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Mechanical and Electrical Behavior of Polyethylene Terephthalate Glycol (PETG) Reinforced with Multiwall Carbon Nanotubes (MWCNT) by using Fused Deposition Modeling 3D Printing

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Abstract - *Additive manufacturing has become flexible technique to manufacture parts that are highly complicated in shape. 3D printing has changed the production system with great flexibility of custom manufacturing, high precision, and capability of furnishing composites materials. Yet the challenges are to develop new dynamic materials or composites which can replace metallic parts used in aerospace, automobile and various sectors. The objective of this research was to characterize the PETG filament reinforced with MWCNT, study it's mechanical and electrical characteristics and compare it with pure PETG by means of experimental analysis. The MWCNT reinforcement fraction of 3%, 7% and 10% per kg weight of PETG was proposed for manufacturing the 3D printing filament and the mechanical tests were carried out according to ASTM D638 standards. It was observed that more percentage of carbon leads to more porosity of the composite i.e. filament becomes brittle in nature.*

Key Words: 3D printing, Polyethylene Terephthalate Glycol, Multiwall Carbon Nanotubes, composites, thermoplasts, reinforcement.

1. INTRODUCTION

Multiwall carbon nano tubes have extremely high electrical and thermal conductivity than that of metals [1]. These nanoscale materials are great filler materials to improve strength, stiffness, hardness, thermal conductivity of the polymer. In Fused Deposition Modeling the component is manufactured using the concept of rapid prototyping and layer by layer deposition of the filament. There are many filaments that are used for 3D printing. Most of the popular materials are ABS (Acrylonitrile Butadiene Styrene), PLA (Polylactic Acid), PETG (Polyethylene Terephthalate), PVC (Poly vinyl Chloride), PP (Polypropylene). All these polymers show different characteristics. There are various advantages of additive manufacturing like freedom of design constraints, reduced wastage of materials, reduced health hazard as compared to the traditional techniques used. In 3D printing process, the product design is made using CAD softwares like Catia, Autodesk, Solidworks, etc and is further exported as .STL file fomat. Cura, pronterface, slic3r are printing media software which enhances the printing method of the product. This software slice the object into smallest possible layers. However the mechanical strength of these thermoplastic products is very low as compared to the conventionally manufactured products.

Many researchers studied that addition of carbon nanotubes in these thermoplastic changes its behavior. The mechanical strength increases twice of that of pure materials [1, 2]. Carbon nano particles show unique behavior of mechanical and electrical hence they have been in used in many 3D printing filaments. H. Kürşad Sezer and Oğulcan Eren et al. presented the tensile strength of 3D printed parts has increased considerably (up to 58 MPa equivalent to 28%increase) with 7 wt. % addition of MWCNTs with ABS. However, ductile to brittle transition was observed with increasing MWCNT concentration [1]. George Tsiakatouras et al. investigated on comparative study of nano tubes reinforced with carbon filaments for 3D printed mechanical parts [4]. IpekBayraktar et al. fabricated a silver nano wire (Ag NW) loaded Polylactide (PLA) nano composites by simple solution mixing method and then 3D printed to obtain desired shapes. Their research shows that nano composites with higher Ag NW loadings showed better results than small amount of Ag NW loading to PLA which was found sufficient for nano composites to show reasonable antibacterial performance at low cost. D. W .Abott et al. studied finite element analysis of 3D printed model and concluded that the 50% infill simulations behaved as expected when compared to that of the dimensions of the 100% infill, representing almost double the displacements [17]. Daniel Farbman et al. reported mechanical behavior of 3d printed parts depending on print orientation, rate of infill, infill structure and CAD design. They concluded that ultimate tensile strength (MPa/g) decreases as the infill percentage decreases and that hexagonal pattern infill geometry was stronger and stiffer than rectilinear infill [18]. Adrián Rodríguez-Panes et al. presented a comparative study of the tensile mechanical behavior of pieces produced using the Fused Deposition Modeling (FDM) additive manufacturing technique with respect to the two types of thermoplastic materials: polylactide (PLA) and acrylonitrile butadiene styrene (ABS). Their aim was to compare the effect of layer height, infill density, and layer orientation on the mechanical performance of PLA and ABS test specimens [paper no]. S. Kanagaraj et al. shows that CNT reinforced

HDPE composites show a good enhancement of mechanical properties with an increase of CNT concentration [19]. T. Venkata Ramana et al. carried the design and manufacturing of crankshaft with CATIA V5 software by using of machine design. The crankshaft changes the reciprocating motion of the piston pin and connecting rod. The crankshaft was designed by multi cylinder engine and its 3D model was created using modeling software [paper no]. Yogesh Avula et al. focuses on modeling and 3D printing of differential permit each of the driving wheels to rotate at different speeds, while for most vehicles supplying equal torque to each of them. A vehicles wheel rotates at different speeds, mainly when turning corners. The differential was designed to operate a pair of wheels with the same torque while permit them to rotate at different speeds [11]. Ryan D et al. worked on 3D printed fluids and 3D printed microelectronics. They evaluated distinctive benefits and constraints; unfortunately, recent works has revealed that current 3D printing techniques that are beneficial for micro fluidics manufacturing suffer from numerous limitations in the context of micro electronics fabrication and vice versa. They have demonstrated that extrusion-based 3D printing methods such as DIW (Direct ink write) provide significant advantages compared together additive processes for fabricating microelectronic system [13]. Yeong-Jae Lee et al. experimented the friction performance of 3D printed ball bearing was evaluated to verify its feasibility as an engineering part. Multi-jet printing (MJP) method was conducted to prepare the ball bearing specimen as it provides the advantages of straightforward manufacturing process and high surface quality and the friction performance and feasibility of 3D printed ball bearings was evaluated. They concluded that 3D printed bearings can be used under constant load and rotational speed [14]. Erin Baynojir Joyee et al. studied the influences of spherical particle chain orientations, volume loading fractions, and 3D printing layer thickness on the stress-strain behavior of 3D printed heterogeneous particle-polymer composites. They conducted mechanical tests and investigated analytical models, to understand how the elastic behavior of the polymer composites corresponds to distribution and orientation of spherical particle assemblies in polymer matrix. It was observed that the influence of 3D printing layer thickness on Young's modulus is negligible. However, the printing layer thickness parameter has some effects on the breaking propagation path due to the internal structure defects associated with large printing layer thickness. Hence to achieve enhanced mechanical properties with reinforced architecture for 3D printed assembled spherical particle-polymer composite, the sample should have a high particle loading fraction and parallel particle chain orientation.

According to extensive research, very few researchers have focused working on PETG reinforced MWCNT filament. Challenges are to make the filament tough, more sustainable to working conditions so that it could meet the replacement of metal parts and to achieve high strength.

2. EXPERIMENT

2.1 Material selection

The very challenging task was to select suitable additive for plastic. According to extensive search found out that Multiwall Carbon Nano Tube (MWCNT) when added into the thermoplastic polymer, it improves the property of plastic. Small addition of multi-walled carbon nano tubes improves the mechanical performance, thermal conductivity, electrical conductivity, and permeation barrier properties of a wide range of polymers and metal composites without significantly changing the product cost. 200 gram of MWCNT powder was purchased from Adnano Technologies Pvt. Ltd. Karnataka.

Applications of MWCNT:

Multiwall carbon nanotubes has been recommended for polymers and metal composites, paints and coatings, civil construction material, oil and refineries, inks, batteries, desalinations, solar, energy conversion, hydrogen storage, super-capacitors, electromagnetic wave absorption and shielding, catalysts, super capacitors, textiles, electronics and sensors etc.

Table 1 Technical parameters of MWCNT

Multi-walled Carbon Nanotubes	*Description*
Purity	99 %
Outer diameter	10-30 nm
Inner diameter	5-10 nm
Length	> 10 µm
Surface area	110- 350* m^2/g
CNT density	95-99%
Bulk density	$0.14 g/cm^3$
Chemical formula	C
Physical form	Fluffy, very light powder
Odour	Odourless
Colour	Black powder

Polyethylene terephthlate glycol:

PET stands for Polyethylene Terephthalate and G stands for glycol. PETG is a plastic resin of the polyester family that is used to make beverage, food and other liquid containers, as well as for some other thermoforming applications. PETG is a clear amorphous thermoplastic that can be injection molded or sheet extruded. PETG was in the form of granules.

Table 2 Characteristics of PETG

Chemical formula	(C10H8O4)n
Molar density	Variable
Density	1.38g/cm^3 at 20°C Amorphous: 1.370 g/cm^3 Single crystal: 1.455g/cm^3
Melting point	240°C
Boiling point	> 350° C (decomposes)

PETG is handily framed, easily cut and punched; PETG is a strong decision for applications wherein complex shapes, subtleties, profound draws and compound bends are highlighted. This thermoplastic material, polyethylene terephthalate glycol, can also be painted, silk screened or hot stepped and is incredibly tough, PETG offers engineers extraordinary plan opportunity and are reasonable for some applications. With preferred effect quality over acrylic and lower cost than polycarbonate, PETG is the prevalent decision for some complex manufactures.

Here are the fundamental advantages to printing with this material and regular PETG fiber properties:

- PETG has exceptionally low shrinkage, and in this way no twisting. Ideal for printing large stuff.
- PETG is likewise extremely solid; it's not fragile but rather can be scratched more effectively than ABS which is more enthusiastically.
- PETG plastic makes a horrendous help structure, since it sticks so well. But since it sticks so well, layer bond is fabulous, so prints come out solid.
- It adheres well to the print bed as well, so be cautious when you're eliminating it subsequent to printing.
- It has an incredible substance obstruction, alongside soluble base, corrosive and water opposition.
- Odourless when printing.
- Typically Polyethylene fiber is provided in a scope of clear tones, and prints with a pleasant shiny completion.
- It makes it ideal for printing whatever should be shatterproof or clear. Many are taking the jump from utilizing PLA or ABS to simply utilizing PETG.

Applications of PETG:

PETG is used in casing, covers, mobile guards, camera guards, PETG withstand properties of ABS and PLA hence it is used in medical braces, graphic displays monitors, etc.

2.2 Manufacture PETG filament with 3%, 7%, 10% MWCNT loading

The fabrication of filament was done on single screw extruder machine of 1.75mm diameter screw at "Rever Industries Pvt Ltd, Nashik, Maharashtra, India" The company is famous for manufacturing and distributing 3D printing filaments across India.

Fig. 3. Single screw extruder

Calculations:

- 1000 gram PETG = 100% Hence 3% = 30 gram of MWCNT
- 1000 gram PETG = 100% Hence 7% = 70 gram of MWCNT
- 1000 grram PETG= 100% Hence 10% = 1000gram of MWCNT

PETG was in the form of granules and transparent colour. Granules don't come with colors. Hence black colour was chosen. Then, 3 spools of PETG reinforced MWCNT was manufactured using single screw extruder. The compounding and mixing of additives was done inside the rotating blender separately at speed of 360 revolutions per minute for 1 hour at 60°C for each percentage of MWCNT loading. Extrusion process is done faster due to low diameter of screw. 1kg of PETG/MWCNT wire was extruded into 320 meter long wire form suitable for 3D printing. The filament coming out of extruder is passed through the laser gauge to monitor consistency of diameter of wire before becoming a spool.

2.3 Design and 3D printing of composite material into standard specimen according to ASTM standards for plastics testing.

Design was implemented according to ASTM D638 standard rules for mechanical testing of plastic composites as shown in the figure 4.

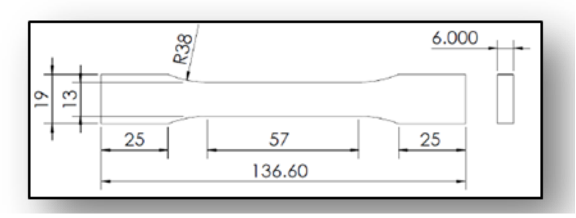


Fig. 4. ASTM D638 standard size of specimen

The CAD model was prepared on CATIA V5 21 software and the converted into .STL file from the software itself. The .STL represents standard tessilation language file which is readable by printing media called as "CURA" which is nothing but the slicing media. The specimen was 3D printed in horizontal orientation at standard default printing format at speed of 40mm/sec on 0.4mm brass nozzle. The bed temperature was set to 70°C and hot end temperature was set to 250°C. The 3D printer of Creality Ender 3 (200x200x300) mm was used. The different specimen of different percentage loading was 3D printed successfully as shown in figure 5.

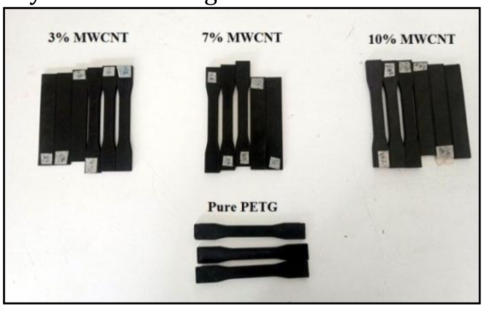


Fig. 5. 3D printed specimens

2.4 Annealing 3D prints

Annealing is the process of heating a part to cause its structure to reorganize. Glass transition temperature of PETG is 85° C. Annealing 3D prints could make 3D prints stronger and heat resistant. During the annealing process the polymer chains that are initially in dominant of amorphous state are re-arranged to crystalline state (orderly fashioned). The crystallinity increases along with change in structure. 0%, 3%, 7%, 10% each samples are annealed at 85° C for 45 minutes and then natural cooled. The process of annealing is carried out in IFB 23L Microwave oven figure 6. Here the 3d printed parts got deformed as shown in figure 7.

Fig. 6. IFB 23L Microwave oven

Fig. 7. Warping of 3D prints

The annealed parts got uplifted from the platform which is called as warping of prints. This happens mainly due to reduced in porosity of material, release of internal stresses, re-arranged structure, etc. This leads to change in geometry of parts.

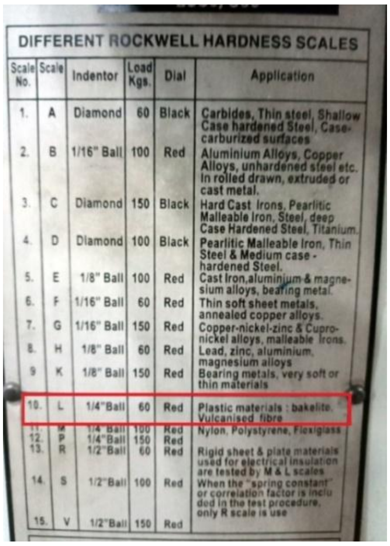
3. MECHANICAL AND ELECTRICAL TESTING

3.1 Hardness test:

Hardness test was carried out on a Rockwell Hardness Testing machine as shown in figure 8. Here L scale was used for composite plastic material. Hence, ¼" ball indentor was used and 60 kg load was applied. Red colour dial was used to note load applied on the specimen.

The Rockwell hardness tester was having the capacity of 150kg load whilst for plastic composite material 60kg load was applied. The flat surface of specimen was kept on the platform of Rockwell hardness tester. Load range can be adjusted using load knob on the right side of tester. The intendor penetrates inside the surface of the specimen while the needle remains stationary and the start unloading. The difference between the final and initial reading is nothing but the actual hardness of the specimen.

Fig. 8. Rockwell Hardness Testing Machine



DIFFERENT ROCKWELL HARDNESS SCALES

Scale No.	Scale	Indentor	Load Kgs.	Dial	Application
1.	A	Diamond	60	Black	Carbides, Thin steel, Shallow Case hardened Steel, Case-carburized surfaces
2.	B	1/16" Ball	100	Red	Aluminium Alloys, Copper Alloys, unhardened steel etc. In rolled drawn, extruded or cast metal.
3.	C	Diamond	150	Black	Hard Cast Irons, Pearlitic Malleable Iron, Steel, deep Case Hardened Steel, Titanium.
4.	D	Diamond	100	Black	Pearlitic Malleable Iron, Thin Steel & Medium case - hardened Steel.
5.	E	1/8" Ball	100	Red	Cast Iron,aluminium & magnesium alloys, bearing metal.
6.	F	1/16" Ball	60	Red	Thin soft sheet metals, annealed copper alloys.
7.	G	1/16" Ball	150	Red	Copper-nickel-zinc & Cupro-nickel alloys, malleable Irons.
8.	H	1/8" Ball	60	Red	Lead, zinc, aluminium, magnesium alloys
9	K	1/8" Ball	150	Red	Bearing metals, very soft or thin materials
10.	L	1/4"Ball	60	Red	Plastic materials : bakelite, Vulcanised fibre
11.	M	1/4"Ball	100	Red	Nylon, Polystyrene, Plexiglass
12.	P	1/4"Ball	150	Red	
13.	R	1/2"Ball	60	Red	Rigid sheet & plate materials used for electrical insulation are tested by M & L scales
14.	S	1/2"Ball	100	Red	When the "spring constant" or correlation factor is included in the test procedure, only R scale is use
15.	V	1/2"Ball	150	Red	

Fig. 9. Rockwell Hardness Scales

The following readings were obtained from Rockwell Hardness Test:

Table 3 Hardness test observations

Sample	Hardness value HRL	Annealed Hardness Value HRL	Increase in hardness due to annealing
Pure PETG	52	53	1.92 %
3% MWCNT	63	66	4.76 %
7% MWCNT	85	90	5.88 %
10% MWCNT	43	50	16.27 %

From the below figure 10 observations clearly shows that:

- Addition of MWCNT in pure PETG increases the hardness of pure PETG.
- Annealing makes 3D printed parts for harder than before.
- At 10% addition of MWCNT, hardness value found to be decreased because of increase in porosity of filament.
- More the percentage of additives, results in increase in porosity of filament material.

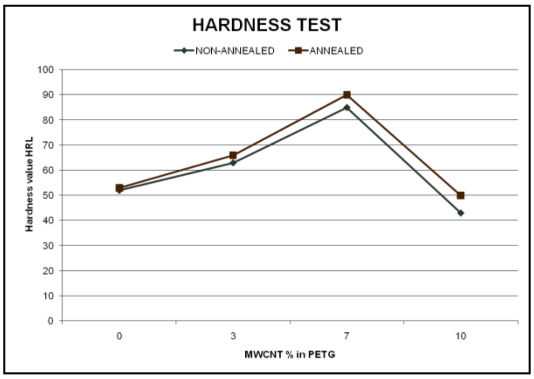


Fig. 10. Graphical representation of Hardness test

3.2 Dimensional observations:

1. The length of annealed tensile specimen got decreased after annealing as shown in figure 11 and figure 12 respectively.

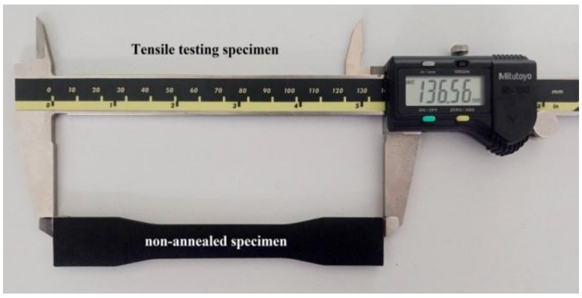


Fig. 11. Length of Non-annealed tensile testing specimen

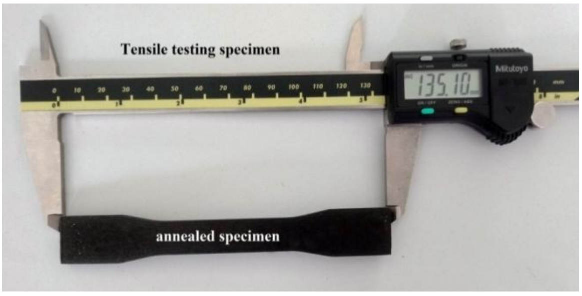


Fig. 12. Length of annealed tensile testing specimen

2. The thickness of annealed tensile specimen got decreased after annealing as shown in figure 13 and figure 14 respectively.

Fig. 13. Thickness of Non- annealed specimen

Fig. 14. Thickness of annealed specimen

3. The width of annealed tensile specimen got decreased after annealing as shown in figure 15 and figure 16 respectively.

Fig. 15. Width of Non- annealed specimen

Fig. 16. Width of annealed specimen

Table 4 below shows the change in dimension of the specimen after the annealing process. During the annealing process the polymer chains that are initially at amorphous state gets ordered into crystalline structure, hence the crystallinity of material increases after annealing process. Table no: 4.2 shows the specimen slightly got shrink from the original dimensions.

Table 4 Change in dimensions

Specimen	Non annealed	annealed
Length	135.56	135.10
Thickness	5.90	5.51
Width	19.17	18.83

3.3 Electrical conductivity test:

Electrical conductivity of Multiwalled Carbon Nanotube is higher than that of metals, hence when added to polymer it is expected to have electrically conductive material composite. Hence taking this into picture, an electrical conductivity test was carried out on a digital multi-meter. As shown in the figure 17.

Fig. 17. Digital multi-meter

Above figure 10 cm wire of each pure PETG, 3%, 7% and 10 % additive filament was taken for testing. The wire was placed right between the two probes of the multimeter and then different values of resistance were recorded.
The calculations for electrical conductivity:

By formula; R=ρL/A and σ= 1/ρ
Where R = resistance,
ρ = resistivity
L = length of wire =10 cm = 0.1 m
A= area of cross section = $\pi r^2 = 3.14 \times (1.75/2)^2$
$= 2.404\ m^2$
σ= Conductivity

Table 5 Multimeter readings

Sample	Resistance
3% MWCNT	0.031 Ω
7% MWCNT	0.025 Ω
10% MWCNT	0.0180 Ω

1. for 3% MWCNT additive:

$0.031 = \frac{\rho\ X\ 0.1}{2.404}$ Hence $\rho = 0.7452\ \Omega m$

$\sigma = 1.3259\ S/m$

2. for 7% MWCNT additive:

$0.025 = \frac{\rho\ X\ 0.1}{2.404}$ Hence $\rho = 0.6010\ \Omega m$

$\sigma = 1.6638\ S/m$

3. for 10% MWCNT additive:

$0.0180 = \frac{\rho\ X\ 0.1}{2.404}$ Hence $\rho = 0.4327\ \Omega m$

$\sigma = 2.311\ S/m$

Table 6 Calculated electrical conductivity

Sample	Resistivity	Conductivity
3% MWCNT	0.7452 Ωm	1.3259 S/m
7% MWCNT	0.6010 Ωm	1.6638 S/m
10% MWCNT	0.4327 Ωm	2.311 S/m

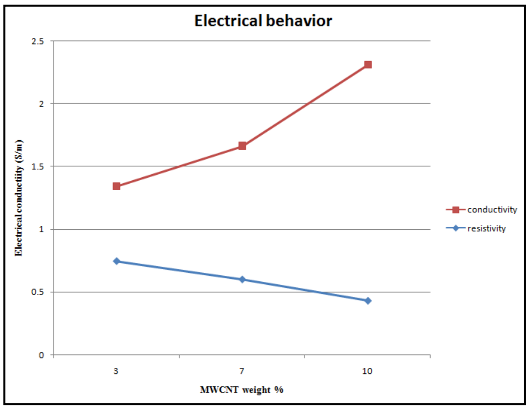


Fig. 18. Graphical representation of Electrical behavior

From the above calculations it is clearly seen that the electrical conductivity of filament increases when the percentage of MWCNT additive increases. Also the inverse relation of resistivity and conductivity is satisfied.

4. RESULTS

Figure 10, clearly shows that the hardness value of the PETG reinforced MWCNT got increased and then decreased.

- The hardness value of pure PETG was 52 HRL, when annealed it got increased to 53 HRL (1.92% increased).
- Similarly the hardness value 3% MWCNT additive was 63 HRL, which is greater than pure PETG. Also, when annealed it got increased to 66 HRL (4.76% increased).
- The hardness value of 7% MWCNT additives was 85 HRL, which is greater than 3% MWCNT additives. This when annealed it got increased to 90 HRL (5.88% increased).
- At 10% addition of MWCNT, there was a rapid change in the hardness value as it got decreased to 43 HRL. But when annealed it got increased to 50 HRL (16.27 % increased).

From the hardness test it can be concluded that, addition of MWCNT in pure PETG increases the hardness of PETG at a certain percentage loading. More the percentage of additives results in increase in porosity of material. Hence the proper bunching of additives is necessary. Moreover, it was found that the hot end temperature (i.e. Nozzle temperature) of PETG remained unchanged even after addition of MWCNT.

On the other hand, warping behavior of 3D prints was observed after annealing it. This occurs mainly in high temperature plastics as the melting temperature is greater than that of commercial plastics. When working with higher temperature plastics, warping can be a common issue. Warping is a phenomenon that the print deforms due the internal stresses releasing from the build platform. From table 4, it is clearly seen that the length of specimen got decreased from 135.56mm to 135.10mm, breadth got decreased from 5.90mm to 5.51mm and width got decreased from 19.17mm to18.83mm.

From figure 18, it is clearly seen that electrical conductivity gets increased as the amount of MWCNT gets increased. At 3% MWCNT additives the electrical conductivity was 1.3419 S/m it then got increased to 1.6638 S/m at 7% MWCNT additives. After that it got increased to 2.311 S/m at 10% MWCNT additives. From table 4.3 the resistance value of 3% MWCNT additive was 0.031Ω. It got decreased to 0.025Ω at 7% MWCNT additives. Also, at 10% it got decreased to 0.0180Ω. This is nothing but the inverse relation between resistivity and conductivity.

5. CONCLUSION

In this work, the effect of inducing nano-particles into the thermoplastic was investigated. The variation of result in this experiment found out to be very interesting. Multiwall carbon nano tubes have extremely high electrical and thermal conductivity than that of metals. These nanoscale materials are great filler materials to improve strength, stiffness, hardness, thermal conductivity of the polymer. The objective of this research was to characterize the PETG filament reinforced with MWCNT, study it's mechanical and electrical characteristics and compare it with pure PETG by means of experimental analysis. The MWCNT reinforcement fraction of 3%, 7% and 10% per kg weight of PETG was proposed for manufacturing the 3D printing filament and the mechanical tests were carried out according to ASTM D638 standards. During the tests it was found that addition of MWCNT in pure PETG around 7% loading increases the hardness of PETG and then rapidly decreases at 10%. Hence it can be concluded that, more the percentage of additives results in increase in porosity of material resulting in decrease in mechanical strength. Hence the proper bunching of additives is necessary. Moreover, it was found that the hot end temperature (i.e. Nozzle temperature) of PETG remained unchanged even after addition of MWCNT. On the other hand, warping behavior of 3D prints was observed after annealing it. This occurs mainly in high temperature plastics as the melting temperature is greater than that of commercial plastics. When working with higher temperature plastics, warping can be a common issue. Warping is a phenomenon that the 3D printed parts deforms due the internal stresses releasing from the build platform. Also our experiment got successful in taking electrical conductivity test. According to extensive research from literature survey, our fabricated material obeyed the same inverse relation between electrical resistivity and electrical conductivity. The electrical conductivity gets increased as we add more percentage of MWCNT loadings.

Future scope

- 3D printing technology will become smarter. Dentists will adopt this technology as a dominant production technology.
- There will be new composites filaments which will lead to huge market opportunity.
- Designing software will be more integrated and easy to use for the user.
- Metal 3D printing will continue to mature.

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