.

Table 2: Contact resistance on different surface finished PCBs for different MWCNT (wt%)-Epoxy composite.

Loading (wt %)	Silver		tin		gold	
	Average resistance	**Std Deviation**	**Average resistance**	**Std Deviation**	**Average resistance**	**Std Deviation**
2%	127.5	73.6	239.625	73.5	X	X
3%	361.125	77.9	387.5	381.5	906.667	100.5
4%	216	102.9	665	325.1	550	317.4
5%	258.25	137.1	478.75	192.6	488.75	99.53
6%	211.25	88.4	353.75	36.1	812.5	238.5

5.2.2 Contact resistance results for MWCNT epoxy: heloxy composite:

The contact resistance values for the epoxy composite were svery high so we tried additional formulations using the heloxy, viscosity modifier. The combinations were 30:70, 40:60, 50:50, 60:40, and 70:30, as before. The MWCNT loadings that we tested were 8%, 12% and 14%. All the loadings were tested on three different PCB surface finishes silver, tin and gold. The values are represented in the following graphs. There are a many values so for better understanding the data were grouped by MWCNT loading.. The graphs are shown in Figure 15, Figure 16 and Figure 17 for 8, 12 and 14% respectively.

From the above figures, it is clear that by adding Heloxy to the composition, the contact resistance values are reduced by 1/10 of the previously observed results obtained with epoxy-MWCNT composition. As Heloxy acts as viscosity modifier of Resin, the % loading of the MWCNTs in the mixture is able to be increased. Previously, the % loading is limited to 12% with only epoxy, but by adding Heloxy, the loading limit is increased to 14 wt%. Above the loading limit, the MWCNTs do not disperse well and the adhesive is not processable by screen printing. It is observed that of all metals, tin has shown least contact resistance with E-H composition. The minimum contact resistance with least variability for all the metals for 8% loading is observed at 2:3 ratio of epoxy-Heloxy, for 12% loading at 3:2 Composition and for 14% at 3:2.

It can also be observed that silver has shown least contact resistance of all the three metals. Gold has the highest value. Tin has shown consistent values at 12 wt% loading. This type of behavior from gold is seen at 14 wt% loading. Silver has shown decreasing contact resistance with increase in Heloxy ratio for 14 wt%. Though the obtained values are not consistent enough for applications, these variations in the values may be due to outlier contacts in the chain. This presence of faulty resistance may increase the average value of the chain, thus increasing the resistance per contact on the whole. The variations in resistance for same batch of mixture for all the three metals may be because of the pot life of the mixture and its process ability after it is stored.

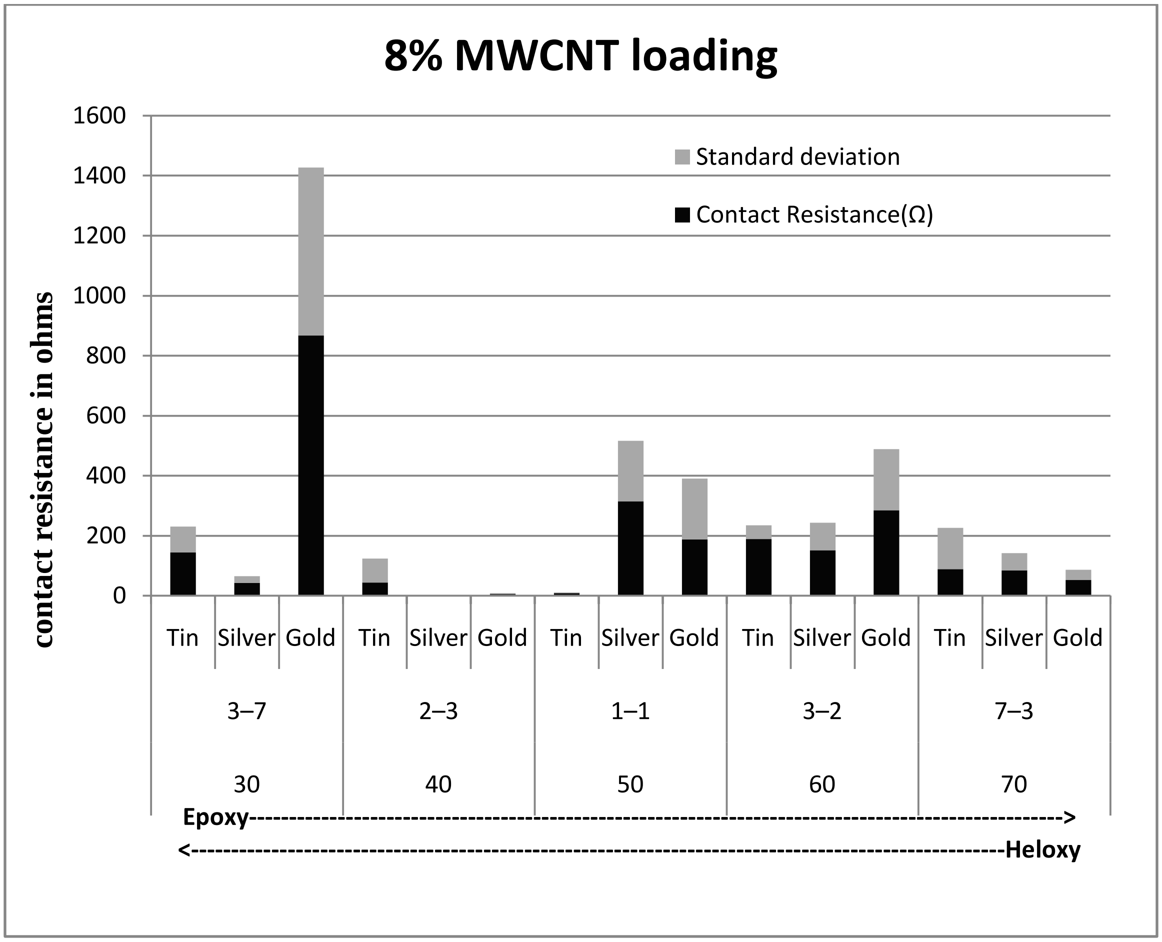


Figure 14: Average Contact resistance on different surface finished PCBs with 8wt% MWCNT loading and varying epoxy-Heloxy ratios.

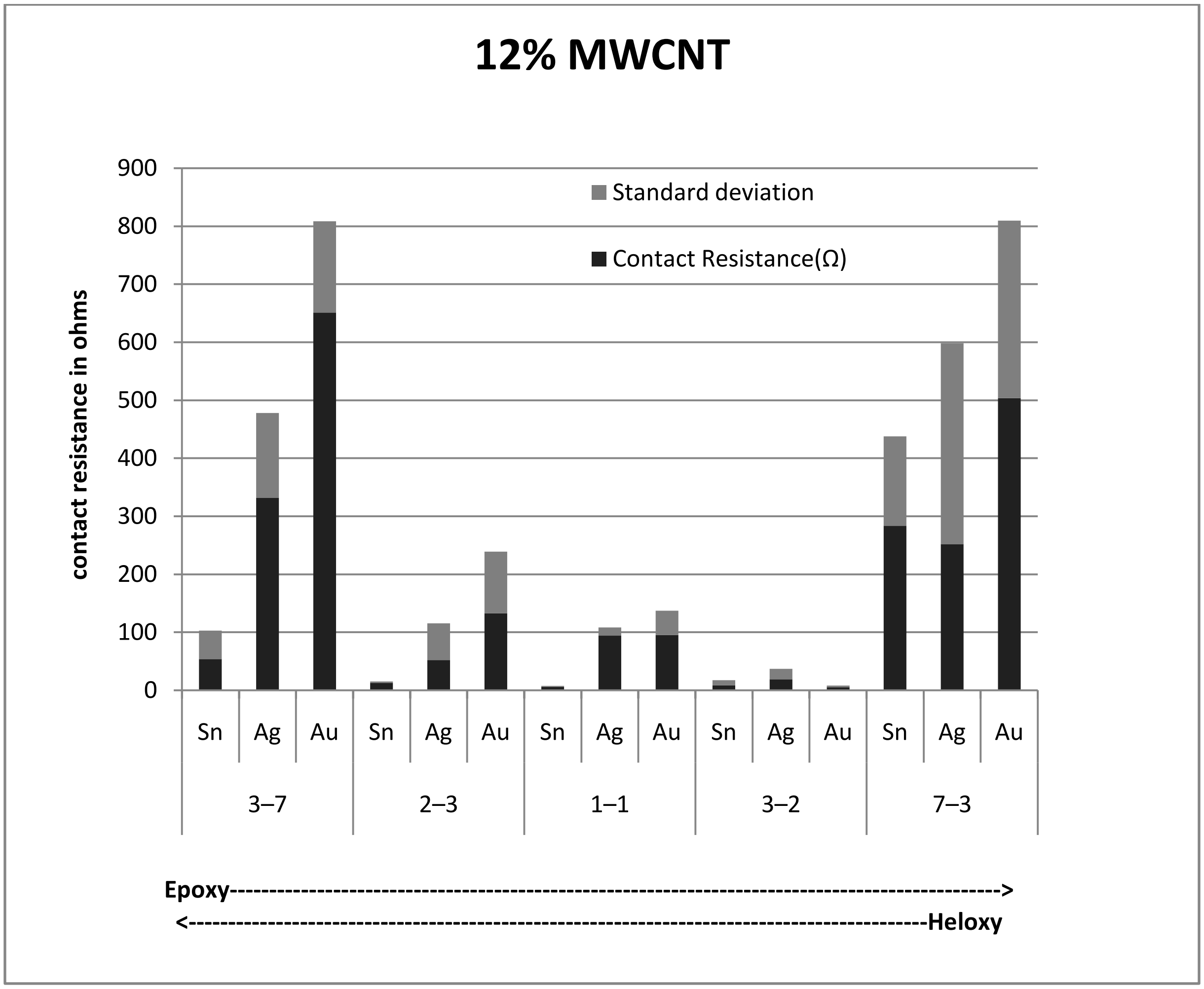


Figure 15: Average Contact resistance on different surface finished PCBs with 12wt% MWCNT loading and varying epoxy-Heloxy ratios.

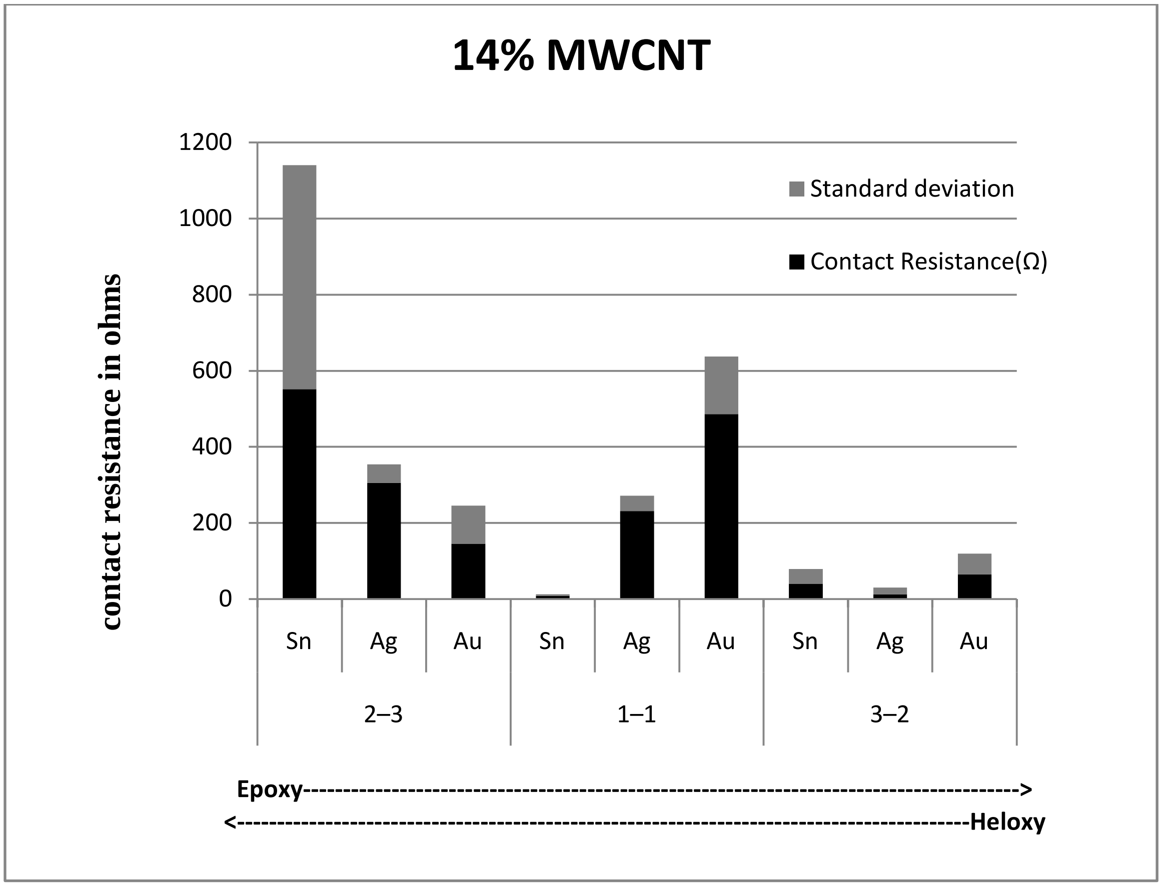


Figure 16: Average Contact resistance on different surface finished PCBs with 14wt% MWCNT loading and varying Epoxy-Heloxy ratios.

5.3 Lap shear Results and Discussions:

The lap shear strength for the samples was tested on silver, tin and gold for the loadings of 2%, 3%, 4% and 8% with the epoxy composite. The strength was measured in pound per square inch (psi). The lap shear strength was observed to be very high when compared to the Ag filled adhesive which is of the order of 200 psi. The values obtained for different metals for different loadings are presented in a graphical plot as shown in Figure 18.

From the graph we can observe that the lap shear strength for gold is higher the silver and tin finishes. The trend shows that the lap shear strength decreases with the increase in the loading of MWCNTs. The lap shear strength is way higher when compared to the Ag filled adhesive.

The next step was the addition of the heloxy, a viscosity modifier to the epoxy composite to help improve the electrical properties. Different epoxy and heloxy combinations were used like 2:3, 1:1, and 3:2. The loadings that were tested on the composite were 8%, 12%, 14%. The tests were conducted on the tin, silver and gold metals. The figures 19, figure 20 and figure 21 show the results of the strengths of all the three metals individually.

From the results obtained we observe that the lap shear strength is high at lower MWCNT loadings and decreases with the increase in the loadings. The best values obtained are at 8% loadings for gold at 2:3 composite ratio, for silver at 1:1 composite ratio and tin at 2:3 composite ratio.

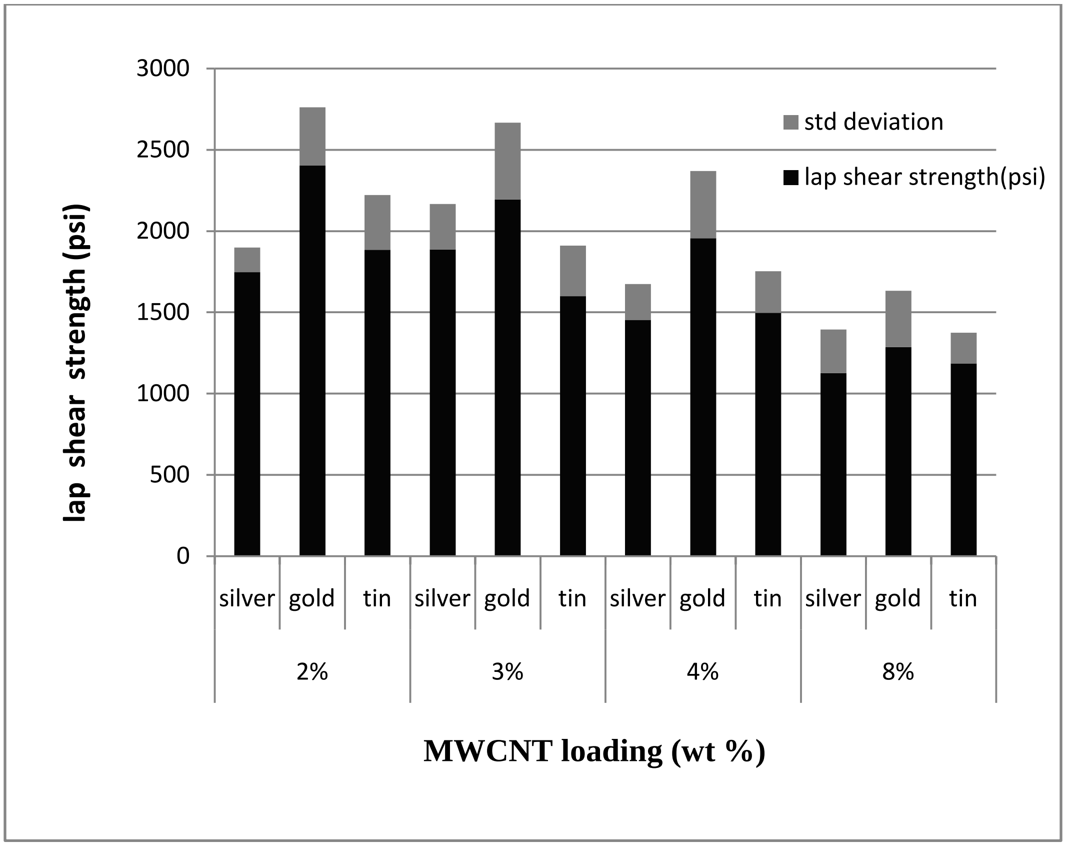


Figure 17: Lap shear Strength for different MWCNT loadings on Gold, Silver and Tin with Epoxy composite.

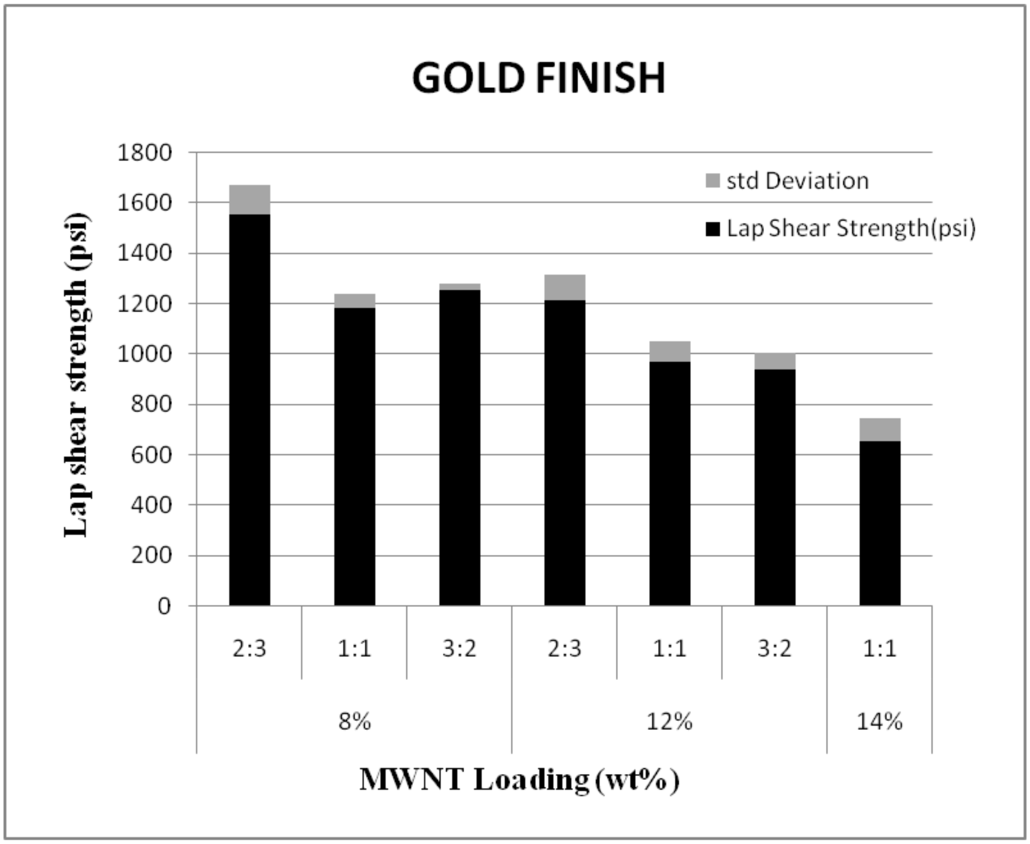


Figure 18: Lap shear Strength for different MWCNT loadings on Gold with different Epoxy: Heloxy composite ratio.

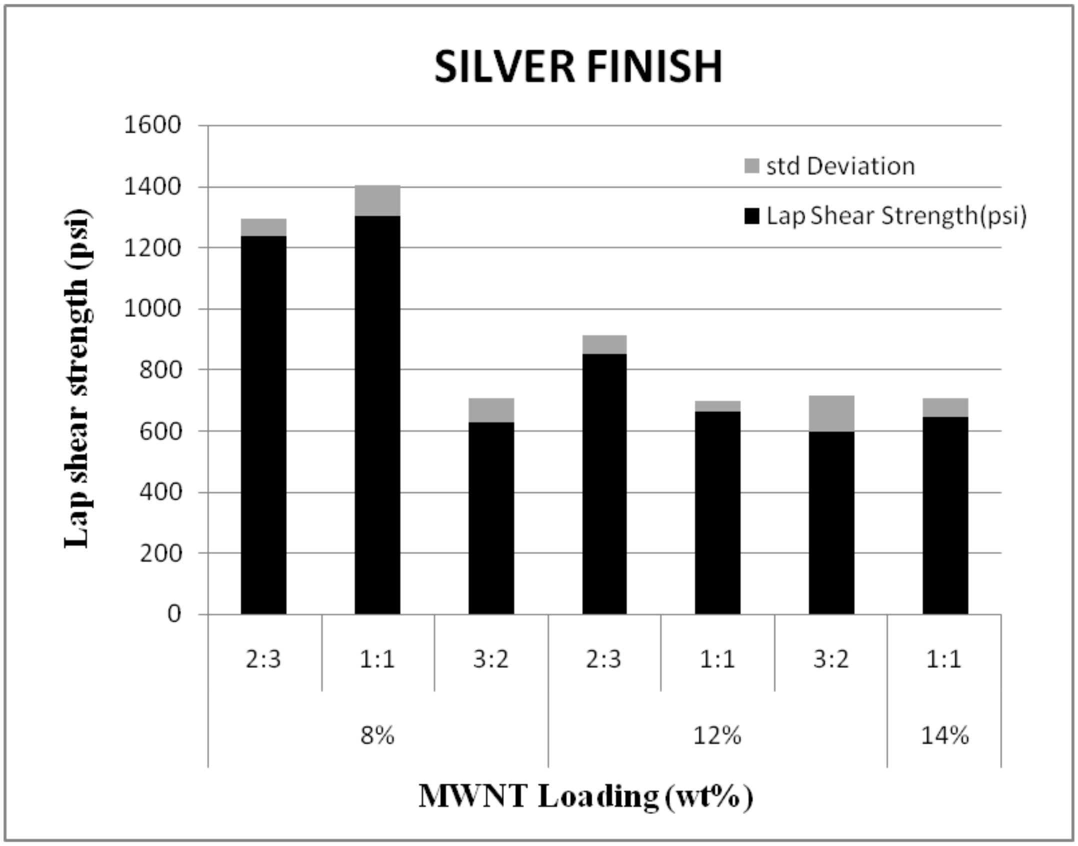


Figure 19: Lap shear Strength for different MWCNT loadings on Silver with different Epoxy: Heloxy composite ratio.

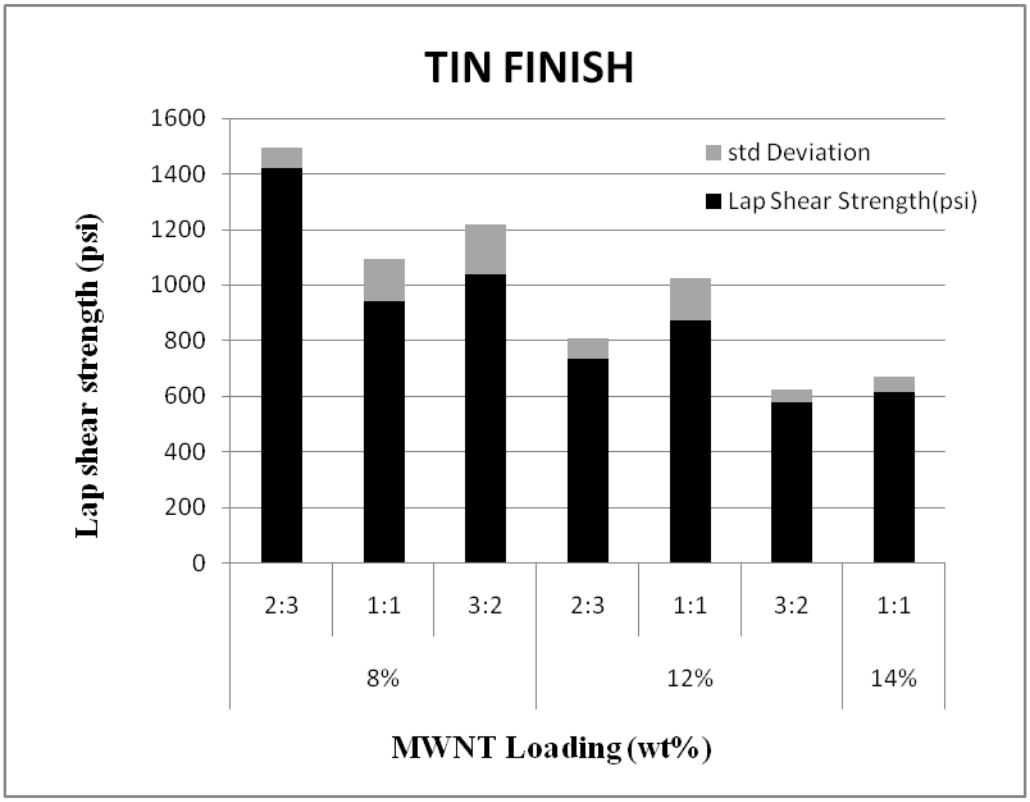


Figure 20: Lap shear Strength for different MWCNT loadings on Tin with different Epoxy: Heloxy composite ratio.

5.4 Die Shear Results and Discussion:

The experiment that was conducted on a test board which had 32 pads each of a different surface finish. The MWCNT/epoxy was dispensed on to the pads by an air pressure dispenser. A manual pick and place a place machine was used to attach the dies on to the pads. The size of the die is 0.06 inch ×0.05 inch = 0.003 inch^2. According to the Mil-Std-883 Method 2019, the minimum strength for this die is 2.5Kg.

The Figure 22 shows the shear strength values obtained for different surface finishes at different MWCNT loadings. It was observed that the values obtained are higher than the minimum requirement. Compared to all the three surface finishes silver has the high die shear strength for all the loadings. The high standard deviation shows there is a large variance in the die shear strength. It may be due to the variance in the amount of composite being dispensed and the adhesion of the die on to the pad.

5.5 Aging Experiments Results and Discussion:

The aging experiments were done all the 12% MWCNT/ Epoxy: Heloxy composite samples. The results indicate that the volume resistivity of the samples increased and decreased for different ratios of Epoxy: Heloxy. It is show in Figure 23 below. We observe for 7:3 there is very slight variation in the values.

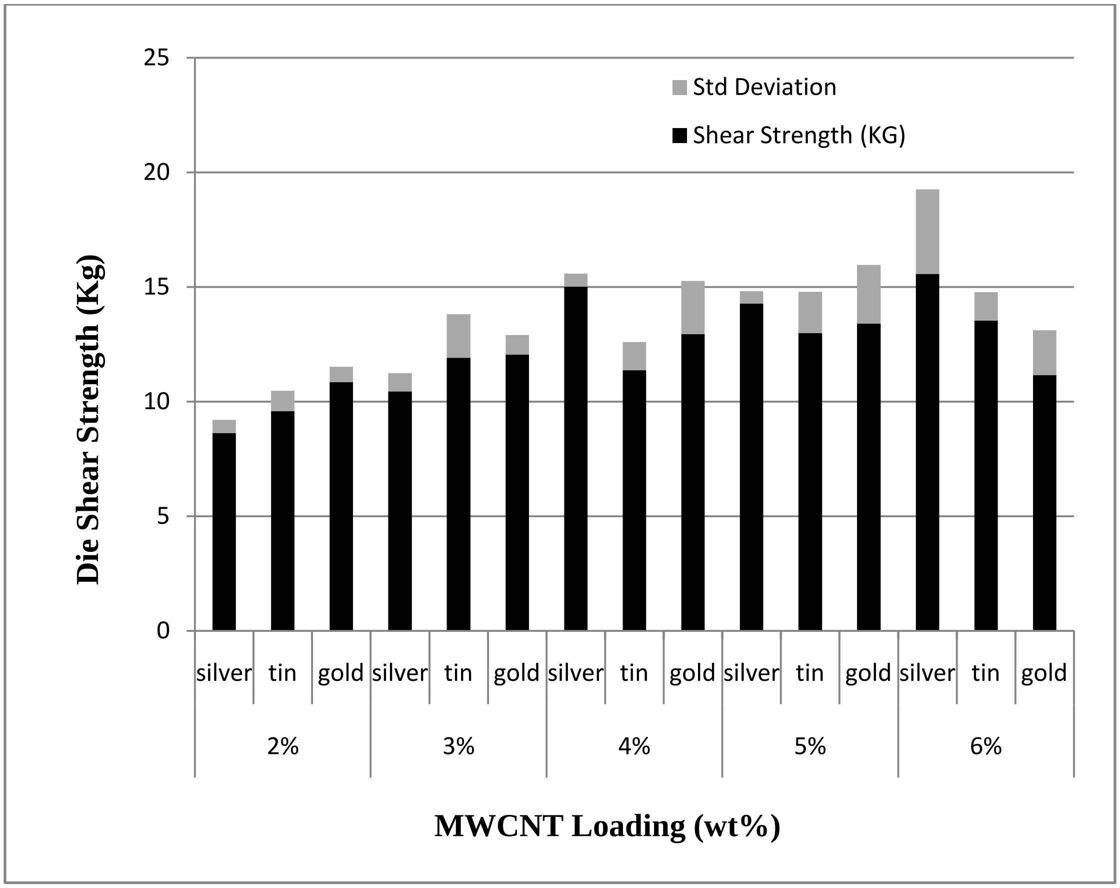


Figure 21: Die shear Strength for different MWCNT loadings on Gold, Silver, and Tin with Epoxy composite.

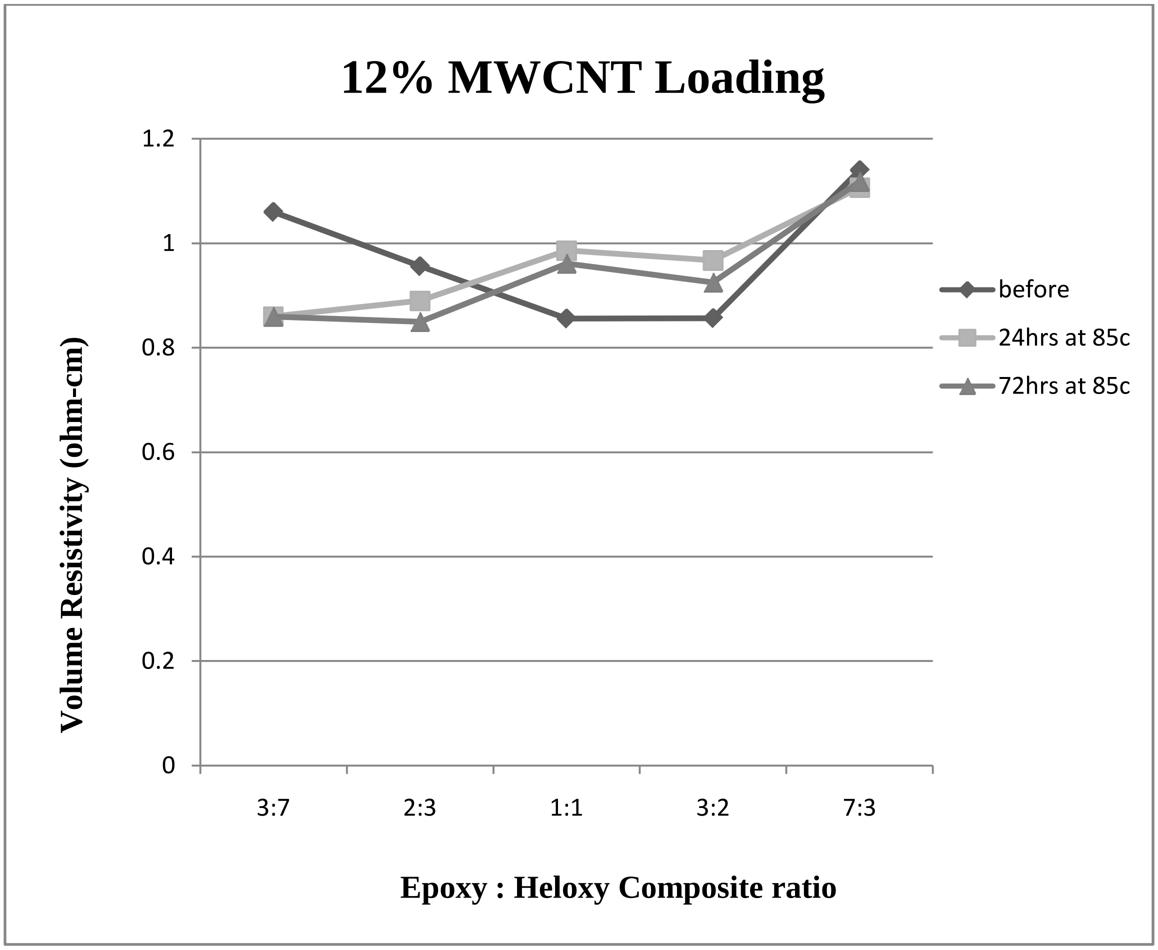


Figure 22: Variation of volume resistivity with aging experiments on 12% MWCNT/ Epoxy: heloxy composite.

Chapter6 Conclusion and Future Work

The research focuses on the potential of replacing metal fillers with MWCNTs in electrically conductive adhesives with good electrical and mechanical properties and testing the conductive adhesives on different surface finishes like gold, silver and tin. Electrical conduction and mechanical strength of the MWCNT/epoxy composite and the MWCNT/epoxy: heloxy composite were analyzed.

The results for the epoxy/MWCNT composite shows that the volume resistivity decreases with the increase in the loading. Above 12% loading the epoxy composite is not processable. The volume resistivity for the epoxy:heloxy/MWCNT composite was reduced even more when compared to the epoxy/MWCNT composite. SMWCNT/epoxy composite gave very high values, although higher loadings up to 20% were possible. Contact resistance values were very high for the epoxy/MWCNT composite. The contact resistance reduced by 10 times for the epoxy:heloxy/MWCNT composite when compared to the epoxy/MWCNT. However, the volume resistivity and contact resistance are not comparable to the commercially available metal filled ICAs.

The results for lap shear strength with epoxy/MWCNT on different metals was high when compared to the metal filled ICAs. The lap shear strength decreased with the usage of heloxy in the composite but was still higher than the metal filled ICAs. The results for the die shear strength for the epoxy/MWCNT composite were higher than the standards. The MWCNT filled adhesive has good mechanical properties than the metal filled ICAs.

The preparation of the composite involved the usage of epoxy, curing agent, heloxy and catalyst. The pot life for the composite was observed to be 72 hrs. The composite was able to be reused when refrigerated. The MWCNT filled adhesives were compatible with oven curing, pressure dispensing and stencil printing.

Based on the results from the thesis, the possible directions to extend this research can be as follows:

- The rework of the conductive adhesives which is analogous to desoldering of solder paste.
- A detailed study of chemical reactions involved between the surface finishes and epoxy mixtures used.
- Investigate the properties of the composite by mixing the MWCNTs with Ag flakes and carrying out electrical and mechanical properties.
- Even though gold does not form oxides we obtained a large value of contact resistances. This can be due to Ni coating on copper pads before gold is coated. A deeper study into the chemical reactions is needed.
- A good detailed study of the accelerated aging tests using the environmental chamber is a good topic.

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Publications:

- Keerthivarma Mantena, Jing Li, Lumpp, Janet K., “Electrically Conductive Carbon Nanotube Adhesives on Lead Free Printed Circuit Board surface Finishes”, *Proc. IEEE Aerospace Conference*, Big Sky, MT, March 2008.

- Keerthivarma Mantena, Dr. Janet K. Lumpp, Naveen Velicheti, Midhun Jasti, Poojitha Sirigiri, “Electrical and Mechanical Properties of Carbon Nanotube Filled Adhesives on Lead Free PCB Surface Finishes”, Proc. *International Microelectronics and Packaging Symposium,*Providence, RI, October 2008.