

CHARACTERIZATION OF THERMO-MECHANICAL PROPERTIES OF COPPER
NANO-PARTICLES INFUSED ACRYLONITRILE BUTADIENE STYRENE (ABS)-
3D PRINTED SPECIMENS

By

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ABSTRACT

CHARACTERIZATION OF THERMO-MECHANICAL PROPERTIES OF COPPER NANO-PARTICLE INFUSED ACRYLONITRILE BUTADIENE STYRENE (ABS)-3D PRINTED SPECIMENS

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Now a days the use of metal nanoparticle infused polymer is becoming widespread, since the development of polymer with enhanced thermal properties are being used for the growth of electronic packaging industry. In this study, copper nanoparticles are mixed with the Acrylonitrile Butadiene Styrene (ABS) thermoplastic in varying mass proportions, to observe the changes in thermal and structural properties of ABS. As expected, the extruded wire with high nano-particle concentration exhibited much higher thermal conductivity than its raw ABS form. For the case of 2% copper nanoparticle infused ABS using one dimensional steady state heat flux method though extrude wire samples and found out approximate increase in 30% of the thermal conductivity, whereas the conductivity of the 3D printed part decreases when compared to thermal conductivity of ABS printed part. Coming to the mechanical aspect of this study it has been observed that the tensile strength of such 3D printed composite ABS samples is enhanced with very small increment of nano-particle proportions, but beyond a specific optimum nano-particle mass proportion the tensile strength starts deteriorating. From this study, an optimum mass proportion of copper nanoparticles in ABS can be reported so as to achieve an efficient combination of thermal and structural properties as per the requirements. Large scale 3D printing and manufacturing of metal infused polymer is promising and can be used to produce components with enhanced properties rapidly at such a scale.

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Chapter 1 INTRODUCTION

Major developments are taking place in additive manufacturing which has led to rapid and scalable manufacturing especially if complex parts were manufactured at a rapid phase. The purpose for this research is to enhance the thermal conductivity of Acrylonitrile Butadiene Styrene (ABS), while keeping the mechanical properties intact. Rapid manufacturing of various components with varying composition can be achieved by this method. Properties can be personalized as per our requirements depending on the application, percentage of copper nanoparticles can be tailored.

Aim is to develop copper nanoparticles infused ABS and evaluate and compare its thermal and mechanical properties with pure ABS. This study is focused on the following points:

- Basis of selection of material and processing it
- Extrusion of the material to wires
- 3D printing of the wire to components which aid in evaluating thermal and mechanical properties

Hence, in this work, we are investigating the mechanical properties of the 3D printed parts by using tensile test according ASTM D638. Further the thermal properties of the material were studied the extruded wire but also the 3D printed parts.

This polymer would have wide applications ranging from polymeric heat exchangers to plastic toys replacing pure ABS.

Chapter 2 LITERATURE SURVEY

With the increase in use of thermal conducting polymer-based material which has various potential application in various sectors of industry [1]. With this increase in the potential use in various aspects such as polymeric heat exchangers there is a wide area of research that needs to be explored [2,3].

Materials that are thermoplastic like ABS and Nylon have various areas of application especially in 3D printing thermoplastic of prototypes [4]. The material nowadays used are composites. These materials are lightweight, have better processing ability, and are shock resistant. Copper particles have high thermal and electrical conductivity are used for making various polymeric composites [5]. Polymers are mostly used as insulators so a lot of research is put into to improve their thermal properties. High thermal conductivity with polymers as this would allow the heat to dissipate faster from the host material. Polymers exhibit a very low thermal conductivity because of their very low atomic density, and very low chemical bond strength, and they have complex crystal structure when they experience molecular vibration [2]. Thermal and electrical conductivity of the polymeric material can be increased by traditional method by addition of thermally conducting materials like carbon black, carbon fiber and other materials include ceramics and other metal particles like silver, copper nanoparticles, etc [6].

Fusion Deposition Modelling (FDM) is most commonly used manufacturing technique which works on additive principle where materials are extruded from the nozzle tip which are printed in the form of layers [7]. The development of materials that are used by FDM has been to increase because of their application in various domains [8,9,10,11]. Plastics can be reused as recycling plastic to explore as it is environmentally friendly, and increases the sustainability [12,13,14]. The implementation of additive manufacturing in rapid manufacturing of prototypes of various materials , structures which are tailored for a specific design, to strengthen these composite materials are increased to reinforcing them with inclusion of other materials[15,16,17], and the interest in mechanical meta-materials [18,19,20] is on a steady rise. The importance of using FDM approach is to check with the compatibility of the materials being used in the same equipment that is readily available. The main purpose is to keep the production cost low without modifying the existing hardware. The thermal, mechanical and electrical properties of these polymers have various industrial application.

Chapter 3 MATERIALS USED

3.1 ACRYLONITRILE BUTADIENE STYRENE (ABS)

ABS is one of the most used low cost engineering polymer which exhibits high impact resistance and toughness. Pure ABS can be tailored to serve various purposes. Impact resistance in ABS can be varied by changing composition of polybutadiene in the manufacturing process. High impact resistance and strength can be achieved by molding ABS at lower temperature. Molding ABS at high temperature improves the gloss and heat resistance of the product [21].

ABS polymer is highly resistant to various aqueous acids, alkalis, concentrated hydrochloric and phosphoric acids, wide range of alcohol, animal secretions, vegetable and mineral oil, they are highly soluble. They are soluble in esters, ketones and ethylene dichloride [22]. The thermal conductivity of pure ABS is considered to be 0.1W/M K [23][24]. Major advantage of pure ABS is it can exhibit constant electrical properties over a wide range of frequencies [25]. ABS can be recycled in specialized plants [26][27].

The advantage of ABS over other manufacturing materials is it can be machined easily. It can bent by using standard heat strips. ABS can be chemically affixed to itself or any other similar plastics [28]. It is also commonly used in 3D printing. The following figure 3-1 shows the pellets of ABS used,



Figure 3-1 ABS pellets [29]

3.2 COPPER NANOPARTICLES

Copper Nanoparticles are available in various sizes. The average size that is used in this study is 100 nm as shown in figure 3-2. It is 99.9% pure copper. They can be manufactured either by natural or chemical synthesis [30]. These particles are of great interest because of their historic application as coolants. Copper nanoparticles have unique characteristics such as include catalytic, antifungal and antibacterial activities which are not observed in commercial copper. Very strong catalytic activities can be achieved as a result of their large catalytic surface area [31]. Because of their very small size and high porosity they achieve higher reaction yield when utilized in organic and organometallic synthesis [30].

Copper nanoparticles have high surface to volume ratio [32] which serves as antifungal and antibacterial agent. These activities are induced because of the close interaction of microbial membranes and metal ions which are released into the solutions [33]. Due to their high catalytic activities they are applied in biosensors and electrochemical sensors. Copper nanoparticles can undergo redox reaction which is reversible. These nanoparticles have low over potentials, so they are used in sensors.[34].



Figure 3-2 Copper Nanoparticles [35]

3.3 PROCUREMENT OF MATERIALS

3.31 COPPER NANOPARTICLES:

Copper nanoparticles are procured from US RESEARCH NANOMATERIAL, INC. which is a technical enterprise focused on development of nanotechnology [36]. The copper nanoparticle have to be shipped carefully as they are highly poisonous if exposed without proper protective gear bit.

Copper nanoparticles are available from 20 nm to 215 nm. Cost of these particles ranges from \$25/gm to \$ 2.5/gm on bulk purchase. Figure 3-3 shows the SEM image of copper nanoparticles.



Figure 3-3 Copper Nanoparticles under SEM from US-nano research [36]

3.32 ABS:

ABS is available in various shapes and sizes. ABS that is required for this research purpose is in the form of small pellets as shown in figure 3-4. They are available in various colors. The color chosen for this research is *White* which is available in open source 3-D printing LLC [37]. Large quantity of 12lb is purchased at open source 3-D for \$97.28. The pellet dimension are as follows.

- Pellet length ranges from 3-3.5 mm and the diameter is consistent at 2.5 mm.
- The pellet size is specifically chosen to maintain the uniform heating when heated in the oven as they are easily exposed from all sides because of their smaller size.
- Smaller pellets are also convenient for mixing process because of their size, they can be easily and uniformly coated with copper nanoparticles.



Figure 3-4 Purchases of ABS Pellets [38]

3.4 GUIDELINES AND SAFE HANDLING OF NANOPARTICLES

As Nanoparticles are extremely small they have to be handled with extra precaution. Nanoparticles and nanoproducts have huge impact on human health and environment [39]. Nanoparticles when inhaled could get into lungs rather than any other organ in the body as they have easy access through respiratory track. The particles then enter the blood stream, as they are carried to major organs by blood stream and mostly get deposited in brain [40][41][42][43]. These nanoparticles pose a higher risk than consumption of larger particles of the same material and of similar mass. University of Texas at Arlington has specific guidelines and trainings for handling nanoparticles.

All the containers that are used for handling nanoparticles have to be maintained separately and should be tagged. While handling these nanoparticles the handlers have to wear lab coat, gloves, glasses and disposable nano mask with specialized filter which prevent nanoparticles from getting in the handler's respiratory system. Once the process of handling the nanoparticles is done there are specific trash bins for the disposal of nano waste.

Chapter 4 EXTRUSION PROCESS

4.1 EXTRUDER KIND

ABS and copper nanoparticle mixture is feed through a extruder which has heaters and thermocouples placed along the length of the screw type extruder. The heater helps in melting off polymer and is fead through shaping dye. The motor helps in rotating the screw which constantly moves the molten polymer through the shaping dye. The equipment has a coolant fan attached outside the extruder dye which rapidly cools the extruded wire, thus solidifying it. The extruded wire is then spooled for further use. The thermocouple that are placed along the length of the extruder helps in checking the temperature which in turn used to maintain the heater temperature. This extruder temperature can be varied from 120°C to 250°C. The temperature of extruder is dependent based on the material that is to be extruded. Filastruder is used to extrude wire. The chassis of the Filastruder filament of the extruder is made of CNC'ed 6061 alloy to improve assembly and also durability. The motor used is GF-45 which is roughly 200 times more powerful than V1.0/1.1 motor. Different kind of motor used than conventional one because of its better quality internal parts and easily replicable brushes. This Filastruder has advanced motor control ability which helps in controlling voltage (speed) and current (torque), i.e., if the nozzle gets clogged, the current is increased so the screw rotates at a higher torque in addition to increasing the temperature. There is an LCD display to show the variation in the voltage and current.

Polymers that can be extruded from this machine include ABS, PLA, HDPE, LDPE, TPE, nylon, polycarbonate, acrylic and polypropylene [48]. This machine can also be used for recycling of printed plastic. The front and side of Filastruder is shown in figure 4-1 and 4-2.

Specifications of Filastruders are as follows:

- Typical extrusion rate is 1 kg in 5 to 8 hrs. and extrusion of wire is 10 to 36 in./min depending on type of material
- Extrusion temperature can go as high as 250°C, this is limited for safety purposes to prevent thermal decomposition of polymer.
- Size: 18 x 6 x 4 in.
- Noise: 52 dBA at 3 ft

- Power: 110 to 240 VAC, 50 to 60 Hz (Cost estimated to 10cents / kg extrusion)

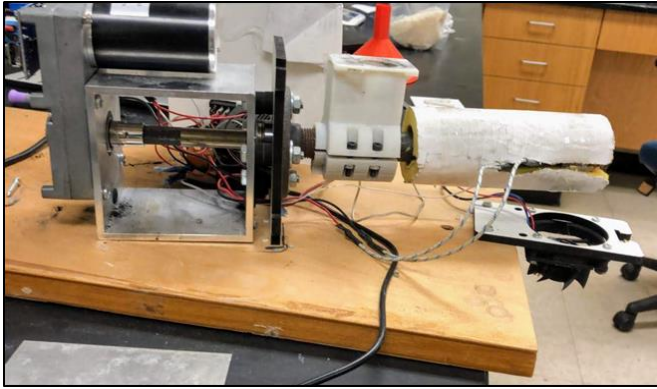


Figure 4-1 Extruder Side view



Figure 4-2 Extruder front view

4.2 TEMPERATURE FOR EXTRUSION

The optimum temperature for ABS extrusion is from 180°C to 190°C. This temperature is selected upon extrusion rate which depends on the diameter to be achieved from extrusion. Higher the temperature, the viscosity of the polymer decreases leading to thinner diameter of the extruded wire and vice-versa. The ideal diameter of the wire is to be maintained at 1.5 mm with accuracy of +/- 0.05 mm.

For the extrusion of copper nanoparticles induced in ABS the temperature is maintained at 190°C as the speed of the fan cannot be varied. This value was experimentally determined.

4.3 PRE-HEATING OF EXTRUDER

Initially when the machine is switched on, the motor is off. The temperature regulator is shown in Figure 4-3. The temperature is gradually increased to 190°C and is maintained for 30 min and then the motor is switched on for extrusion of polymer. The time interval of 30 mins for uniform heating of screw extruder was experimentally determined. Once the motor is switched on, the fan which is present exactly below the nozzle is automatically switched on. Depending on the temperature of the room the nozzle sometimes falls below 190°C which is shown by thermocouple. To maintain the temperature, the extruder temperature is manually set for higher value. The temperature of the nozzle is directly related to the diameter of the wire to be extruder. Because of such high temperature the extruder is wrapped with

thermally insulating material to prevent heat loss. The nozzle and the freshly extruded wire should be handled with high precaution due to high temperature.



Figure 4-3 Temperature Regulator

4.4 WIRE EXTRUSION SIZE AND COOLING

As previously discussed, the wire extrusion takes place at 190 °C. The diameter of the wire is maintained by manipulating the temperature of the extruder as the wire comes out of the nozzle directly passes over a cooling fan. This fan sets the wire immediately as a result of rapid cooling. The wire loses its elasticity when subjected to rapid cooling. If the temperature is maintained below the required temperature it becomes hard for the extruder to push the material through the nozzle leading to varying diameter of wire as the chances of the screw stopping is high. Thus, due to the weight of already extruded wire, the diameter of filament that is extruded later on decreases at a much higher rate. If the temperature is maintained high viscosity of the material decreases thereby the diameter of the extruded wire is very thin. The nozzle diameter can be changed by changing the nozzle. Readymade nozzles are available for 1.25 mm, 1.5mm and 1.75 mm diameter.



Figure 4-4 Extruded Wires

4.5 CLEANING THE EXTRUDER

After each batch of extrusion of polymeric material, the extruder must be cleaned to make sure that no nanoparticles are trapped inside the equipment. To maintain purity of each batch 150 gm of pure ABS is passed through the extruder. The wire usually comes out in darker color as it collects all the trapped nanoparticles from the similar extruder. Towards the end of the cleaning cycle the extruded wire has the color of pure ABS. Whenever a new batch is extruded the initial 3 mts of wire is discarded to maintain purity of the batch. Figure 4-5 shows the extruder extruding pure ABS at the end of cleaning cycle.

After extrusion of each batch there is presence of polymeric material in the extruder from the previous batch, to prevent this contamination, the above-mentioned cleaning process is followed. To maintain consistent properties throughout the wire, random sections of the wire is fed through the printer for printing of parts.

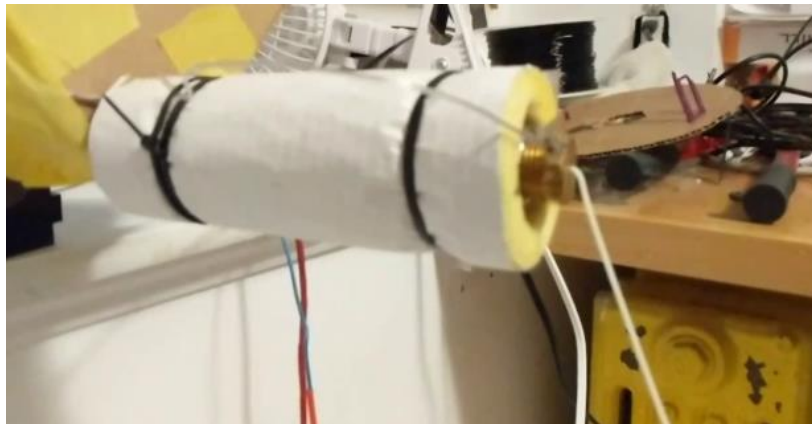


Figure 4-5 Extruder cleaning procedure at the end of the cycle

5.1 FLOW PROCESS OF MANUFACTURING PRINTED PARTS

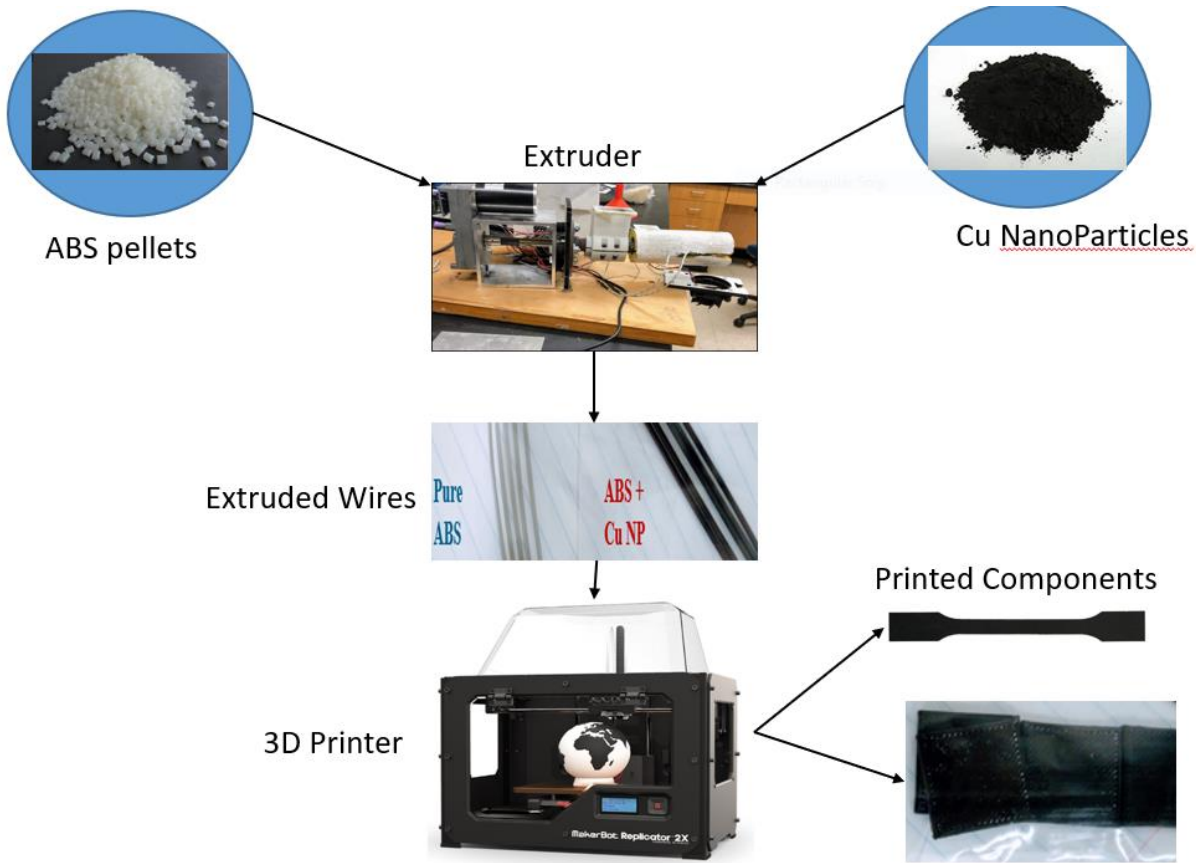


Figure 5-1 Flow chart of the procedure.

In this section, we will be looking into basic procedure of making our desired polymer as shown in flow chart 5-1. The first step is taking required amount of ABS and copper materials, mixing them together to create a polymer and then putting them through Filastruder equipment to extrude desired diameter wires. The wire that is produced from the set process is then fed into 3D printer (Makerbot Replicator 2X). The extruded wire is then used to print the necessary dog bone structure for tensile testing and necessary FOX50 samples to conduct required thermal conductivity.

5.2 PRE-HEATING OF POLYMER

ABS when delivered are in a sealed packaging to prevent them from exposure to any moisture present in the atmosphere as ABS has tendency to absorb moisture. The packaging comes with little silica gel moisture absorbent which are used to absorb any trapped moisture to prevent its effect on the properties of ABS. As a precautionary step the required amount of ABS is taken into a dry clear glass container and is baked in the oven for 30 minutes at 200° F (94° C approx.) as shown in figure 5-2. This process helps in the removal of any extra moisture that might be trapped in ABS.

ABS is then let to cool down in the oven after heating for about 20 minutes as hot polymers are hard to handle. Other reason for letting the polymer to cool down is ABS tends to stick to each other when heated to such temperature, this causes problems during dry mixing as ABS sometimes doesn't get broken down into its basic pellet form. It is also hard to blend hot polymer as the chances of copper nano particle adhering to hot polymer, on the upper side which leads to improper mixing of the composition. To prevent such problems that leads to agglomeration of copper nanoparticles, ABS can cool down for about 20 minutes which is experimentally found to bring back heated ABS to room temperature.

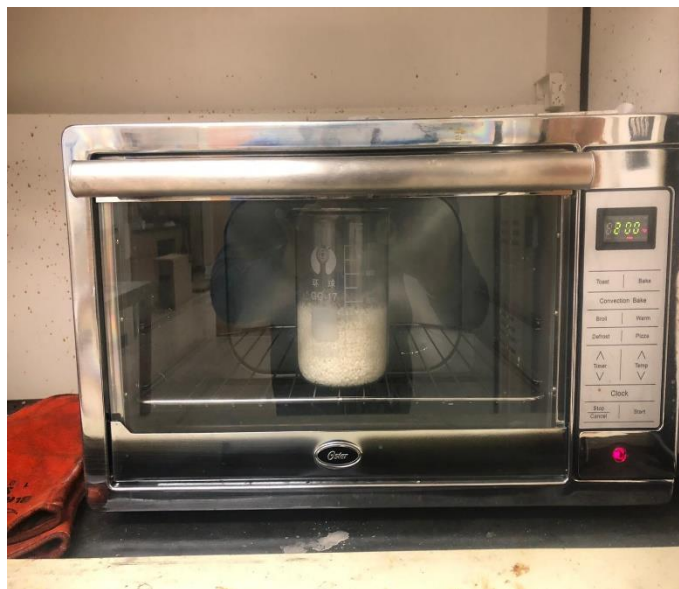


Figure 5-2 Pre-heating the ABS

5.3 WEIGHT PERCENTAGE OF SOLID MIXTURE

In chemistry the mass fraction of a substance within a mixture is the ratio w_i of the mass m_i of that substance to the total mass M_{tot} of the mixture [50] which is expressed as:

$$W_i = m_i / M_{tot}$$

This equation is used for the calculation of various composition of polymers that are to be extruded. For example, if we require a 50% copper nanoparticle material with ABS, we would take 50gms of ABS and mix it with 50gms of cu nanoparticles which would in-turn create a total of 100gms of polymer which is of composition 50% copper nanoparticle.

By using a very sensitive scale, we have created a polymeric blend for 0.5% copper nanoparticle, 1% copper nanoparticle, 1.5% copper nanoparticle and finally 2% copper nanoparticle.

5.4 METHOD OF MIXING

5.4.1 MIXING:

Mixing is a process in engineering in which two or more homogenous physical systems are combined to form a single homogenous material. Modern industrial processing mostly involves some sort of mixing [49]. Some chemical reactors are also classified under mixing processes. With specific equipment, it is possible to mix solid, liquid or gas into another solid, liquid or gas. Blending of solid powders is one of the most used technique in manufacturing of required tablets in pharmaceutical industries.

5.4.2 CLASSIFICATION OF MIXING:

Mixing of materials is classified into various forms depending upon the state of material that is to be mixed is classified into the following groups [51]:

- Liquid-Liquid mixing.
- Gas-Gas mixing
- Solid-Solid mixing
- Liquid-Solid mixing
- Liquid-Gas mixing
- Gas-Solid mixing
- Multi-phase mixing

The procedure that is used for mixing of our polymer is dry mixing.

5.4.3 DRY MIXING OF MATERIALS:

Powder blending is a ancient operation that is being taking place in solid handling industry. For many decades Solid-Solid mixing is used to homogenize two or more bulk materials. Various machines were invented and designed specifically to handle various bulk solid properties. Figure 5-3 shows the primitive mixing machine used for mixing two or more materials together. Based on these machines and engineering knowledge developed over the years used to design more reliable equipment and for a perfect blend of materials. Now a days mixing technology has various applications ranging from increasing the quality of product to coating of particles and to fuse materials together. Solid-Solid mixing can be performed in batch mixers or in continuous dry mixers which are more complex but have a superior material in terms of segregation capacity and validation [52].

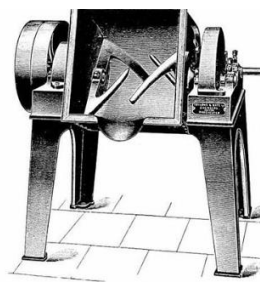


Figure 5-3 Ancient mixer [53]

5.4.4 USE OF BLENDER:

For the required polymer to be achieved, we have taken a simple blender which works on circular whirling motion to create a vortex or a spiral movement for the materials so as to coat the copper nanoparticles evenly onto the ABS [54]. The purpose of this dry mixing is not to crush the ABS pellets into finer components but to simply surface coat the copper nano particles on top of ABS pellets.

The blender is run for about 6 minutes to maintain homogenous mixing throughout the polymer. Once the blender is run for 3 minutes all the surfaces of the blender are tapped so that all the nanoparticles that adhere to the surface of the blender falls back into the material so that it would not effect the composition of the polymer. The blender is run for the next 3 minutes to complete one perfect cycle and the surface is tapped once again as shown in figure 5-4. After blending, the polymer is allowed to settle down because during the mixing process copper nanoparticles form a thick cloud, if let to escape that would create a polymer with lesser amount of copper nanoparticles than desired. This blended polymer is fed to the extruder to get a desired composition of extruded wire.



Figure 5-4 Mixer used for blending

CHAPTER 6 3D PRINTING OF PARTS OR ADDITIVE MANUFACTURING

6.1 INTRODUCTION

3D printing is a additive manufacturing process in which a model which is in a software is converted to an actual model [55]. The model is achieved by additive process. That is the model is created by successive layering of materials. These layers are thin slices of horizontal cross section of the object. 3D printing is the opposite of hollowing out of material by machining. This procedure is used to produce intricate shapes that are hard to manufacture by any other method. The CAD model was drawn in 3-Dimensions using Solidworks software. The file is stored as STL file that is later used in Makerbot software to print soecimens.

The additive manufacturing method is easy, cost-effective and produces parts at a much rapid phase. Additive manufacturing is method saves wastage of material. The application of 3D printed parts varies from toys to application various industries. 3D printing has made prototyping very easy which is termed as rapid prototyping. Figure 6-1 shows the MakerBot Replicator used for printing various components in this thesis.

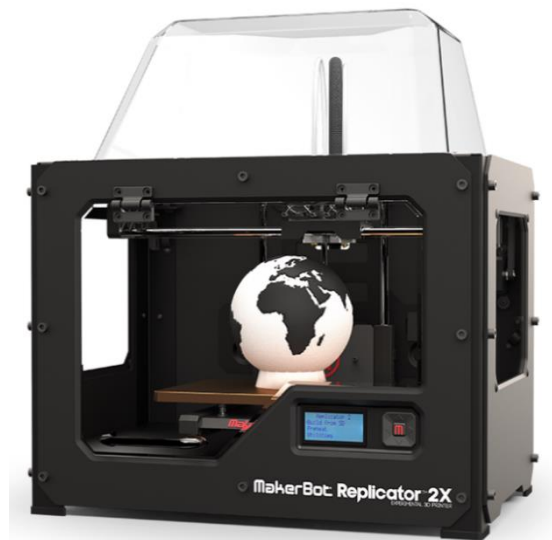


Figure 6-1 MakerBot Replicator 2X used for printing of parts [56]

6.2 ROLE OF 3D PRINTER

Before printing any parts, the model has to be designed using the CAD software that are available to us. For this thesis we have used SolidWorks. From this application we have taken the 3D model and stored it in a STL file which cuts this model into a small thin layer that helps in the printing process. The STL file is used to print components using a 3D printer.

For printing of the parts, filament which is spooled and is used for 3D printing of the parts. The spool is attached to a connector behind the 3D printer. The 3D printer controls the nozzle temperature through which the polymer is to be deposited. The bed temperature is also regulated to prevent the warpage of the printed parts. The extruder is controlled by servo motors. The extruder heats up a section of the polymeric wire which melts and then passes through the nozzle which is deposited on the bed. By this process each layer of a 3D printed part is printed. This is the main reason for a 3D printer can print various intricate shapes at a low price and with great ease. Figure 6-2 shows the schematic diagram of a working 3D printer.

Present days there are multiple ways in which additive manufacturing processes occur. This difference is due to various materials, printing conditions and intricacy of the shape. Most used procedure for printing of Polymer is Fused Deposition Modelling (FDM).

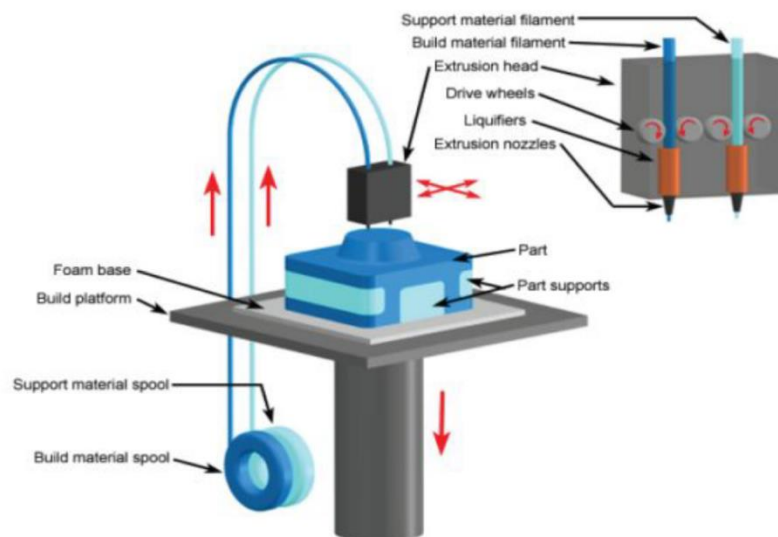


Figure 6-2 Schematic picture of the 3D Printer [56].

6.3 ADVANTAGES OF ADDITIVE MANUFACTURING:

Additive manufacturing of parts has been used in various application which has rapidly picked up over the past few years due to major developments in this field. This has directly led to technical benefits in the area of additive manufacturing which turned out to be profitable.

6.3.1 COST EFFECTIVE:

Compared to other available manufacturing processes 3D printing is one of the most effective and easiest way of manufacturing as it requires very limited number of humans for fabrication of various materials. As compared to any other method of manufacturing, 3D printing this procedure leads to eliminating of multiple steps like assembly and finishing the product. leads to saving valuable time and money. Labor plays a huge role when it comes to determine the cost of individual product due to minimizing the labor this method have effectively decrease the cost of each part.

6.3.2 RAPID PRODUCTION:

3D printing has major advantage is that complex parts can be manufactured with ease as it is multiple times faster when compared to conventional machining. Parts can be printed overnight with little to no requirement for monitoring which is not possible by conventional techniques. Thus, this process has let to manufacturing components on demand which has again saved the manufacturing companies valuable capital. This leads to cost savings in storage of the manufactured parts. Figure 6-3 shows few rapid prototyped 3D printed parts



Figure 6-3 Rapid prototyping using 3D Printing.[57]

6.3.3 MATERIAL SAVINGS:

Because this process is additive in nature, meaning the part is printed to our requirements rather than taking a raw material block and machining it to our required dimensions. Thus, leading to eliminating which require material to be multiple procedures though which materials are carved out of the component. 3D printing requires little material for manufacturing of the exact similar part, waste is reduced which leads to minimal waste during manufacturing process. 3D printing is not only cost effective but also environmentally friendly as the impact on the environment decreases when compared to any other method of manufacturing. Reusing of materials in 3D printing is simple and straight forward when compared to other manufacturing process.

6.3.4 INTRICATE SHAPES:

Manufacturing of complex shapes is always time-consuming process if conventional material reduction process is followed. Usually intricate shapes were manufactured using either by casting or by using machining techniques like CNC and cutting. Figure 6-4 shows an intricate part printed using 3D printer. These processes have lot of limitation when it comes to machining of intricate shapes where 3D printers have stepped in. Due to additive manufacturing of each individual layer it is easy to manufacture parts by 3D printing rather than machining them. The limitation due to tool sizes, shapes and access to the corner of the parts is eliminated. Need of having wide range of tools is eliminated by this method of manufacturing. 3D printing helps in endless customization when compared to other manufacturing methods. Parts can be printed with very little tolerance levels than machined parts.

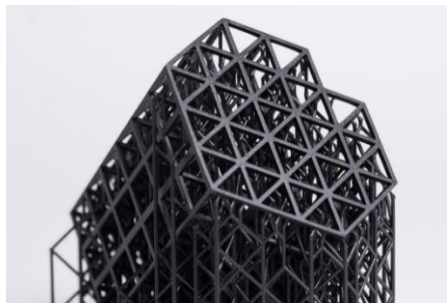


Figure 6-4 Intricate shapes printed by 3D Printer [59].

6.3.5 USE OF VARIOUS RAW MATERIALS:

Multiple materials can be used in printing of parts by varying the printing properties. If conventional machining is followed a lot of compromises have to be made by reducing the complexity of the part by eliminating certain aspects where machining is not an option. Multiple materials are incorporated to print one component as per the manufacturer specifications. This however is not possible when we implement conventional manufacturing processes which in turn would drive up the cost of the component. With increase in technology, 3D printers are used to print and manufacture various parts by multiple materials that include glass, ceramics, biomaterials, etc. as shown in figure 6-5.



Figure 6-5 Various materials used in 3D Printer.[60]

6.3.6 RISK MANAGEMENT:

Manufacturing of components by conventional process would be very expensive if the mold or dye that is designed is faulty or changes must be made to the component. Small changes in fabrication would lead to large financial impacts. Its increasing used for prototyping to check on the feasibility and the part is manufactured ready to order which eliminates the process of investing in equipment like tools and special jigs. This in turn reduces the risk factor and brings down the prototyping process.

6.4 PRINTING PARAMETERS:

3D printing depends on various factors which include shape, complexity of the part, the material used in printing and the printing parameters of the parts. The four main aspects which involve the manufacturing of 3D printed parts are as follows:

- Extrusion Parameters
- Design Parameters
- Material Properties and
- Slicing Parameters

Each of these major parameters have effect on the 3D printed parts, the extrusion parameters are as follows: the kind of printer used, the temperature at which extrusion occurs, speed at which extruder is moving and nozzle extrusion speed.

The design parameters are as follows: the design of the component, the method in which the loading occurs, stress concentration's that occur in the part because of the sharp change in geometry, the kind of surface finish that is desired and geometry of the part itself.

The material properties that effect the component are as follows: the strength of the material, the elastic modulus of the printed part, the shear modulus, Poisson's ratio and condition of the material at which it was cured.

The slicing parameters that effect the shape and size of the printed part as follows: the orientation of plain on which the part is printed, the orientation of t6he raster, the height of individual layer in the part, the infill pattern of the part, the pattern of infill and the number of shells in the part, the number of air gaps present ion the part.

The following are the main parameters that effect the 3D printing of a part:

6.4.1 INFILL PERCENTAGE:

Infill in 3D printing refers to the material that is to be filled in a structure that is to be printed. Infill percentage basically defines the amount of material that is to be deposited in printed part. The infill also determines how hollow the structure is when its printed. Infill pattern determines the strength of the parts. Higher the infill percentage higher the strength of the part. Sometimes this infill percentage is used just for decorative purposes. Figure 6-6 shows various infill percentages printed parts. Higher the infill percentage the heavier, component and has stronger print. Higher the infill percentage more time it takes for the printer to print the components. The infill percentage that was used for printing of our parts is 100% infill [61].

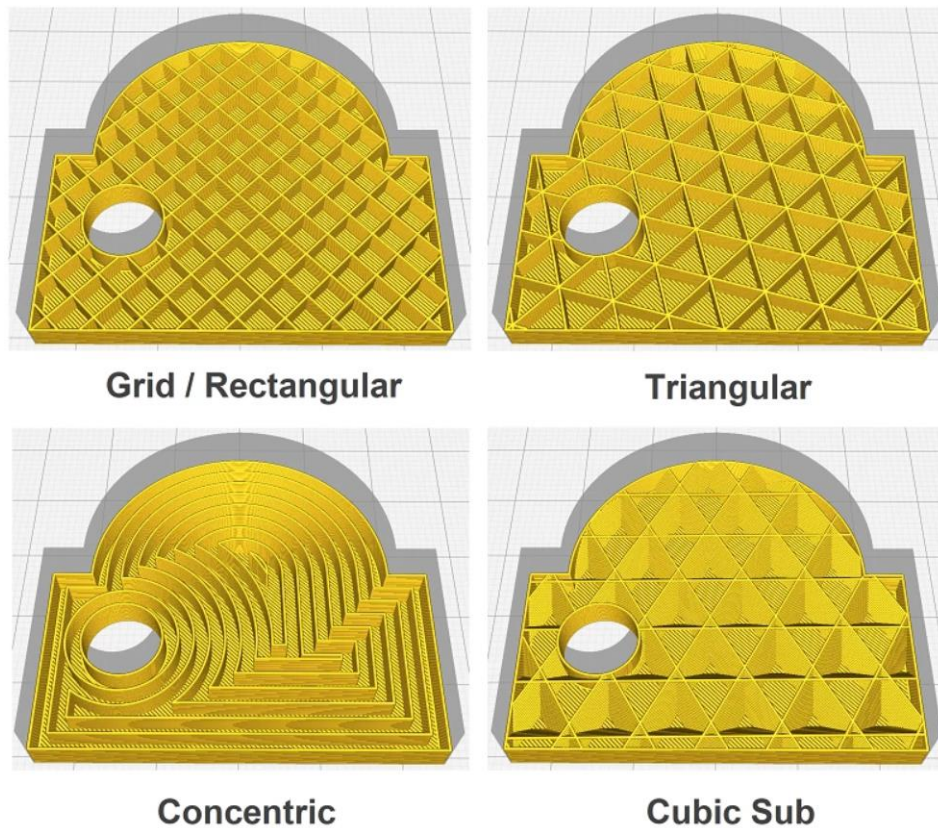


Figure 6-6 Infill percentages in 3D Printer [62].

6.4.2 INFILL PATTERN:

There are several typical patterns which can be used for printing of the components, the most standard patterns that are readily available. The common patterns include standard or basic geometry as shown in figure 6-7. The infill shapes depend on the type of strength required. The basic patterns are rectangular which is easy to print and provides rigidity in all direction. Triangular pattern is preferred if strength required is unidirectional. Wave or wiggles are preferred when they are used for components that are either flexible or twistable. Honeycomb infill is used to provide greater strength in all direction. Honeycomb takes a longer time to print when compared to rectangular pattern [63].

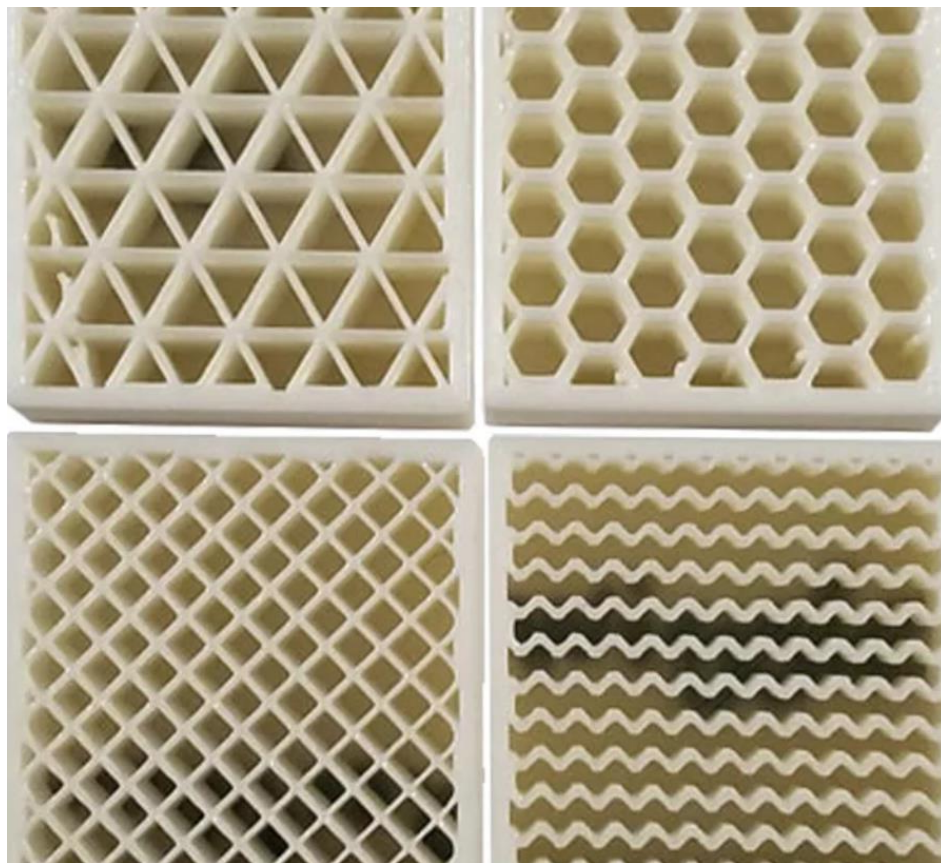


Figure 6-7 Infill pattern in 3D printer [64].

6.4.3 LAYER HEIGHT:

Layer height is self-explanatory i.e. the height of each printed layer in a 3D printed structure. Figure 6-8 shows various layer heights that can be achieved using 3D printer. Lesser the size of each layer smoother the component. If properly manipulated this would lead to increased resolution of the printed part and would increase the smoothness of the printed part.

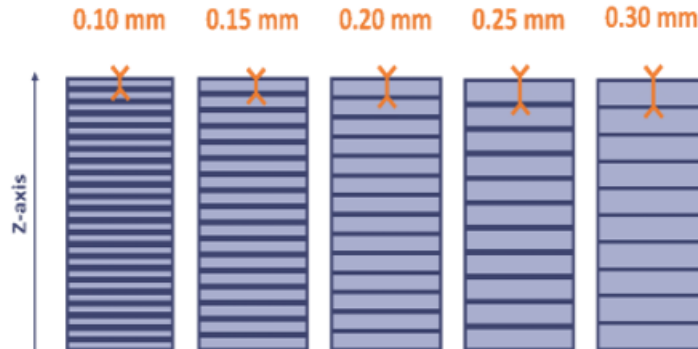


Figure 6-8 Layer height in 3D printer [65].

6.4.4 PLAIN OF ORIENTATION:

3D parts can be printed in all three orientation. XYZ plain where the part is printed in XY direction with increments in positive Z direction. Likewise, in XZY plain and ZXY plane. Figure 6-9 shows three plains of orientation used for printing 3D parts. Selection in which plain it is to be printed make the manufacturing of the part easy and gives optimal strength. The parts are printed for this research is in XY direction with increments in positive Z direction for maximum strength and also stability of the component.

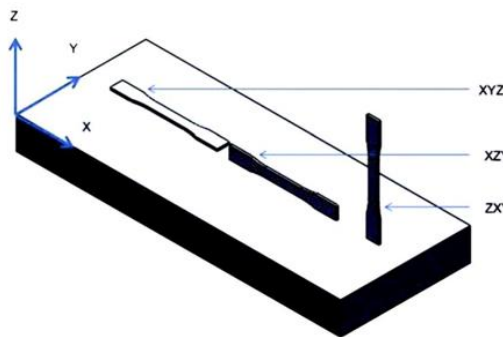


Figure 6-9 Plain of orientation in 3D printed part [66].

6.4.5 RASTER ORIENTATION:

The main advantage of 3D printing is the direction of manufacturing can be controlled. Figure 6-10 shows few commonly used raster orientations in 3D printed parts. The tensile tested parts printed for this study have an outer shell then followed by 0° and the next layer is 90° and the following layer is at 45° . This process of layering is repeated until the part is completely printed. Raster orientation helps in increasing the strength other than unidirectional strength along the direction of printed filament.

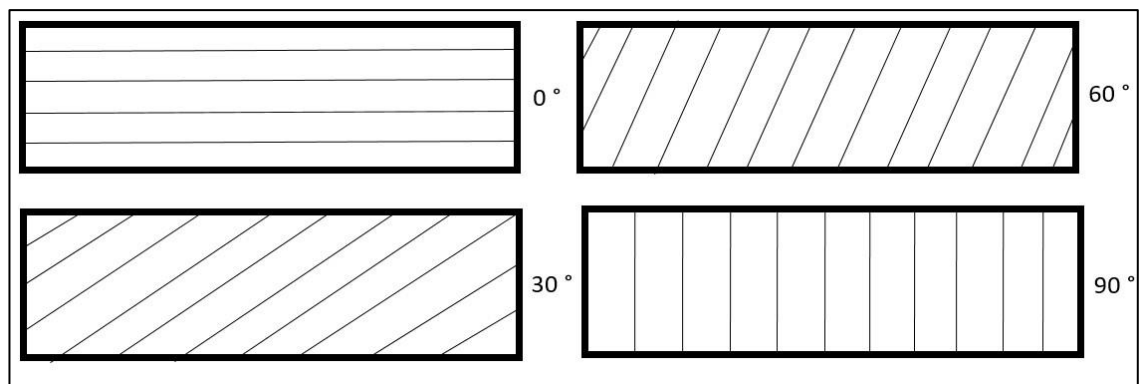


Figure 6-10 Raster orientation in 3D printed parts [67].

CHAPTER 7 Tensile Testing

7.1 INTRODUCTION

Tensile testing is a destructive testing technique that is used to find the ultimate tensile strength of the material [68]. The stress vs strain curve is plotted to understand various stages of the material to be tested. The stress vs strain curve helps us in selecting of a material for a specific application. Shows us how that specific material would perform under varying forces. To demonstrate the proof of concept of a new product. Which helps us in identifying the type of materials like tensile or brittle. Tensile testing is used to determine the quality of the material. Used for providing scientific data and engineering function of that specific material.

7.2 TYPE OF UNIVERSAL TESTER USED

Our lab has Shimadzu's AGS-X series universal test frame which is a modern testing system which has a wide capacity range of load cell as shown in figure 7-1. This tester is compact and a countertop tester. The table top test frame which has a capacity of 10 KN.

This universal tester also has height extended height options available. The load cell ranges from 1 N to 300 KN. It also has a chamber for testing in various temperatures. The main features of this test is integrated operation panel which allows us to change from one test to another. This panel allows us to change and store various test like tensile, compression, bending, cyclical loading and fracture test. Other main advantage of this system is its ability for quick test setup and exchange. The use of one fixed and other moving joint allows us to make fixture joint exchange easy. Shimadzu has a high comprehensive safety Measures. The safety measures that are include emergency-stop button, it has limit switch and also anti-Collision system which helps us to prevent accidents.

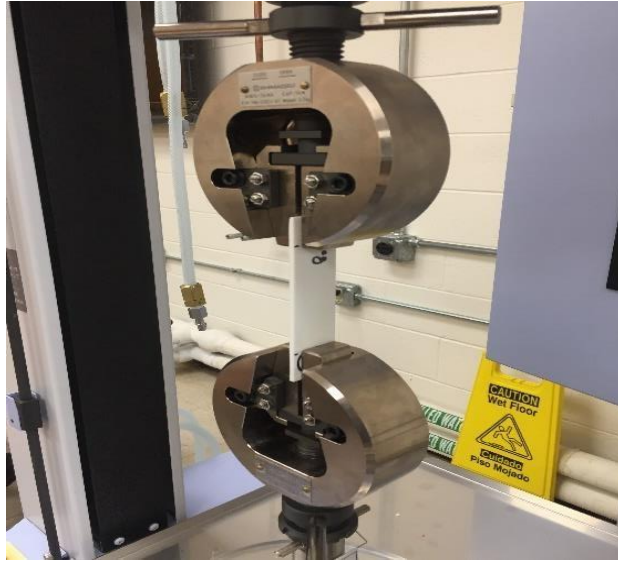


Figure 7-1 Tensile Tester [69]

7.3 SAMPLE DIMENSION

The samples used for testing are printed to ASTM standard standards for consistency of the results. The test sample that is printed as per ASTM polymer sample size as per required specification. The dog bone sample were prepared and were tested on the tensile testing machine. Each batch has 3 samples and they were tested are from the same batch and same composition.

The dog bone sample selected is of ASTM D6388 standard Type 1 as shown in figure 7-2. The dimensions of the sample are as follows [70]:

- The length of the sample is 165 mm.
- The gauge length is 50 mm.
- The width of the dog bone is 19 mm.
- The width of the gauge is 13 mm.
- Thickness of the specimen is less than 3 mm.

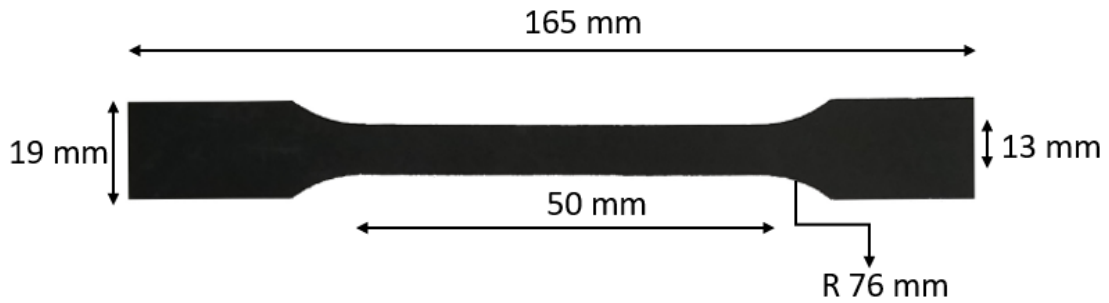


Figure 7-2 Tensile Test Specimen [71].

7.4 RESULTS AND DISCUSSION

The values we get when we conduct tensile test is force and displacement of that sample which when divided the dimension of the sample, we get values of stress and strain. The values of stress vs strain were plotted into a graph. To get the value of stress we divide the force by area which is a product of thickness and width of the gauge. To get the strain of the specimen we divide displacement with the length of the gauge. Figure 7-3 shows ideal Stress vs Strain curve.

$$\text{Stress} = P/A$$

P is the force (N), A is the cross-section area (mm^2) [72].

$$\text{Strain} = D/L$$

D stands for displacement (mm), L is length (mm).

The graphs that are put up shows the tensile test results of various percentages of copper nanoparticles. The stress strain graph for pure ABS, 0.5% Cu nanoparticles all the way up to 2% Cu nanoparticles with an increment of 0.5% Cu nanoparticles. The ultimate tensile stress of each percentages is provided in the table following the chart. The end tensile stress of all the percentages of Cu nanoparticles are plotted.

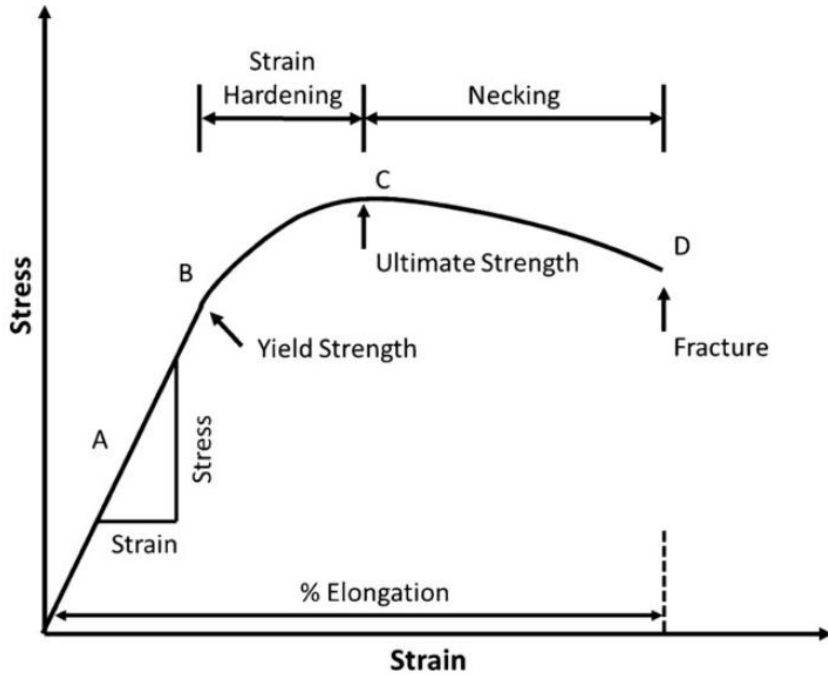


Figure 7-3 Ideal Stress vs Strain Curve [73].

The following graphs and corresponding tables show the various stress strain curves of three samples with varying copper Nano particles along with their peak Ultimate Tensile Stress:

7.4.1 TENSILE TEST OF PURE ABS:

Table 7.1-Pure ABS Ultimate Tensile Stress

Pure ABS	Ultimate Tensile Stress (MPa)
Sample 1	23.1864
Sample 2	21.8651
Sample 3	23.6495

Pure ABS has the second highest Ultimate Tensile Stress with an average of 22.901 MPa. The following graph 7-4 shows the stress strain curves [74] for the three various samples.

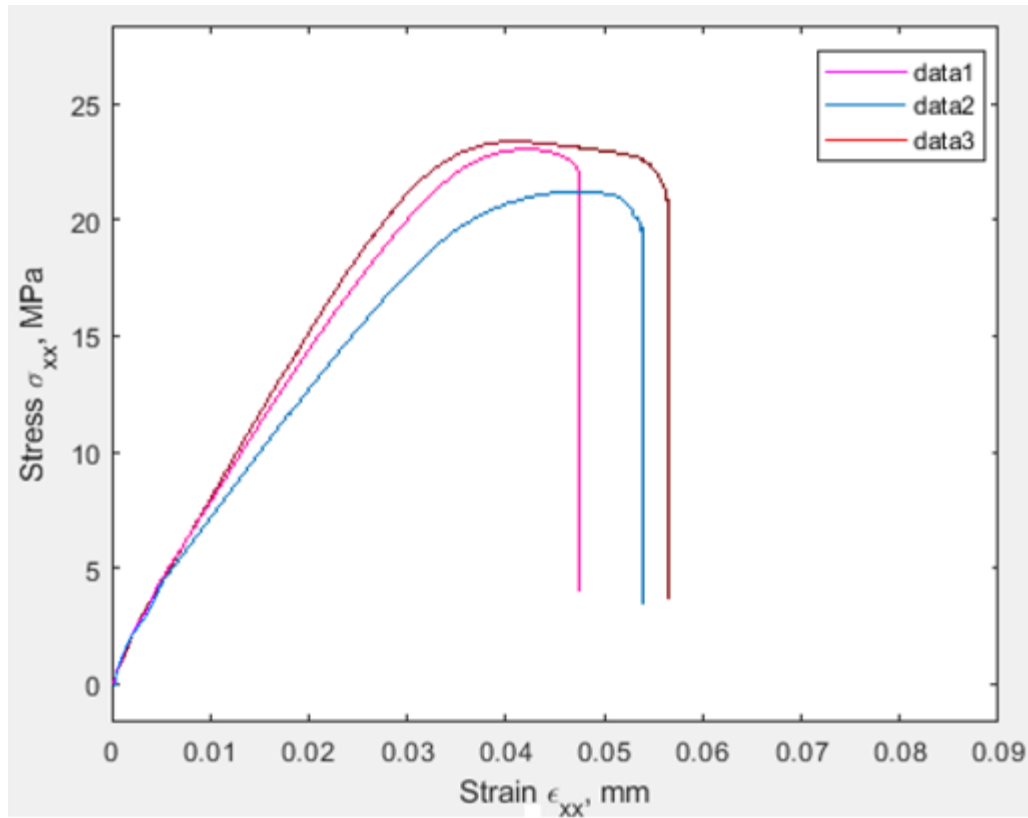


Figure 7-4 Stress vs Strain Curve for pure ABS.

7.4.2 TENSILE TEST OF 0.5% COPPER NANOPARTICLES:

Table 7.2-0.5% Copper Nanoparticles Ultimate Tensile Stress

0.5% Cu Nanoparticles	Ultimate Tensile Stress (MPa)
Sample 1	25.4097
Sample 2	24.6301
Sample 3	25.0063

0.5% copper nanoparticles has the highest Ultimate Tensile Stress with an average of 25.0153 MPa. The following graph 7-5 shows the stress strain curves [74] for the three various 0.5% copper nanoparticles samples.

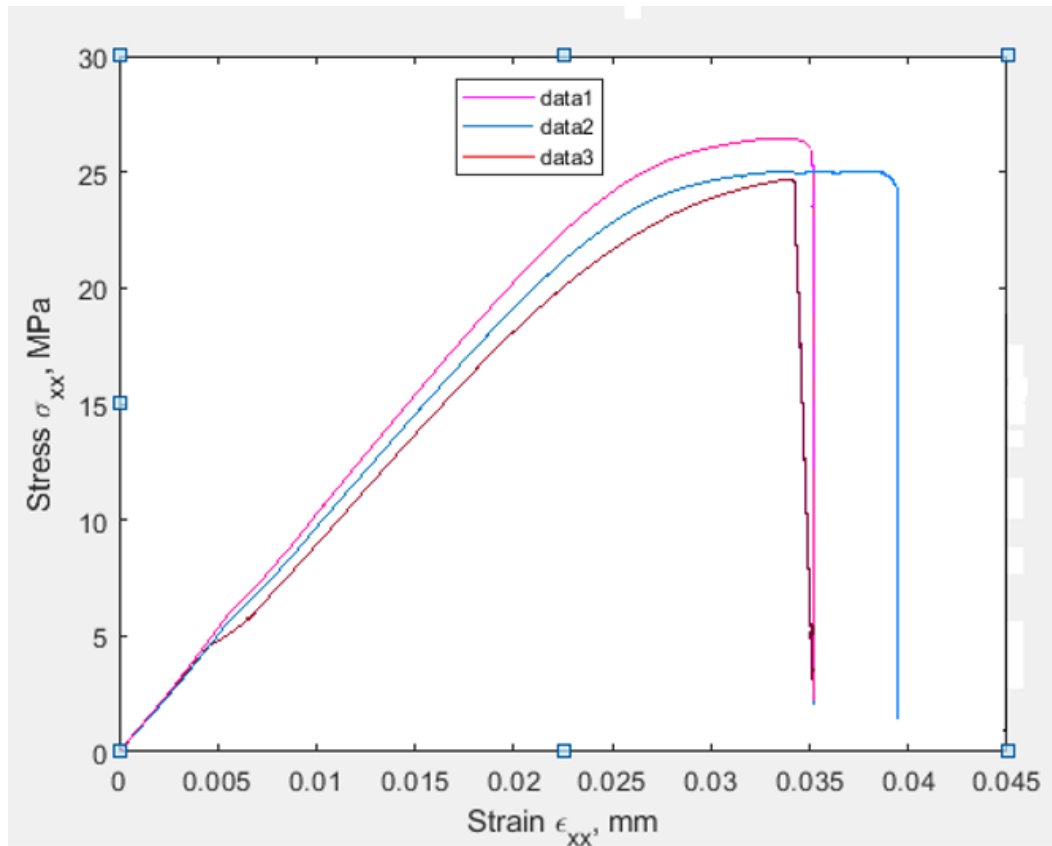


Figure 7-5 Stress vs Strain Curve for 0.5% Copper Nanoparticles.

7.4.3 TENSILE TEST OF 1% COPPER NANOPARTICLES:

Table 7.3-1% Copper Nanoparticles Ultimate Tensile Stress

1% Cu Nanoparticles	Ultimate Tensile Stress (MPa)
Sample 1	23.4646
Sample 2	23.4451
Sample 3	22.6581

1% copper nanoparticles polymer has the Ultimate Tensile Stress with an average of 23.1892 MPa which is higher than pure ABS. The following graph 7-6 shows the stress strain curves for [74] the three various 1% copper nanoparticles samples.

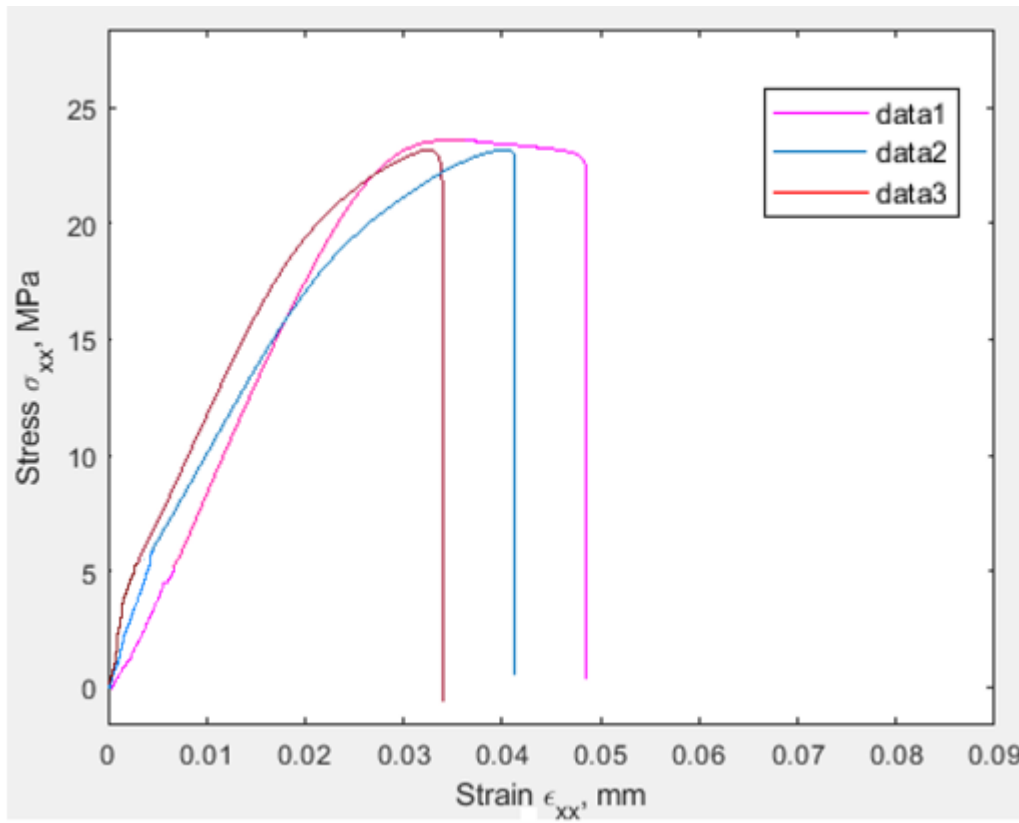


Figure 7-6 Stress vs Strain Curve for 1% Copper Nanoparticles.

7.4.4 TENSILE TEST OF 1.5% COPPER NANOPARTICLES:

Table 7.4-1.5% Copper Nanoparticles Ultimate Tensile Stress

1.5% Cu Nanoparticles	Ultimate Tensile Stress (MPa)
Sample 1	22.2883
Sample 2	22.9772
Sample 3	23.9505

1.5% copper nanoparticles polymer has the Ultimate Tensile Stress with an average of 23.072 MPa. The following graph 7-7 shows the stress strain curves [74] for the three various 1.5% copper nanoparticles samples.

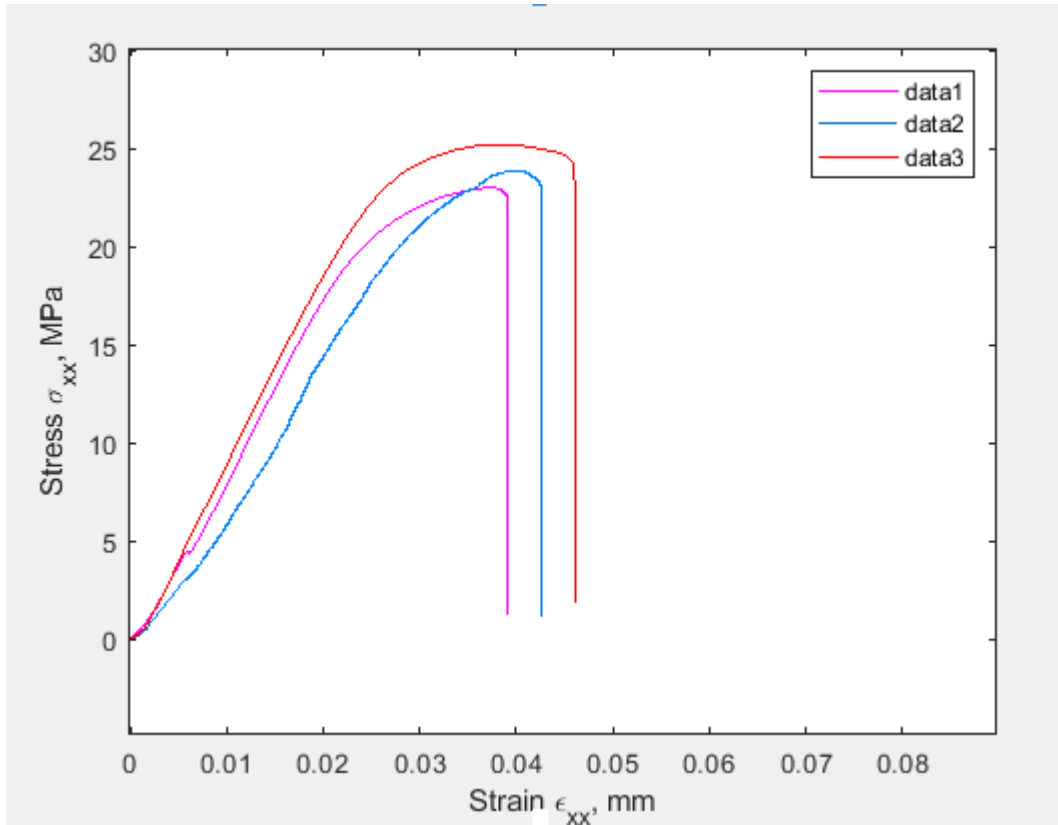


Figure 7-7 Stress vs Strain Curve for 1.5% Copper Nanoparticles.

7.4.5 TENSILE TEST OF 2% COPPER NANOPARTICLES:

Table 7.5-2% Copper Nanoparticles Ultimate Tensile Stress

2% Cu Nanoparticles	Ultimate Tensile Stress (MPa)
Sample 1	19.8491
Sample 2	20.6554
Sample 3	19.2110

2% copper nanoparticles composition has the lowest Ultimate Tensile Stress with an average of 19.9052 MPa. The following graph 7-8 shows the stress strain curves [74] for the three various 0.5% copper nanoparticles samples.

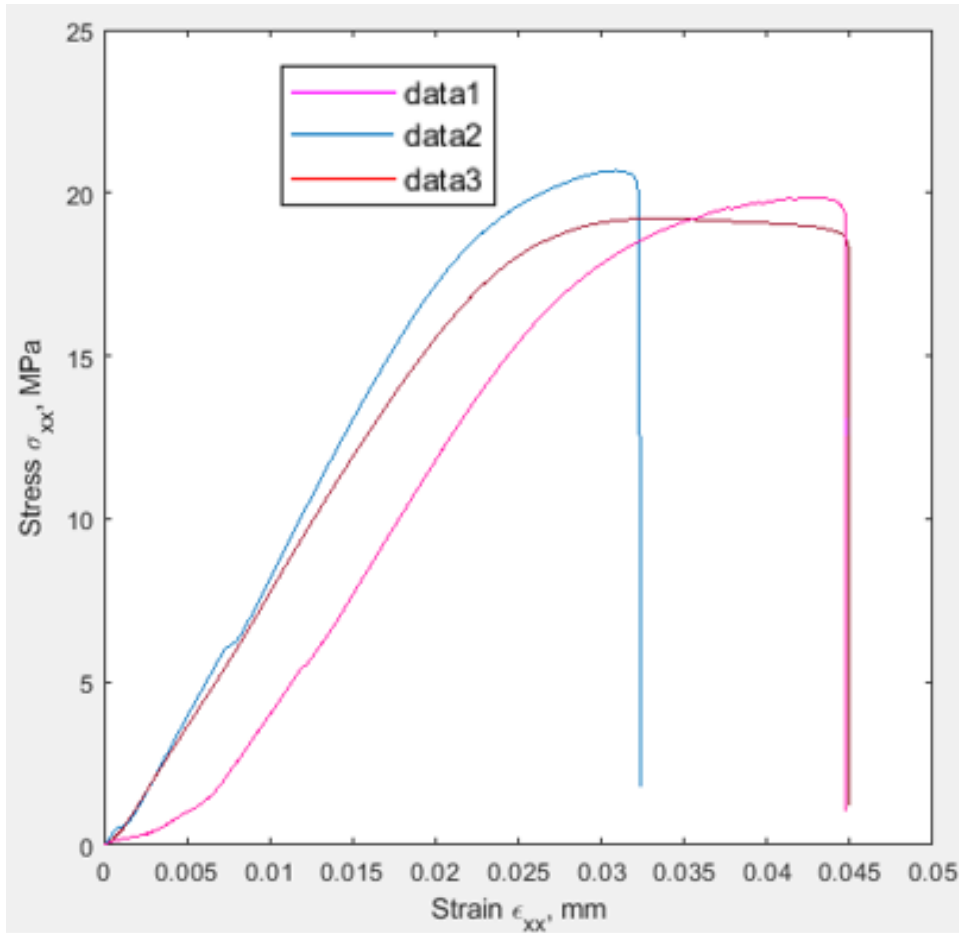


Figure 7-8 Stress vs Strain Curve for 2% Copper Nanoparticles.

7.4.6 GRAPH INCLUSIVE OF ALL TENSILE TEST:

The following graph 7-9 shows the varying percentages of copper nano particles including pure ABS along with their respective tensile stress. This graph helps us to easily understand the Tensile Stress of various

composition of polymer with varying composition including the range of all the test done for the same composition. As we have seen in the results as the percentage of copper nanoparticles were increased the material becomes more brittle and thus the mechanical properties are lost. We have also found out that as the percentage of copper nanoparticles were increased agglomeration of copper nanoparticles occur. To prevent this we could start with wet mixing to make sure agglomeration does not occur.

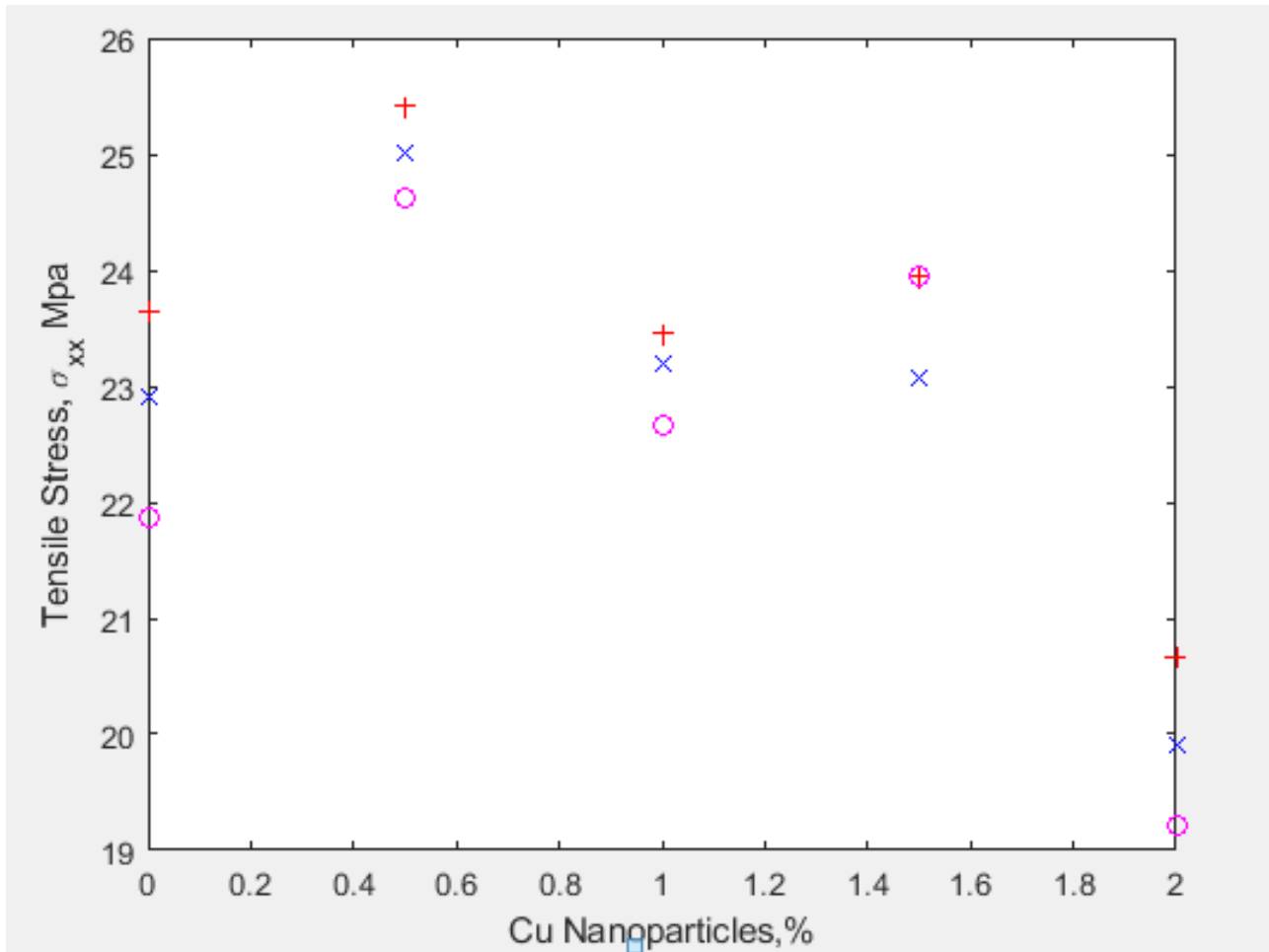


Figure 7-9 Ultimate Tensile stress for various composition.

CHAPTER 8 THERMAL CONDUCTIVITY

Thermal conductivity is one of the major part of this project. This section explains specific thermal tests done. We have conductivity in two aspects of the test the first is thermal conductivity of the extruded wire and the second part is trying to validate the thermal conductivity of the 3D printed parts. As indicated earlier, this part of this thesis was conducted in collaboration with Dr. Ankur Jain and his research group.

8.1 *THERMAL CONDUCTIVITY OF WIRE:*

Thermal conductivity [75] test is done to estimating the conduction of the wire with copper nanoparticles in comparison with pure ABS. The wires to be used are cut into equal length of 4 inches. The wires are cut from various sections of the spool to get a broad spectrum of the results. The wire conductivity experiment is done in comparison with the true sample, so this experiment produces only the percentage increase in conduction rather than an absolute value. The purpose of this reading is to understand the conductivity of the wire in comparison to pure ABS. As the main purpose is to explore the thermal aspect of the design as its main application of such polymers is in polymeric heat exchanger. Therefore, it is a measure of thermal conductivity of the unknown wire in comparison with that of the standard wire.

8.1.1 *THE EQUIPMENT USED:*

The equipment used here is a custom-built system to test the conductivity of the wires. The system is designed and built by Dr. Ankur Jain's lab. Here, this equipment is used to test thermal conductivity in polymeric nanocomposites. A brief description about this equipment is provided below.

According to the block diagram in figure 8-1(b), there is a heater plate which is maintained at a specific temperature so heat could be transferred to the aluminum block. The aluminum block is chosen as it is a good conductor of heat, copper is not chosen as it conducts heat rapidly. The aluminum block has holes are drilled equidistant from the heater block so that the wire samples can be inserted as shown in figure 8-1(b). An infrared camera is placed just above the wires to capture the thermal conductivity that

occurs in the wires. The figure 8-1(a) shows the actual experimental setup designed by Dr. Jain's lab and figure 8-1(b) shows the block diagram of the experimental setup.

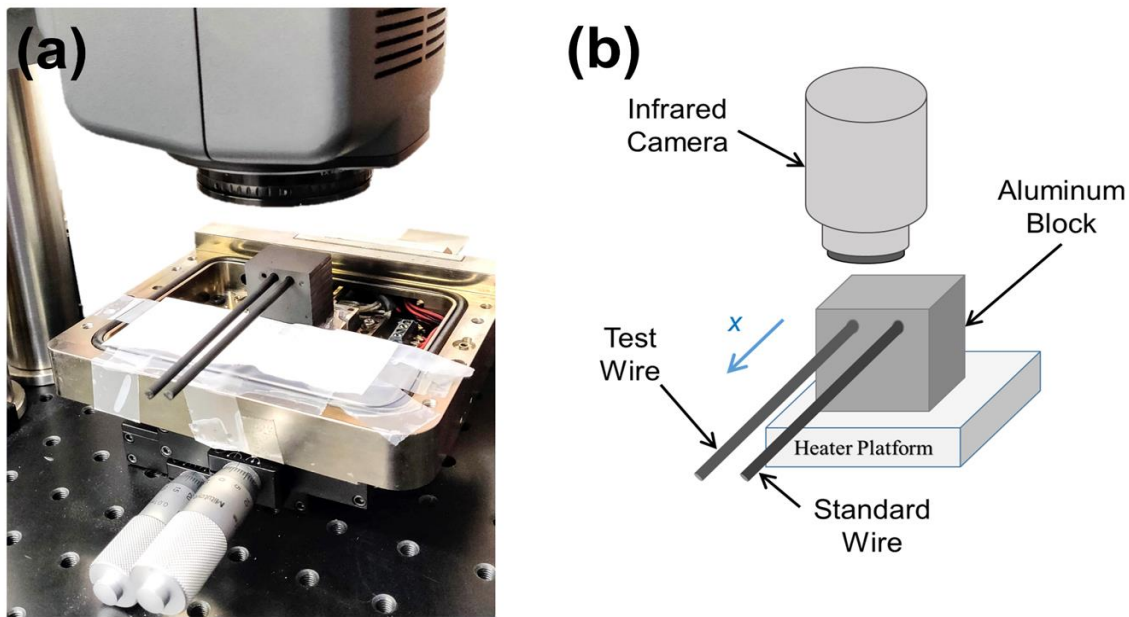


Figure 8-1(a) Thermal conductivity of wire setup, (b) Thermal conductivity of wire block diagram

[Designed by Dr. Jain's Lab, UTA].

The flowing figure 8-2 shows how the aluminum block has the holes drilled out to accommodate different diameters of the wires. The figure 8-3 shows the heater plate which evenly heats up the block. The experimental setup is verified by verifying the values that are obtained by measuring standard wires and then comparing it with the data taken from the manufacturers.

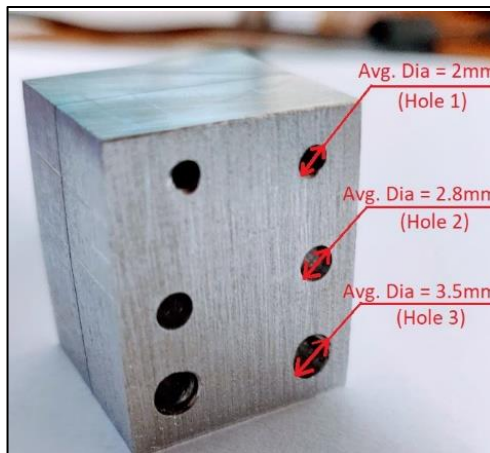


Figure 8-2 Aluminum block used for thermal conduction [Designed by Dr. Jain's Lab, UTA].

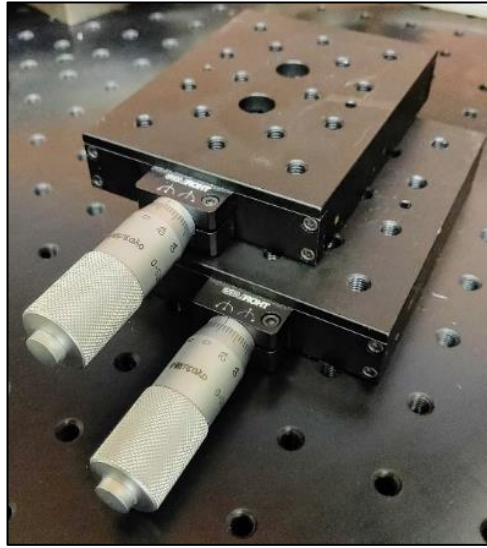


Figure 8-3 Base heater plate [Designed by Dr. Jain's Lab, UTA].

8.1.2 THEORY AND PRINCIPLE:

The basic theory that is used is on standard heat transfer in extended surfaces. The governing equation is for Fins of uniform cross-section area is:

$$\frac{d^2T}{dx^2} - \frac{hP}{kA_c}(T - T_\infty) = 0$$

And one of the governing equation:

$$\theta(x) \equiv T(x) - T_\infty$$

On subsuming the above-mentioned governing equation in the standard uniform cross-section of fins we get

$$\frac{d^2\theta}{dx^2} - m^2\theta = 0$$

Where m is:

$$m \equiv \sqrt{\frac{hP}{kA_c}}$$

Where m is the slope of the logarithmic nondimensional temperature distribution Vs Length.

h stands for conductive heat transfer.

A_c stands for area of correction of the wire.

k is value is simply shorthand for thermal conductivity.

Temperature distribution for infinitely long fin is as follow:

$$\frac{\theta}{\theta_b} = e^{-mx}$$

Since the slope is in exponential form, applying logarithm on both sides of the above equation we get

$$\ln \frac{\theta}{\theta_b} = -mx$$

So this equation is used to get the slope so that we can plot the graph to understand the thermal conductivity of the wire.

The following equation is used to determine the relative slope of the wire with and without the diameter inconsistency:

- Wires of different diameters -

$$k_{Unk} = k_{Std} \times \frac{m_{Std}^2 D_{Std}}{m_{Unk}^2 D_{Unk}}$$

- Wires of equal diameter -

$$k_{Unk} = k_{Std} \times \frac{m_{Std}^2}{m_{Unk}^2}$$

In the formula that we used K_{Unk}/K_{Std} is considered to be 1.

8.1.3 PROCUREMENTS OF THE GRAPH:

The infrared camera that is used to take the imaging of the wires which is used to get the slopes of the wires. Temperature distribution data for both the wires is taken from the IR camera frame by frame. We use ResearchIR software to synthesis the heat flow to get the slope. Each frame is 612 x 512. Each frame can capture the length of 8.5 mm approximately. Each pixel represents the dimension of 13.281 microns approximately. Figure 8-4 shows the image captured from the IR camera.

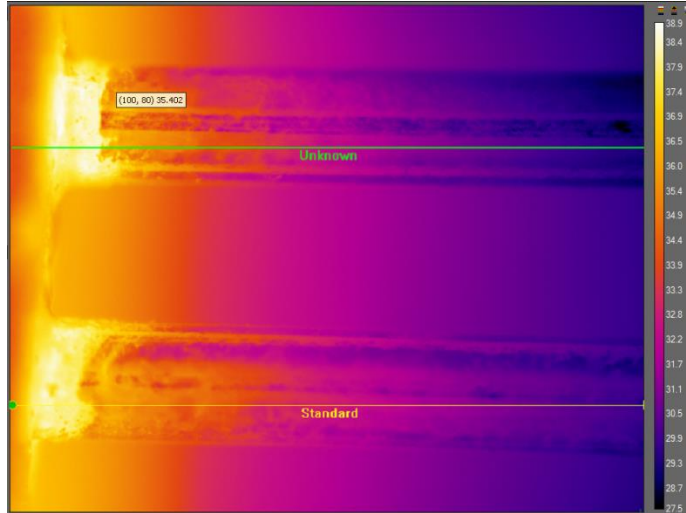


Figure 8-4 Image from IR Camera [provided by Mr. Swapnil Suryakant Salvi]

The figure 8-5 is obtained from the ResearchIR software which is taken for the sample material (Carbon Steel and Nickel Alloy Steel). And the figure 8-6 shows after the graph is stitched and cured for continuous even curve so the slope which is obtained from one single continuous curve.

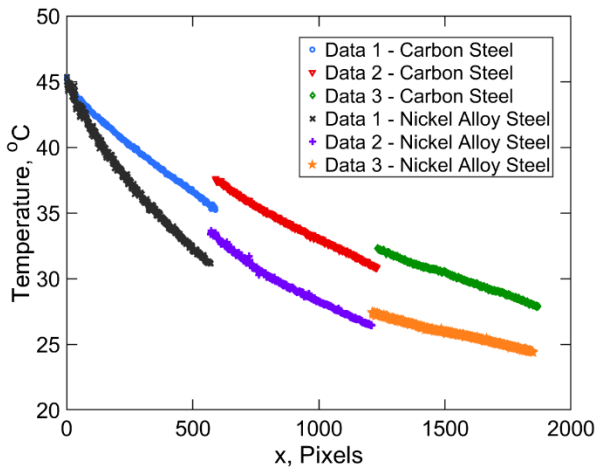


Figure 8-5 Slopes plotted from ResearchIR.

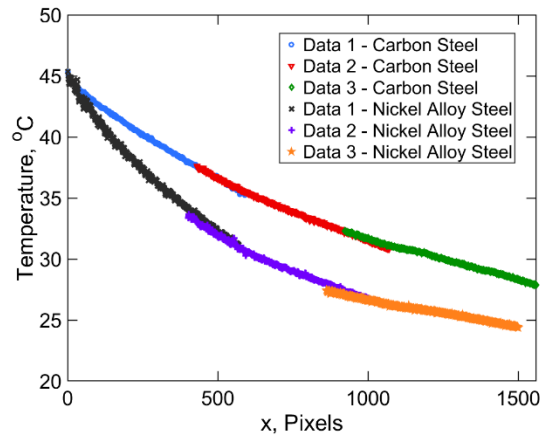


Figure 8-6 Cured and Stitched slopes.

[test data generated by Mr. Swapnil Suryakant Salvi]

8.1.4 RESULTS AND CONCLUSION:

The conductivity of the polymer is a comparison of copper nanoparticles induced ABS with pure ABS. Therefore, the figure 8-8 is a graph with percentage increase in thermal conductivity with respect to percentage of copper nanoparticles in ABS.

Table 8.1- Thermal conductivity of the wire

% Cu NPS in ABS	% Increase in Thermal Conductivity
0	0
0.5	2.12
1	16
1.5	29.4
2	30

From the above stated table 8-1, we know that as the copper nanoparticles increase the conductivity of the ABS as the theory suggested. The maximum is for 2% but there is no substantial increase from 1.5%. One of the reasons for this problem is believed to be due to agglomeration of copper nanoparticles which form into small bubble.

The following graph 8-7 clearly shows the increase in the thermal conductivity.

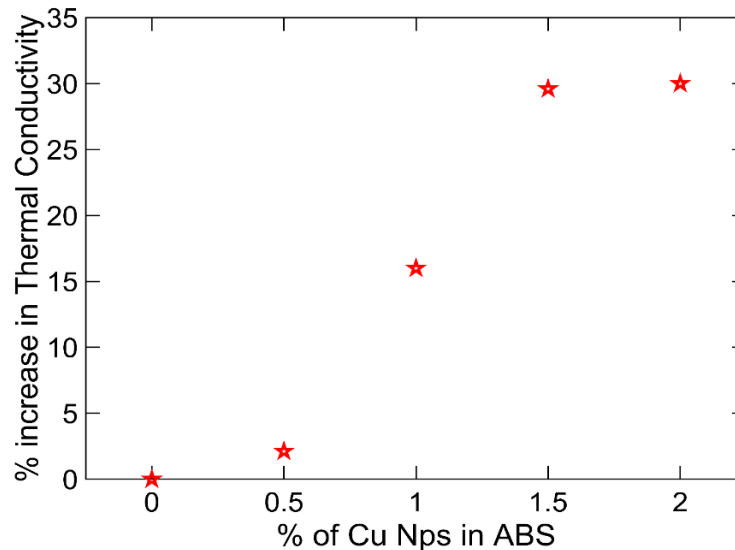


Figure 8-7 Plot of Thermal Conductivity [75] of Wire. [test data generated by Mr. Swapnil Suryakant Salvi]

8.2 THERMAL CONDUCTIVITY OF PRINTED PARTS:

To compare the thermal conductivity [76] of the wire to the thermal conductivity [76] of the part, the parts are printed, tested and the cross section is studied under microscope to provide us with a clear picture.

8.2.1 FOX50:

FOX50 is the most commonly used heat flow meter which is one of the most accurate instruments which is very easy to use FOX50 to provide rapid results. This instrument is used for identically high-performance features. This instrument is for thin heat flux transducers, uses digital thickness measurements. FOX50 has a high responsive temperature controller and has a integrated contact-resistance correction [77].

As shown in Figure 8-8, FOX50 has a very wide range of temperatures to choose from, FOX50 is an accurate choice for measurements as it covers a wide range of medium-conductivity materials such as plastics, glasses, ceramics, composites, concrete and many more materials [77].

Because of its compact size and its cost-effectiveness FOX50 system is used for measuring thermal conductivity. FOX50 has a solid-state heating and a cooling system for exact temperature control. It comes with a optical encoder for the precise measurements of sample thickness. It has a proprietary thin film heat flux transducer for repetitive testing of similar samples. Pyrex reference standards for calibration and verification. FOX 50 also comes with an automatic sample feeder for high-throughput analyses [78].

The specification of the FOX50 are as follows in table 8.2:

Sample Diameter	51 mm ²
Temperature Range	-10 °C – 110 °C

Temperature Resolution	± 0.01 °C
Thermal Conductivity Range	0.1 to 10 W/mK
Accuracy standard	$\pm 3\%$
Heat Flux Transducers Area	25 x 25 mm
Instrument Dimension	250 x 170 x 360 mm
Instrument weight	11 kg

Table 8.2- Specification of FOX50.



Figure 8-8 FOX50 Equipment [83].

8.2.2 SAMPLE SIZE AND PRINT PROPERTIES:

The samples that were used for the testing are of the following dimensions:

- 30 x 30 x 8 mm [79]
- 30 x 30 x 4 mm [79]

The parts are printed with the following printed parameters in mind:

- The speed of printing is maintained at 3600 mm/min.
- The thickness of the layer is at 0.2 mm.
- Infill percentage is maintained to be at 100% which is a standard.
- The temperature of the nozzle is maintained to be at 220 °C.
- The base is maintained at 100 °C to prevent it from warping.
- The filament that is extruded from the nozzle is around 0.4 mm.

The parts are printed in positive Z direction as shown in figure 8-19. The heat flows through the positive Z direction, i.e., flow of heat is perpendicular to the direction of the flow of the fibers [80].

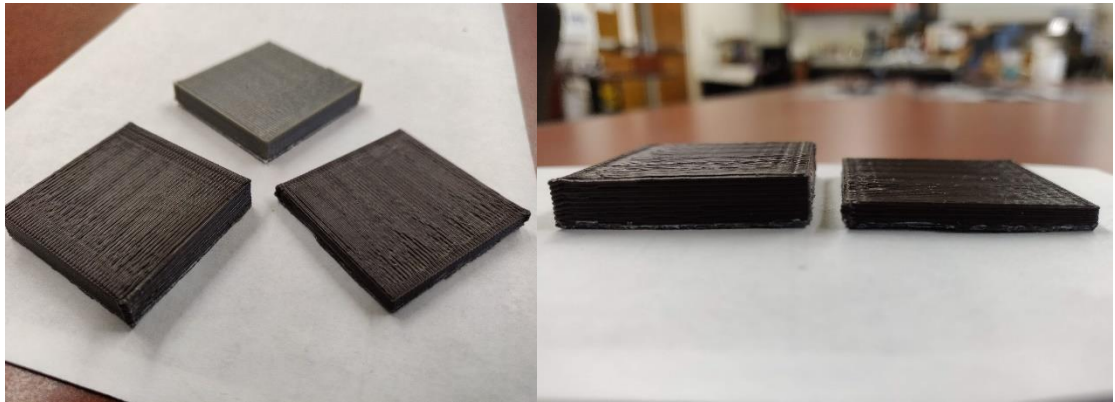


Figure 8-9 FOX 50 printed parts [printed by Mr. Swapnil Suryakant Salvi]

8.2.3 RESULTS AND DISCUSSION:

Various sample polymer from pure ABS to 2% copper nanoparticles with an increment of 0.5% copper nanoparticles are printed and tested and the results are as follows:

Table 8.3-FOX50 results of printed part.

% Cu NPS in ABS	Thermal Conductivity W/mK
0	0.150
0.5	0.156
1.0	0.158
1.5	0.144
2.0	0.128

The values are then plotted into a graph 8-10 which appears as following:

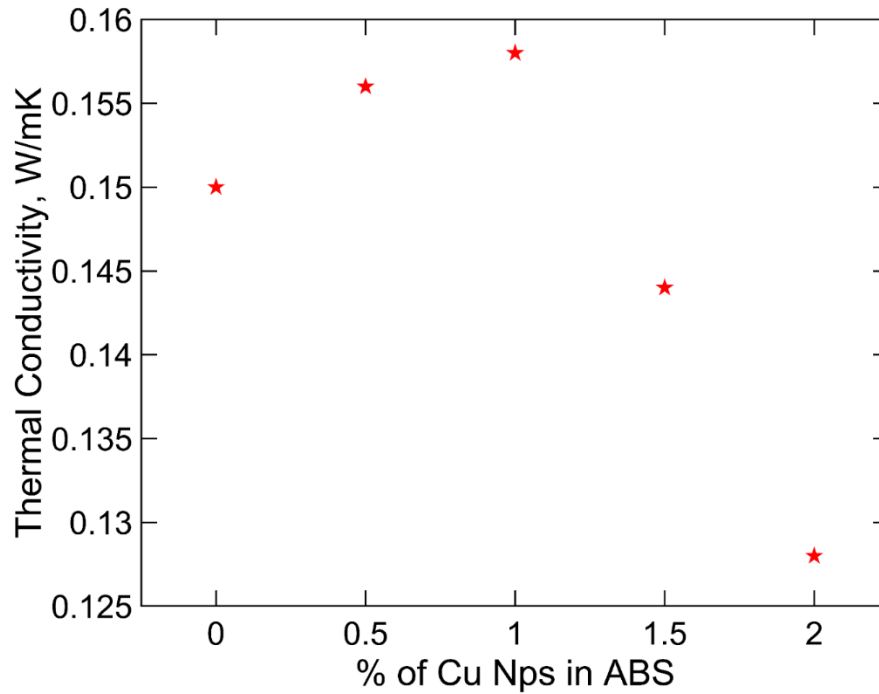


Figure 8-10 Graph of FOX50 results. [test data generated by Mr. Swapnil Suryakant Salvi]

We can see from the graph that there is a gradual trend in increase in conductivity till it reaches 1% and then it drops drastically with increase in the percentage of copper nanoparticles. According to theory, with the increase in the copper nanoparticles [81] the thermal conductivity of the part should increase like the thermal conductivity of the wire. But that does not occur as there are multiple factors that affect printing of the parts. The major factors that affect the thermal conductivity of the parts include, the parts not being properly placed between the two plates. If the parts are improperly placed and if there is an unevenness in printing of the part there is variation in the values as air acts as a perfect insulator, this would lead to false values.

Other reason is also because of the presence of voids in the parts. This would lead to improper conduction of heat through the parts as air acts as a perfect insulator. The voids that occur in the parts may be for the following reasons: because of the agglomeration of the copper nanoparticles in the wire that forms small voids which form like a small bubble. These bubbles collapse during printing and causes improper deposition. The other reason is because of the rapid heating and rapid cooling of the filament that is deposited, because of this shrinkage of the wire during deposition there is a creation of voids which leads to false readings. The presence of copper nanoparticles help this rapid heating and cooling because copper nanoparticles cannot hold heat for a longer duration of time. We believe this problem increases with increase in the copper nanoparticles.

To show the voids in the printed parts, the parts are taken and are frozen using liquid nitrogen then grooved a little on all the sides to create weakness so breaking the part is easy and occurs in that specific plain. If the part is cut into two pieces using hack saw blade this would lead to improper surface to study as the layer would be distorted because of the cutting motion. To prevent this from happening we freeze the parts and break them to get a perfect profile across the surface. The following image 8-11 and 8-12 shows the contrast and the difference between the two parts pure ABS and 2% copper nanoparticles. The voids are approximately around 1.5 microns.

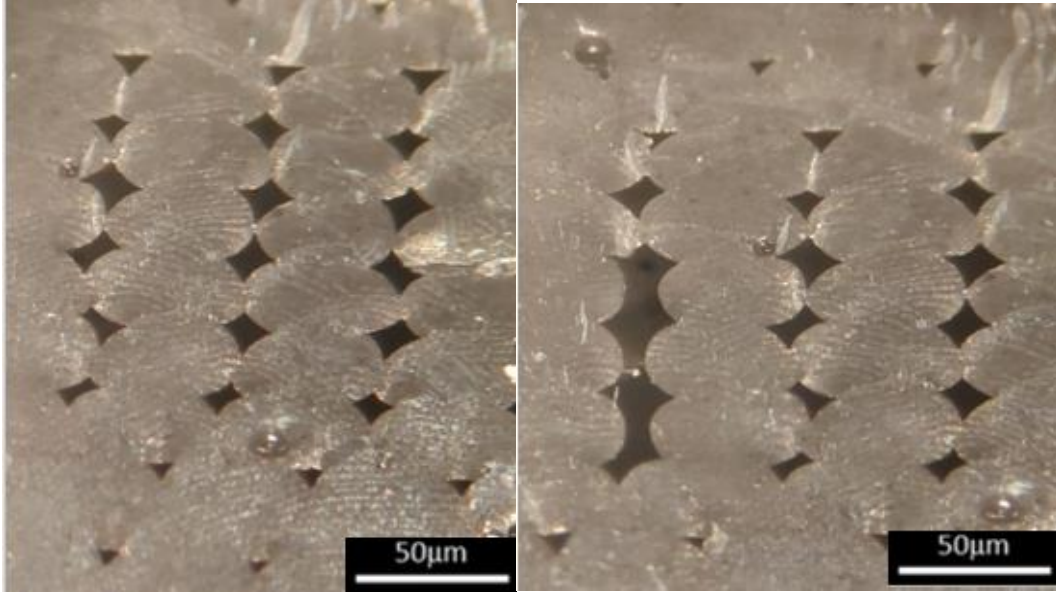


Figure 8-11 Microscopic image of pure ABS. Figure 8-12 Microscopic image of 2% Cu NPS.

[test data generated by Mr. Swapnil Suryakant Salvi]

CHAPTER 9 CONCLUSION

The research that is done here is substantial used to increase the conductivity of ABS by trying to keep the mechanical properties intact. As expected, the mechanical strength of the polymer when copper nanoparticles are added, is maximum at 0.5% copper nanoparticles composition but then it starts decreasing as expected. Thermal conductivity by adding 2% copper nanoparticles in ABS has increase the conductivity by approximately 30% in the wires. This however is not translated into the printed parts. The thermal conductivity in the parts are lowest for 2% copper nanoparticles infused with ABS. this problem must be rectified by varying various printing parameters so that the voids in the printed part do not occur. We also wanted to increase the percentage of copper nanoparticles up to 8%, the extruded wire is too brittle because of the agglomeration of copper nanoparticles. To enhance further study of the increase in copper nanoparticles in ABS a different manufacturing method must be chosen. The following image 9-1 is a microscopic image of 8% copper nanoparticles infused with ABS.

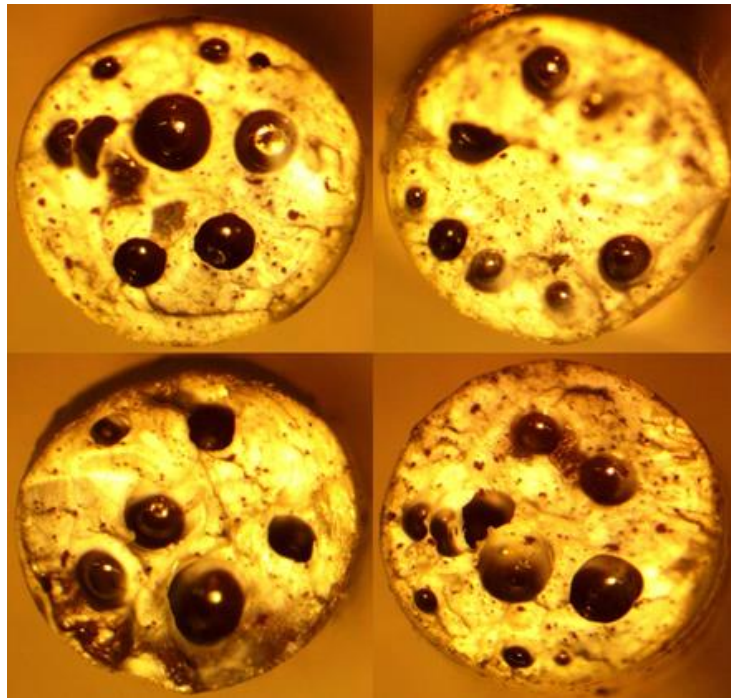


Figure 9-1 Microscopic image of 8% Cu NPS wire.

[test data generated by Mr. Swapnil Suryakant Salvi]

CHAPTER 10 FUTURE WORK

Future work includes changing of the mixing procedure of copper with ABS with wet mixing. This would eliminate the chances of agglomeration of copper nanoparticles either during the mixing procedure because of the use of blender which may create static electricity. The other aspect which can be improved is 3D printing of the parts so that voids do not occur. This problem can be avoided by using a different printer or by varying printer parameters. Other important aspect that could be used is by pre and post heat-treating of the printed parts which helps us in reducing the voids present in the printed parts. A throw SEM study could be done to understand the problems that occur in the printed layers. This study would give us a clear understanding of the properties that occur in the Nano scale. More research could include the varying of differing copper nanoparticle sizes and understand the variation of thermal and mechanical properties [82]. This research could be extended to use micro particles and understanding their effects on thermal and mechanical properties.

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BIOGRAPHY

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