

Study of Mixing Ratio Effect on Physical & Mechanical Properties of Carbon fiber- Phenolic resin Composites



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NUST201260698MSCME67712F

**This thesis is submitted as partial fulfillment of the requirements for
degree of
MS in Materials & Surface Engineering**

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Jun 2016**

Certificate

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DEDICATED TO

Prophet MUHAMMAD

صلى الله عليه وآله وسلم

A Mercy to The Worlds

Acknowledgements

All Praise to **Almighty ALLAH**-the Expeditor, the Cherisher, the Nourisher and the Sustainer for His boundless mercies. I am really indebted to Him for blessing me with all strengths and capabilities to complete this research thesis. There are no words of acknowledgment that can do full justice to the everlasting love, never-ending prayers and sacrifice of my family, especially my parents. I simply could not have reached this stage without them. I dedicate all my success to my parents because they are the ones who put in all their efforts for my success.

I would like to express my heartiest incessant gratitude and the most sincere appreciation to my Supervisor Professor Dr. Ahmad Nawaz Khan for his valuable guidance, advice, inspiration and encouragement to the successful completion of this thesis.

Abstract

Over the last four decades, carbon fibers have emerged as the main reinforcement filler for high performance composite materials. They can be employed in various polymer matrices, however Phenolic based carbon composites are an attractive choice for high temperature aerospace application on account of their high strength and thermal stability. In present study, increasing chopped carbon fiber wt % (40 ~ 55 %) was employed in phenolic matrix to obtain best mixing ratio for optimum mechanical & thermal properties for a specific high temperature aerospace application. Samples were prepared by compression molding technique and then characterized for morphology, mechanical & thermal properties using SEM, Tensile Testing Facility, TGA & DSC technique. Mechanical properties test result showed that tensile strength, elongation, modulus and impact strength were increased by increasing fiber wt % in phenolic matrix. Ablation & TGA tests revealed that thermal properties were also improved by addition of carbon fibers to phenolic matrix. Glass transition temperature was however reduced by addition of carbon fiber to phenolic matrix. Analysis of test results concluded that 45 wt% Phenolic/Carbon Composite has shown optimum properties with better tensile strength, modulus, impact strength, good ablation rate and thermal decomposition temperature, fulfilling all requirements of specific application.

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1. Introduction

1.1 Composites

Composite materials are constituted from two or more materials that while forming a single component, they remain separate and distinct. These two materials must be chemically inert, however small degree of inter diffusion at the matrix and reinforcement interface is allowed to enhance bonding.[1]

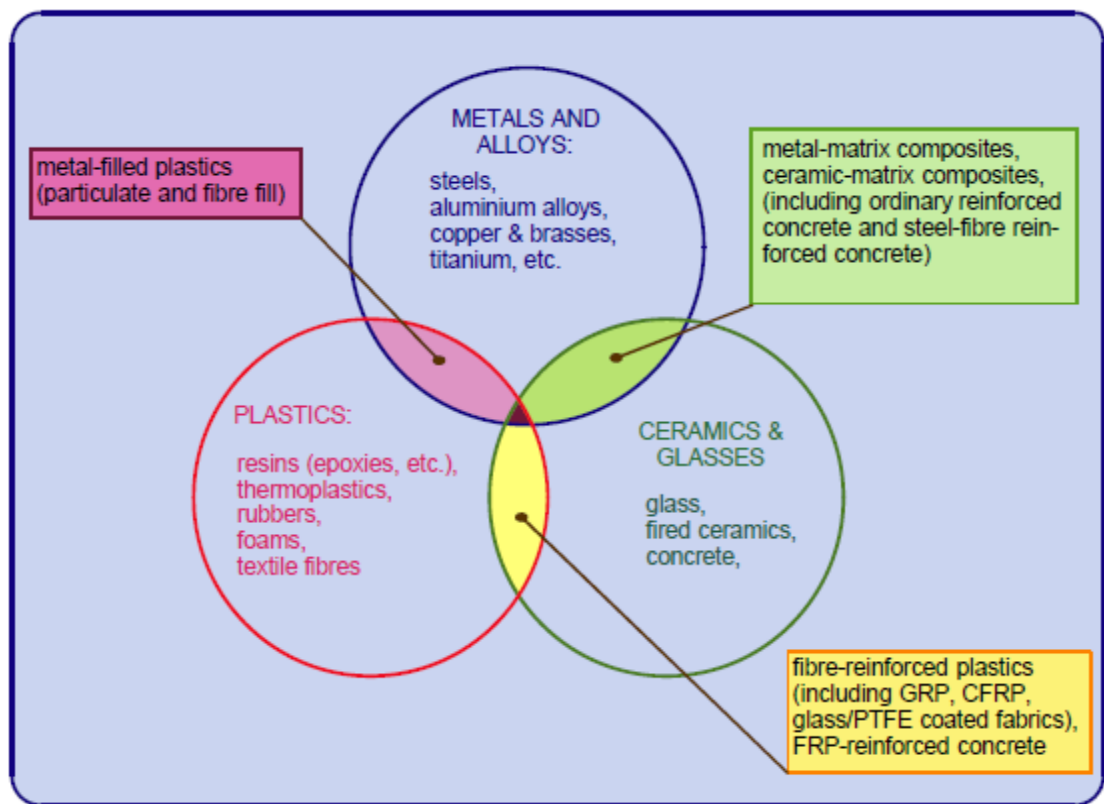


Figure 1 Relationship between Classes of engineering materials, showing evolution of composites [2]

1.2 Constituents of a Composite

1.2.1 Matrix

- Holds the reinforcement and distributes the load in the composite
- Forms a continuous phase throughout the composite

1.2.2 Reinforcement

- Bears the load, and provides strength to composite

- Usually embedded in the matrix

1.3 Main Types of Composites [4]

On the basis of matrix materials, composites can be categorized into three major groups:

1.3.1 Polymer Matrix Composites (PMC's) – Also known Fiber Reinforced Polymers (FRP) – matrix in these materials is constituted of polymer-based resins and reinforcement is constituted of fibers such as glass, carbon and aramid.

1.3.2 Metal Matrix Composites (MMC's) - Matrix in these materials is constituted of metals such as aluminum, and reinforcement is constituted of fibers/particles, e.g. silicon carbide. Their major use is in automotive industry.

1.3.3 Ceramic Matrix Composites (CMC's) - Matrix in these materials is constituted of ceramics and reinforcement is constituted of short fibers or Silicon carbide and boron nitride whiskers. Their major applications is at high temperature environments.

1.4 Fiber reinforced Polymer Matrix Composites (FRP)

Fiber reinforced polymer matrix hold major share in composite materials used worldwide and includes reinforcing fibers embedded in polymer matrix. Fiber included glass, carbon, aramid, or a hybrid fiber (composed of two different fiber materials). Epoxy, phenolic, vinylester or polyester are generally employed matrix. FRPs find major applications in the automotive, aerospace, marine and construction industries etc.

1.4.1 Fiber and Its Types

Fibers have small diameter and can be polycrystalline or amorphous in nature. From structural viewpoint, fibers can be polymers such as aramids, glass, carbon or ceramics such as aluminum oxide, silicon carbide etc.



Figure 2 Fiber Glass



Figure 3 Carbon fiber



Figure 4 Aramid Fiber

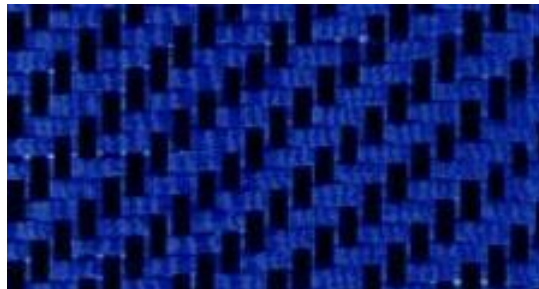


Figure 5 Kevlar/Carbon Hybrid

1.4.2 Advantages of FRP

They have many obvious advantages over traditional composites as:

- a. Low thermal conductivity
- b. Low density
- c. Excellent corrosion and chemical resistance
- d. High strength/weight ratio
- e. Design flexibility

- f. Cost-effective production of complex 3D structures
- g. Excellent fatigue and impact properties
- h. Radar/Sonar transparency
- i. Low maintenance

1.4.3 Properties of Fiber reinforced Polymer Matrix Composites

Since fiber reinforced PMC's combine resin as a matrix and fibers as reinforcement, therefore properties of composite material so formed will have combine properties of the resin and that of the fiber with some effect of individual properties. [4]

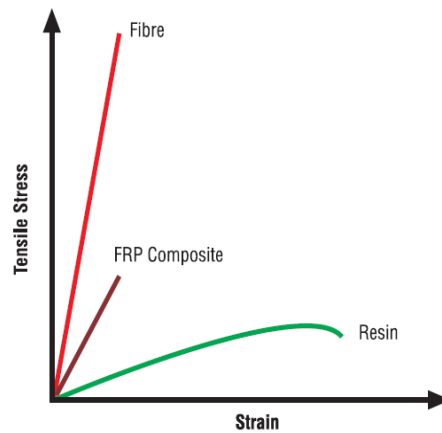


Figure 6 Stress - Strain Curve showing behavior of Fiber, Resin and FRP Composite Sample [4]

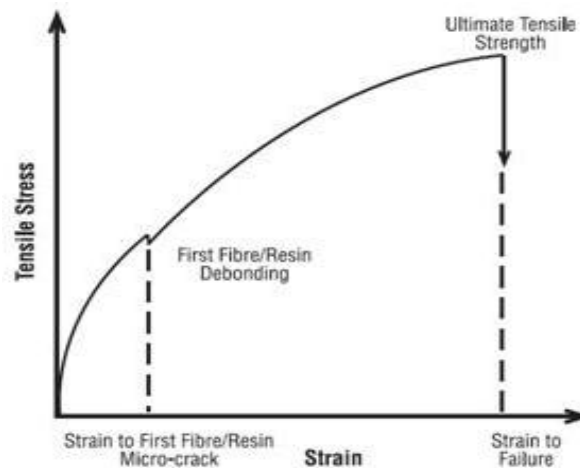


Figure 7 Curve Showing Behavior of Typical FRP sample during Axial Loading under Stress [4]

1.5 Carbon Fiber Phenolic Composites

In these composites high strength carbon fibers act as reinforcement embedded in phenolic matrix.

1.6 Carbon fiber

Carbon fibers are composed mainly of carbon atoms and have diameter in range of 5–10 μm .



Figure 8 Carbon Fiber Roving

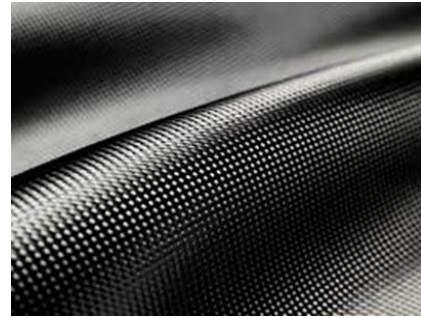


Figure 9 Carbon Cloth

1.6.1 Properties of Carbon Fibers [5] [19]

- a. High stiffness and tensile strength
- b. Low weight
- c. High Strength/weight ratio
- d. Rigidity
- e. High chemical resistance
- f. High temperature tolerance
- g. Corrosion resistance
- h. Electrical Conductivity
- i. Fire Resistance
- j. Low coefficient of thermal expansion
- k. Non poisonous

- l. Biologically inert
- m. Self Lubricating

Table 1 Properties of Carbon Fiber [20]

S No	Property	Unit	Value
1	Tensile Strength	MPa	2600 ~ 3600
2	Young's Modulus	GPa	200 ~ 400
3	Elongation at failure	%	0.6 ~ 1.5
4	Density	g/cm ³	1.7 ~ 1.9
5	Coefficient of Thermal Expansion	10 ⁻⁶ K ⁻¹	Axial -0.1 ~ -1.3 Radial 18
6	Fiber Diameter	nm	6 ~ 7
7	Fiber Structure	-	Anisotropic

1.6.2 Manufacturing of Carbon fiber

Carbon fiber is manufactured by process involving controlled oxidation, carbonization and graphitization of carbon-rich precursor such as polyacrylonitrile (PAN), pitch or cellulose. However, PAN fiber gives the best carbon fiber properties. Variation of the graphitization process produces fibers having high strength (temp ~ 2,600°C) or high modulus (temp ~ 3,000°C). After manufacturing, carbon fiber receive a surface treatment and chemical sizing for improvement in matrix bonding at composite formation stage and protection during handling.[13]

The process of producing carbon fibers is both chemical and mechanical in nature. Manufacturing sequence is elaborated as under. [1][17]

- 1.6.2.1.1 Spinning - Precursors such as PAN or Pitch are spun into fibers by mixing with chemicals and forced into a chemical bath or quench chamber through tiny jets. Here the mix solidifies in to the resulting fibers. Other methods heat the mixture while pumping it into a chamber through tiny jets where the solvents evaporate. The remaining material is the fiber. The internal atomic structure of the fiber is determined by the spinning process. It is this very reason that much research has been done in this step alone. Fibers are given washing treatment and are straightened afterwards to required diameter. This straightening aligns of the molecules of fiber, creating tightly bonded carbon crystals when carbonized.
- 1.6.2.1.2 Stabilizing - Inherently the resulting fibers are thermally unstable. it was because initial fibers decomposed during heat treatments that initial research was halted. The fibers must be chemically treated to thermally stabilize the atomic bonding. The solution is to heat them at about 200-350°C for several minutes. This allows oxygen to diffuse into the atomic structure increasing the strength of the bonds and its thermal stability.
- 1.6.2.3 Carbonizing - The next step is to heat the fibers between 1,000 to 3,000°C for several minutes. In order to prevent decomposition an inert gas is used. This removes any non-carbon atoms from the atomic structure such as water vapor, carbon monoxide, carbon dioxide, and hydrogen. The resulting fiber will have tightly bonded crystalline structures with better alignment perpendicular to the fiber axis.
- 1.6.2.4 Treating the surface - The drawback with having tightly bonded crystal structures is that they are inherently inert themselves. Of specific nuisance is the inert surface of carbonized fibers. This is bad when trying to combine fibers and matrix materials. The

surface must be slightly oxidized to provide better bonding characteristics. These processes also etch and roughen the surface giving more surface to area for bonding. Ideal gases for oxidizing are air, carbon dioxide, and ozone. Ideal liquids include sodium hypochlorite or nitric acid. Electrolysis can also be used by coating the fibers when treating them as a positive terminal in a bath of electrically conductive materials. Warning must be given to producing surface defects which could be sources of failure.

1.6.2.5 Sizing - Now that the fibers have been surface treated, they must undergo a process called sizing where fibers are applied with a coat for protection against damage during winding or weaving. Epoxy, polyester, nylon, and urethane are examples of commonly used coating materials.

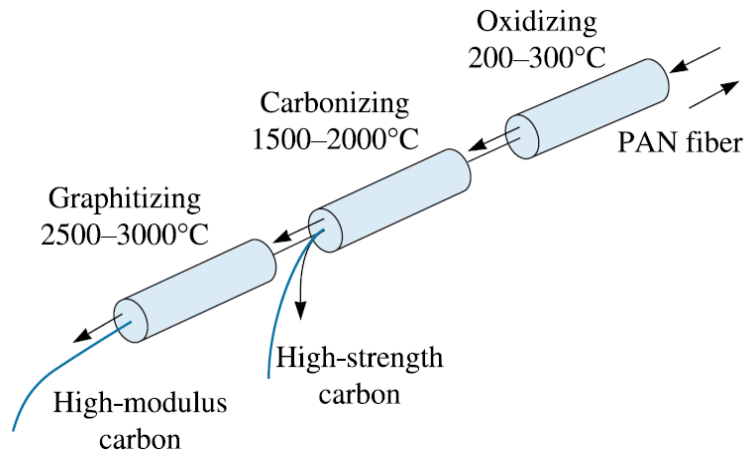


Figure 8 Schematic of Processing Steps for Producing Carbon Fibers [13]

1.6.3 Applications of Carbon Fiber

Table 2 Applications of Carbon Fibers [18]

S No	Types	Specifications	Major Usage
1	Filament	Filament is ayarn is made up of abundant twisted, untwisted, twisted-and-untwisted fibers	Used in resin reinforcement for Fiber reinforced composites, having application in aerospace, marine, sports and construction industry
2	Tow	Tow is untwisted bundle of yarn consisting of numerous fibers	
3	Staple Yarn	Staple yarn is made by spinning of staples	Heat Insulation, Abrasive, Carbon-Carbon composite parts
4	Woven fabric	It is a sheet made by weaving filament or staple yarn	Used in resin reinforcement for fabric reinforced composites, having application in aerospace, marine, sports and construction industry
5	Braid	Braid is made of filament or tow	Usually used in manufacture of tubular products
6	Chopped fiber	A chopped fiber made by chopping fibers to desired lengths	Employed with resins or cement for improvement in mechanical properties, abrasion behavior, heat resistance and electric conductivity.
7	Milled	Milled is a powder made by milling fiber in a ball-mill etc.	
8	Felt.Mat	A felt or mat made by layering up of staple by carding etc. then needle-punched or strengthened by organic binders	Heat insulator, protective layer ,corrosion-resisting filter
9	Paper	A paper made from staple by dry or wet paper-making	Anti-static sheet, electrodes, and heating plate etc
10	Prepreg	“Intermediate material” in a form of half-hardened sheets made of Carbon/silica fibers impregnated with thermo-	Aerospace parts, sport accessories and industrial parts where light weight and high performances are required

		setting resin.	
11	Compounds	Made of mixture of thermo-plastics or thermo-setting resins with addition of chopped fibers for feeding in to injection molding	Finds application where electric conductivity, rigidity and lightness in weight is required

1.7 Phenolic or Phenol formaldehyde resins

Phenolic resins are produced by condensation reaction of phenol and formaldehyde under appropriate conditions which leads to a cross-linked polymer structure of phenol-formaldehyde.



Figure 9 Phenolic Resin Sample

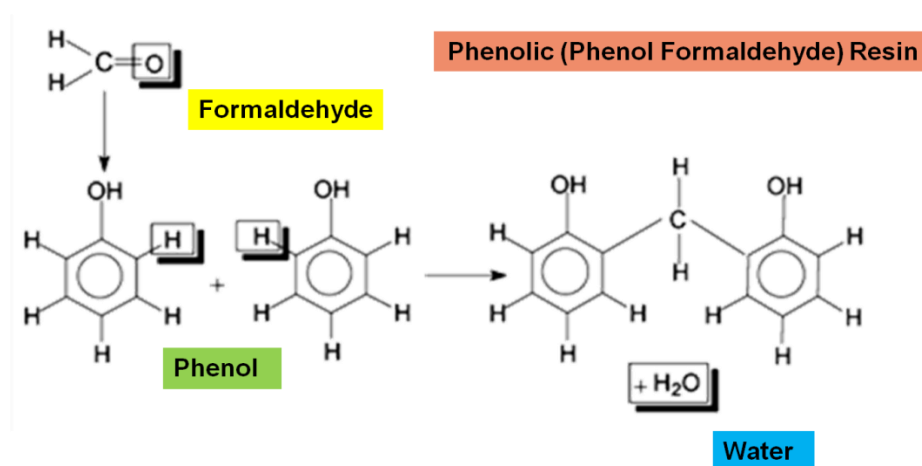
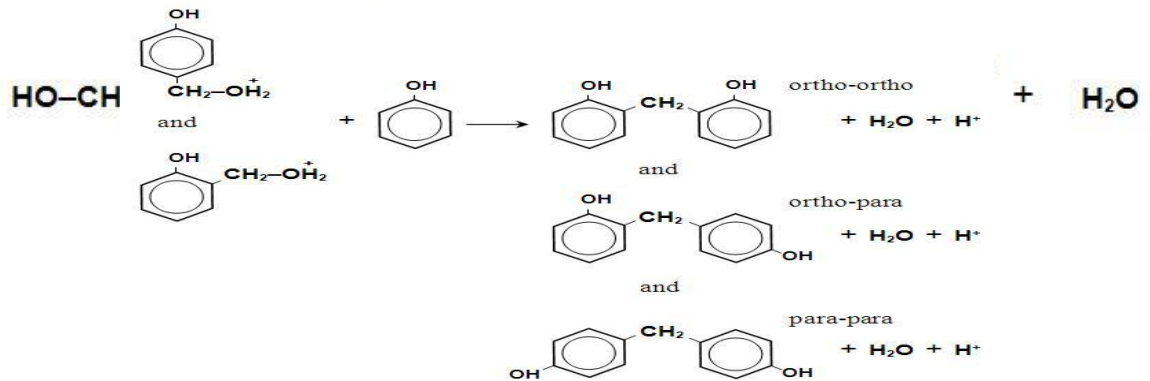


Figure 10 Condensation Reaction for Formation of Phenolic Resin

1.7.1 Types of Phenolic Resins [8]

Phenol and formaldehyde react to form two general resin types i.e Novolac Resin and Resole Resin.

1.7.1.1 Novolac Resins - Novolac resins are produced using acidic catalyst and a molar excess of phenol to formaldehyde. Reaction starts between methylene glycol and phenol and continues with addition of phenol, and formation of water.

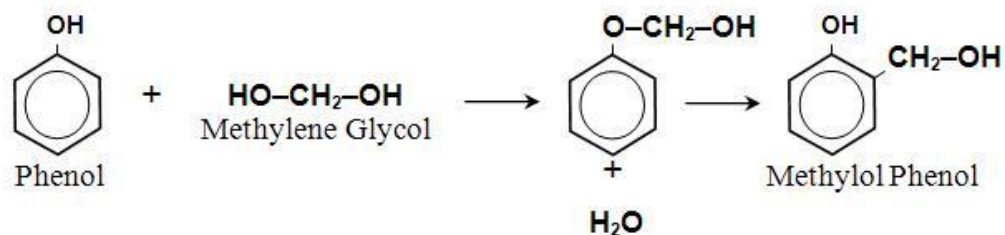


The reaction stops unless a cross-linking agent hexamethylenetetramine HMTA is added to complete polymerization. Therefore novolacs are called "two-stage" products.

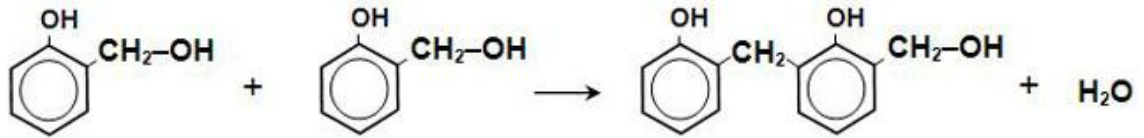
1.7.1.1.1 Characteristics of Novolac Resins

They are amorphous thermoplastics, solid at room temperature and soften and flow between 65°C - 105°C. Average molecular weight is between 250 and 900. They are insoluble in water and soluble in polar organic solvents such as alcohols, acetone.

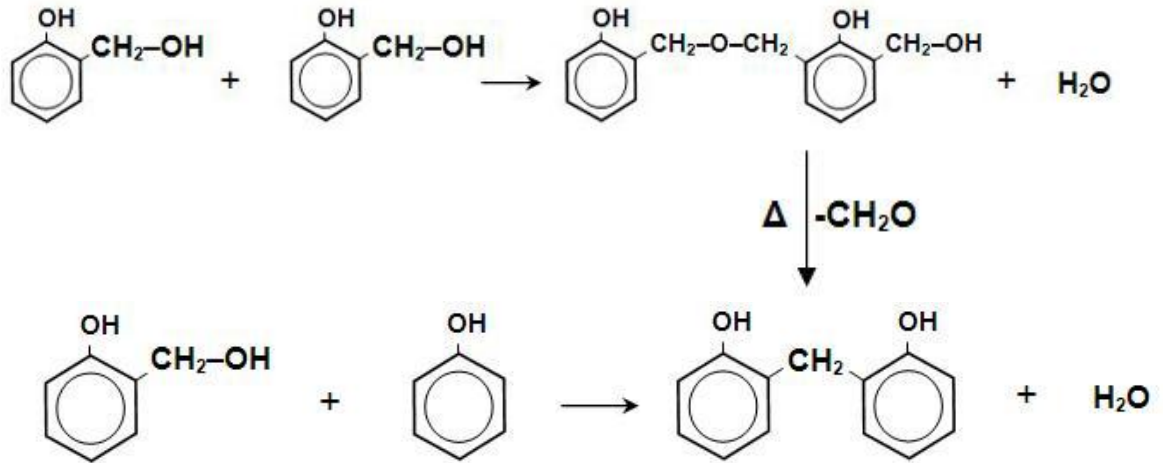
1.7.1.2 Resol Resins - Resole resins are produced using basic catalyst and a molar excess of formaldehyde. Initially phenol reacts with methylene glycol to form methylol phenol:



Then Methylol phenol either react with itself to form a longer chain molecule methylol-phenolic:



or form dibenzyl ether: or react with phenol to form a methylene-bridge.



When an excess of formaldehyde is used in resole manufacture, sufficient number of methylol and dibenzyl ether groups remain reactive to complete the polymerization and cure the resin without addition of a curing agent. Therefore, resoles are known as "single-stage" products.

1.7.1.2.1 Characteristics of Resole Resin

Average molecular weight resole resin is between 200 and 450. They are liquids having viscosity in range 50~50,000 cps, solids granules, or powders. Resole resins are partially soluble in water.

1.7.2 Properties of Phenolic Resin

Table 3 Properties of Phenolic Resin

S No	Property	Unit	Value	Ref
i	Density	g/cm^3	1.098	
ii	Tensile Strength	MPa	11.42	[16]
iii	Tensile Modulus	GPa	3.0 - 3.8	[26]
iv	Flexural Strength	MPa	80 - 115	[26]
v	Flexural Modulus	GPa	3.0- 3.5	[26]
vi	Impact Strength	J/mm^2	1.9	[16]

1.7.3 Characteristics & Applications of Phenolic Resins [6] [8] [10]

1.7.3.1 Performance at High Temperature

Phenolic resins have the ability to withstand high temperature under mechanical stress with minimal deformation. They provide required rigidity to maintain structural integrity and dimensional stability under intense conditions. This feature finds application of phenolic resins in refractory, friction, foundry and aerospace products. Parts produced includes valves, automotive brake pistons, pulleys, and water pump housings and seals.

1.7.3.2 Bonding Ability

The most important utilization of phenolic resin is as bonding agent for the reason that it easily penetrates and adheres to the structure of reinforcements by wetting and cross-linking throughout composite. This property provides the ability to manufacture components with desired mechanical, thermal, and chemically resistant properties. Applications of phenolic resins include refractory products, abrasive grinding wheels and friction linings. It is also used in particleboard, plywood, hardboard, melamine laminates. Composites for unique applications such as on oil platforms, missile components, and heat

shields are produced using phenolic resins employing various processes like pultrusion, filament winding or transfer molding.

1.7.3.3 Low Smoke and Toxicity [6]

Burning of phenolic resin produces hydrogen, hydrocarbons, water vapor, and carbon dioxide. Phenolic resin have low toxicity and produce very less smoke. This ability enables phenolic resin to be used in air support systems for the mining industry and supports for electrical channels.

1.7.3.4 Resistance to Chemical Environment

Phenolic resins can sustain exposure to chemical environments. Such as gasoline, alcohol, oil, glycol, brake fluid, various hydrocarbons, and also weak acids and bases. Protective linings inside chemical transfer tankers are composed of phenolic resins.

1.7.3.5 High Carbon and Char Yield

Phenolic resins have higher char yields when exposed at high temperatures (300 - 1,000°C) in an inert atmosphere. They transform to a structural carbon known as “vitreous carbon”, a material similar to ceramic. This property enables phenolic resin to be used in electrodes, rare earth metal melting crucibles, nozzles for rocket, high temperature bearings, missile heat shields automotive brake pads/linings, and clutch facings.

1.7.3.6 Ablation

Phenolic resin chars when heated to temperatures >500°C and converts to amorphous carbon. This property gives ablative phenomena to a surface acting as heat shield produced to wear away at a controlled rate fashion at high temperatures. Examples includes rocket blast shields , nozzles and reentry shields for space shuttles

1.7.3.7 Adhesives

Due to special “affinity” for wood, phenolic resin are also used in wood bonding applications such as particleboard.

1.7.3.8 Bonded Abrasives

Phenolic resins are used for manufacturing bonded abrasives for grinding wheels as matrix binder.

1.7.3.9 Coatings

Phenolic resins have good chemical resistance therefore are coated on inside of chemical storage tanks and heat transfer equipment

1.7.3.10 Binder for Carbon

Due to good binding ability with carbon/graphite, phenolic resins are used in manufacture carbon-carbon composites, washers and carbon brushes for electrical motors.

1.7.4 Disadvantages of Phenolic Resin

Phenolic resins find many applications, however have certain drawbacks as formation of voids due to condensation reaction. Moreover they are very brittle and have low mechanical properties. [25]

1.8 Processing Techniques of Carbon Fiber Phenolic Composites

There are two major manufacturing techniques for Carbon Fiber phenolic Composites

1.8.1 Tape/Filament Winding Process

This technique is employed to produce conical and cylindrical products such as pressure vessels, storage tanks, pipe, and rocket motor cases & nozzles.

In this process pre impregnated tapes or fibers are wound on a mandrel rotating in a programmed pattern. After winding, composite is cured and the mandrel is removed.

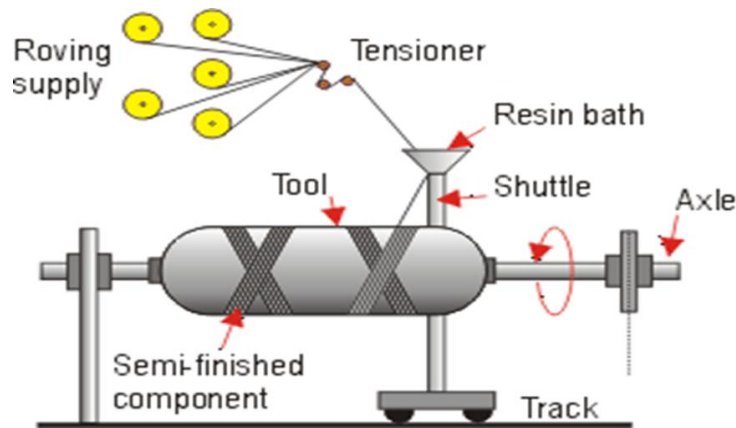


Figure 11 Schematic of Filament Winding

1.8.2 Compression Molding Process

In this technique metallic dies are mounted in a mechanical or hydraulic press. The premixed material is fed in the die and heat and pressure is applied. Curing time and cycle time depends on part geometry.

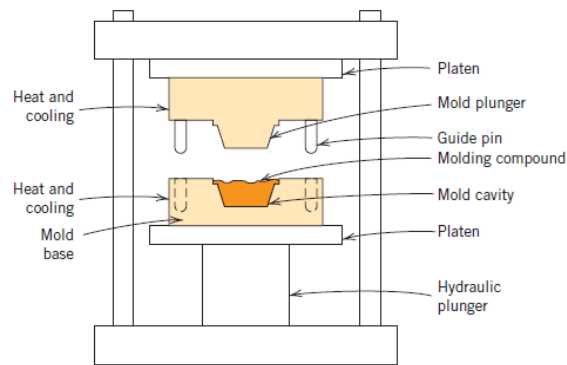


Figure 12 Compression Molding Apparatus [3]

1.9 Applications of Carbon Fiber Phenolic Composites

1.9.1 Space Shuttles/ Rockets

Carbon Phenolic composites are ideal for manufacturing rocket nozzles, reentry bodies and certain other high temperature aircraft applications due to excellent ablative properties and chemical resistance [9][14].



Figure 13 Nozzle of Space Shuttle

1.9.2 Interior of Commercial Airplanes

Due to low generation of smoke and toxic gases [6], carbon phenolic composites are employed in manufacture of aircraft interior parts.



Figure 16 Interior of Airplane

1.9.3 Exterior of Air Craft/Oil and Gas Applications

Carbon phenolic composites with higher carbon fiber content are used in manufacture of parts for exterior structure of aircraft as well as oil & gas applications.

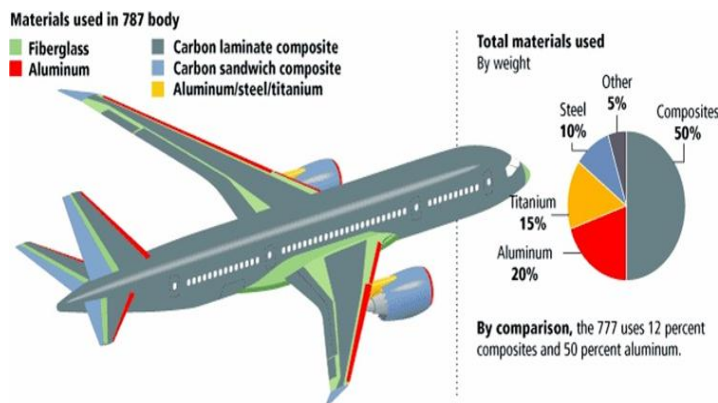


Figure 14 Carbon Composite Application on Aircraft Exteriors

1.9.4 Automobile Brake Pads

Temperature as well as strength imparted by carbon fibers makes them an appropriate choice for modern automobile brake pads. The brake pad material is manufactured using carbon fiber as reinforcement with phenolic resin as matrix having metallic powders such as barium sulphate [12]



Figure 15 Brake Pads



Figure 16 Carbon Phenolic Brake Pads in Modern Cars

1.9.5 Composite Spherical Bearings

Due to thermal stability and self-lubricating property, carbon phenolic composites are used in manufacturing of bearings of elevation driving mechanism of battle tanks. [11]

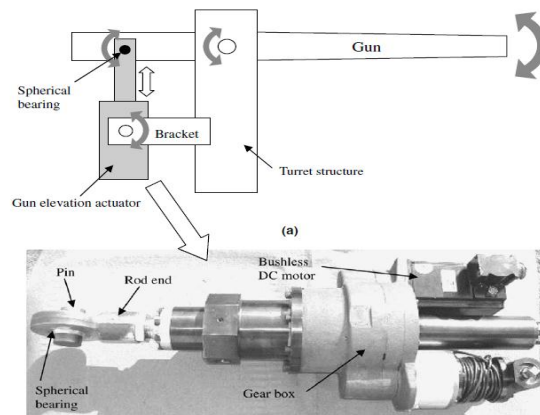


Figure 17 Carbon Phenolic Bearing

2. Literature Review

In present research work, the mixing ratio effect was studied to find out the mixing ratio providing optimum strength combined with desirable ablative properties for a specific aerospace application at 3000 °C. Composite materials with ablative properties are ideal choice at high temperatures. Controlled ablation rate is necessary for optimum performance at high temperatures.

2.1 History of Work

Properties of Phenolic composites are improved to much greater extent by addition of various coupling agents as described by M.H. Choi et al in their research [22] and Z. J. Wang et al in study [23]. Mechanical properties of carbon phenolic composites may also be improved by hardener addition in phenolic resin up to 15% as worked out by S. Sulaiman et al.[31]

VinetaSrebrenkoska et al studied the mechanical and thermal properties with different fiber lengths and content. Optimum results were obtained at approx 60% carbon fiber content. [14]

Thermal and mechanical characteristics of carbon composites were studied by N. Winya et al and found that use of carbon phenolic composite can safely be employed as thermal insulation in rocket nozzle. [9]

H. Hatta and K. Goto described composites with a phenolic matrix as having an inherent risk of trapping gases expelled by the solvents during the curing process [21].

Erosion Rate of Carbon phenolic composites was studied using modeling and experimental Approach by Ahmadi et al in which they introduced a model to elaborate the erosion rate of carbon fiber composites exposed high velocity flame. In this study mathematical equation was derived for calculation of erosion rate [24]

Increase in modulus and flexural strength of the carbon fiber phenolic composites was observed by S.S Tzeng et al by addition of Carbon nano tubes (Carbon nano fillers) in phenolic matrix.[32]

3. Experimental Work

3.1 Raw Materials Information

For analyzing effect of mixing ratio on physical & mechanical properties of carbon phenolic composites, chopped carbon fibers and phenolic resin were used as raw materials for preparation of composite samples.

3.1.1 Properties of Chopped Carbon Fibers

Carbon Fibers T300 (Kollan-Australia) were used as reinforcement.

Important parameters measured are enlisted below in Table 4:

Table 4 Properties of Chopped Carbon Fibers

S No	Parameter	Index
a	Density	1.69 g/cm ³
b	No of monofilaments in Tow	3000
c	Chopped Tow Length	25 ~ 30 mm
d	Monofilament Diameter	6μm

3.1.2 Phenolic Resin

Phenolic Resin with following properties as described in Table 5 was used :

Table 5 Properties of Phenolic Resin

S No	Parameter	Index
a	Appearance	Reddish Brown Transparent Liquid
b	Solid Contents	63.98%
c	Gel Time	74 sec
d	Free Phenol Contents	0.11%

e	Density	1.098 g/cm ³
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3.2 Sample Identification

In order to find out an appropriate mixing ratio of carbon fiber and phenolic resin giving best combination for optimum strength and ablation rate, following 04 x samples as shown in Table 6 with increasing carbon fiber wt% were analyzed:

Table 6 Sample Identification

Sample ID	Fiber wt%	Mixing Ratio	Wt of Carbon Fiber (kg)	Wt of Phenolic Resin (kg)
Sample # 01	40 wt % Fiber	40% Carbon Fiber 60% Phenolic Resin	2	3
Sample # 02	45 wt % Fiber	45% Carbon Fiber 55% Phenolic Resin	2.25	2.75
Sample # 03	50 wt % Fiber	50% Carbon Fiber 50% Phenolic Resin	2.5	2.5
Sample # 04	55 wt % Fiber	55% Carbon Fiber 45% Phenolic Resin	2.75	2.25

3.3 Characterization Techniques employed

Following techniques were employed for characterization of samples as mentioned in Table 7

Table 7 Characterization Techniques

S No	Test ID	Test Equipment	Testing Standard	Test Conditions
i	Optical Microscopy	Carl Zeiss Optical Microscope	-	-
ii	SEM Analysis	Jeol 6490 LV SEM	-	-
iii	Density	Mettler Toledo Density Kit	GB 1033	Room Temperature
iv	Tensile Test	Tensile Testing Machine	GB 1447-82	Room Temperature

		MTS Sintech 10GL		
v	Impact Test	Plastic Impact Testing Machine JX 29.6 J	GB 1043-79	Room Temperature
vi	Ablation Rate Test	Custom made Ablation tester		3000 °C
vii	DSC	DSC Q100 V8.2 Build 268	-	20 °C ~ 200 °C Inert Atmosphere
viii	TGA	TGA Qgo V6.2 Build 187	-	Room Temperature ~ 1000 °C

3.4 Sample Preparation Technique:

Compression molding technique was used to prepare composite samples. In this technique, charge (Premixed material) is fed into a metallic die and a heating and pressure cycle is applied to cure the sample. After cooling, cured sample is ejected by pushing ejector pin placed in the lower part of die. Schematic of process is shown in Fig 20. A metallic die of Dia 250 mm and height 15 mm was used to mold a round sample of each wt % carbon fiber as shown in Fig 22.

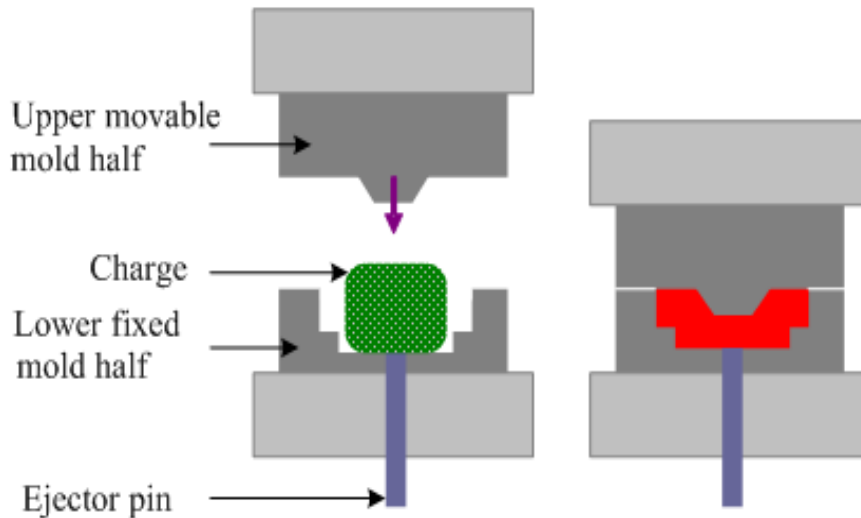


Figure 18 Schematic of Compression Molding

3.5 Sample Preparation Sequence

Following steps were involved in sample making as per requirements mentioned in Table 6

3.5.1 Premixing

Carbon and Phenolic Resin premix is required to be prepared. Mixing ratios as shown in Table 6 were adopted by weight proportions for preparation of premixed material for 04 x samples.

3.5.1.1 Premixing Sequence

Following sequence was followed for Premixing:

- i. Chopped carbon fibers of length 25 ~ 30 mm were weighed using electronic balance according to Qty mentioned in Table 6 for specific weight percent sample. .
- ii. Fibers were preheated at 100 °C for 20 minutes to remove moisture, dirt and grease.
- iii. Weighed Qty of Phenolic resin (according to weights mentioned in Table 6) was diluted with Ethyl Alcohol (50 ml) and put in mixer.
- iv. After cooling to room temperature, carbon fibers were impregnated in a mixer for a20 minutes with Phenolic resin.
- v. Afterwards pre impregnated material was loosen by hand and placed at room temperature for evaporation of volatile materials for 40 minutes.
- vi. Then this premixed material was preheated at 65 ~ 70 °C for 1 hr in hot air oven for precuring the resin before pouring the material into die to avoids its squeezing out when pressure is applied in hydraulic press.
- vii. After cooling to room temperature, material was packed in polythene bags and kept at temperature 25 ± 2 °C.

3.5.2 Compression Molding

- i. The die used for sample making required 1.5 kg material for making a round sample of dia 250 mm and thickness 15 mm, therefore 1.5 kg premixed material was weighed for first sample (45 Wt% Fiber).
- ii. Premixed material was preheated at 85 ± 5 °C for 10 ~ 15 min.

- iii. Meanwhile molding die was clamped on 315T Hydraulic Press and heaters were switched on. When the temperature of die rose to $75 \pm 5 \text{ }^\circ\text{C}$, molding die was filled with preheated premixed material (1.5 kg). and pressurized to 3 ~ 5 MPa
- iv. When the temperature of the mold rose to $85 \pm 5 \text{ }^\circ\text{C}$, it was pressurized to 14 ~ 15 MPa
- v. When the temperature rose to $115 \pm 5 \text{ }^\circ\text{C}$, temperature holding was performed for 1 hr
- vi. When the temperature rose to $175 \pm 5 \text{ }^\circ\text{C}$, temperature holding was performed for 1.5 hr and heating device was switched off.
- vii. When the temperature of the mold fell below $60 \text{ }^\circ\text{C}$ demolding was performed.
- viii. This molding process was repeated for molding Sample No 2 (45 Wt% Fiber), Sample No 3 (50 Wt% Fiber) and Sample No 4 (55 Wt% Fiber).

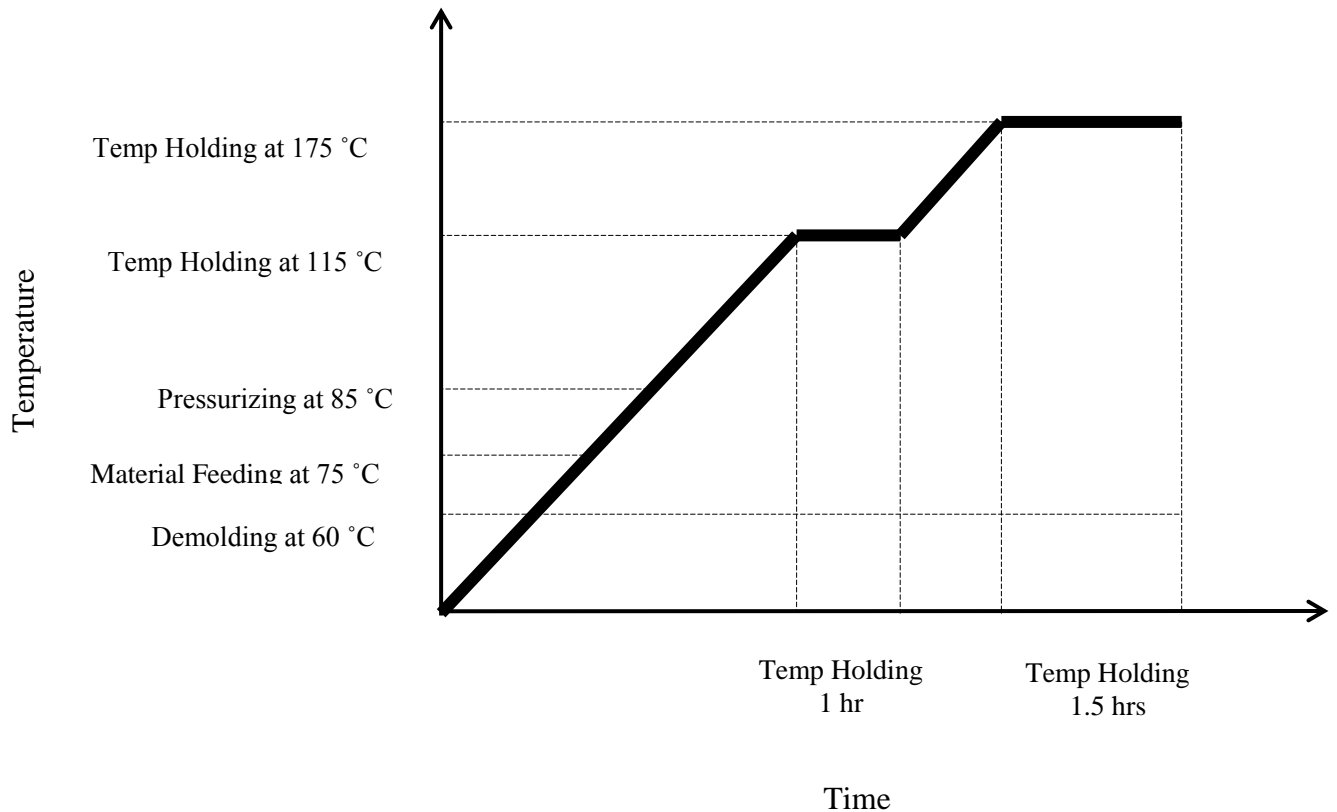


Figure 19 Schematic of Curing Cycle

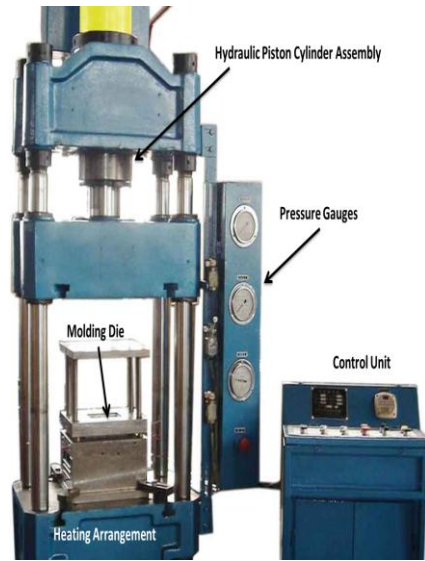
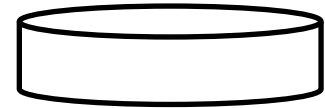


Figure 21 Hydraulic Press for Compression Molding



Sample dimensions

Figure 20 Molded Composite Sample

3.5.3 Machining

After molding machining was performed to acquire specimens for density, tensile test, impact test and ablation rate out of all four samples.

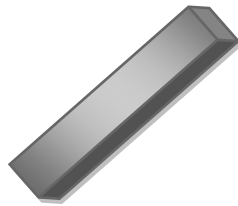


Figure 22 Impact Test Specimen



Figure 23 Tensile Test Specimen

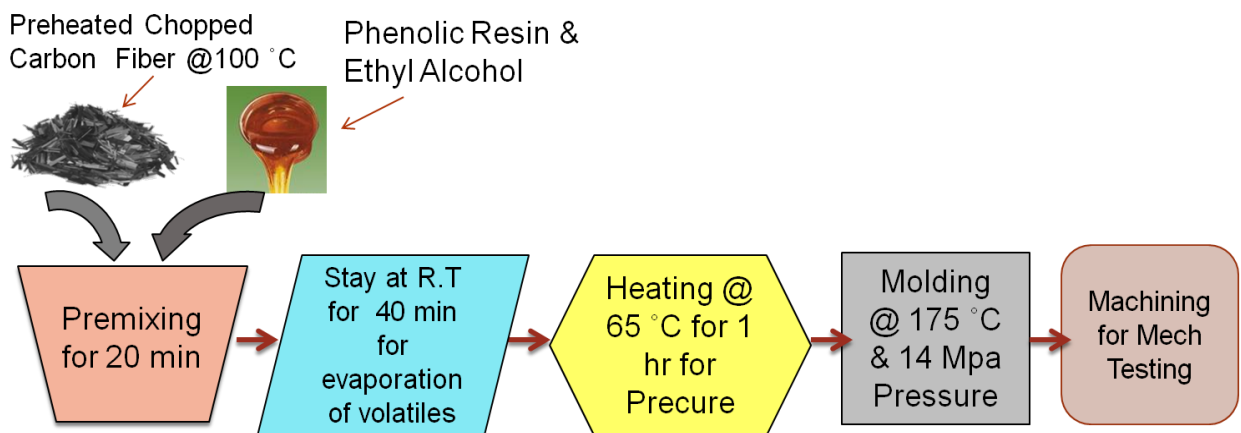


Figure 24 Schematic of Sample Preparation Sequence

4. Results & Discussion

In this chapter experimental results of 04 x samples after analyzing by characterization techniques mentioned in Table 7, will be discussed.

4.1 Study of Morphology

Before investigating mechanical properties, morphology was studied using optical and scanning electron microscope.

4.1.1 Optical Microscopy

Microstructure analysis was performed on Optical microscope to have an idea regarding phase distribution of reinforcement i.e. Carbon fibers and matrix i.e. phenolic resin on macro scale.

4.1.1.1 Sample Preparation

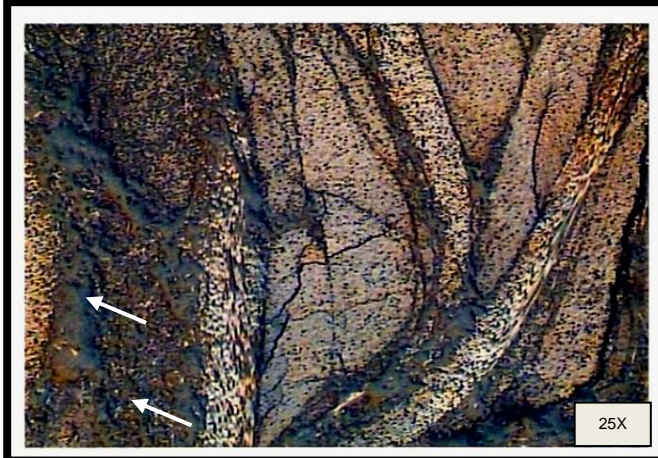
Specimens from all 04 x Samples as per Table 6 were cut using abrasive cutting wheel. Then these specimens were grinded on series of emery papers as # 320,600,800 &1200. Polishing was performed using 3 μ m diamond paste. Afterwards these specimens were observed under Carl Zeiss optical microscope.

4.1.1.2 Optical Microscope Observations

Optical microscope images and observations are summed up in Table 8

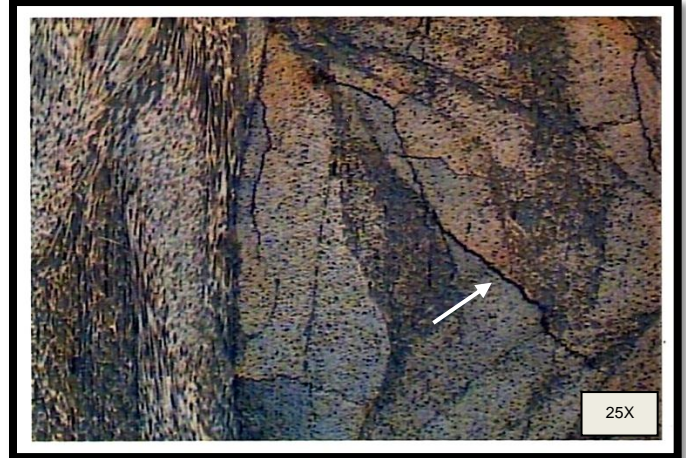
Table 8 Optical Microscope Observations

Sample No 01
40 wt% Fiber



Carbon fibers may be seen randomly orientated as seen in the micrograph. Qty of fibers is however less if compared from micrographs of other samples. Visible cracks and free resin patches are observed, showing resin concentration at various areas.

Sample No 02
45 wt% Fiber



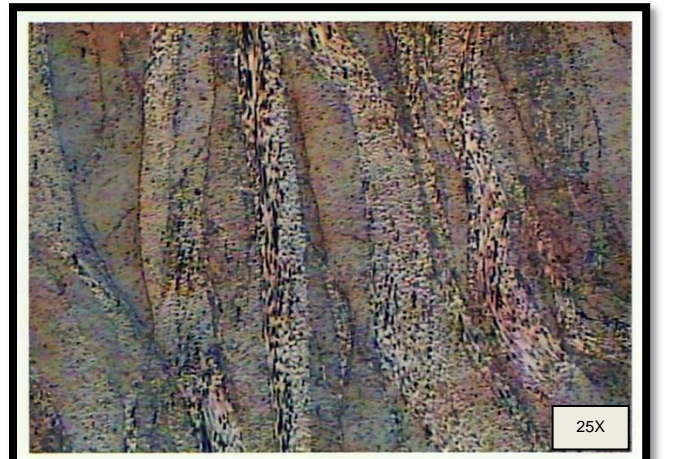
Qty of carbon fibers has increased, however large cracks are observed in resin concentration areas, showing brittle nature of phenolic matrix. It is obvious from micrograph that crack propagation path is deviated whenever it encounters a bunch of carbon fibers

Sample No 03
50 wt% Fiber



Few small cracks are observed in resin concentration areas showing that addition of fibers has limited the crack propagation throughout matrix. Qty of carbon fibers is increased if compared with Sample No 02.

Sample No 04
55 wt% Fiber



Even distribution of carbon fibers in phenolic resin matrix is observed. No resin patches and cracks are observed. Quantity of carbon fiber has an obvious increase as compared to rest of samples

4.1.2 SEM Analysis

For SEM analysis, samples were immersed in liquid nitrogen for 20 minutes and then fractured on impact testing machine to get neat and fresh surfaces for observation. Gold sputter coating was performed to make samples conductive. Observations are elaborated in Table 9 ~ 12.

Table 9 SEM Analysis 40 wt% Sample

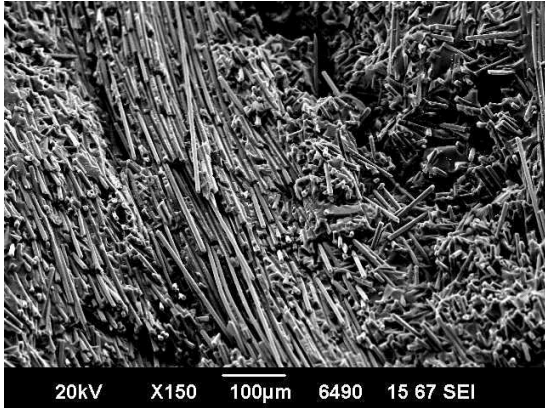
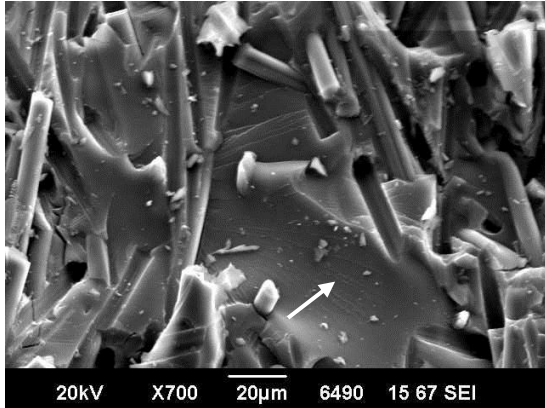
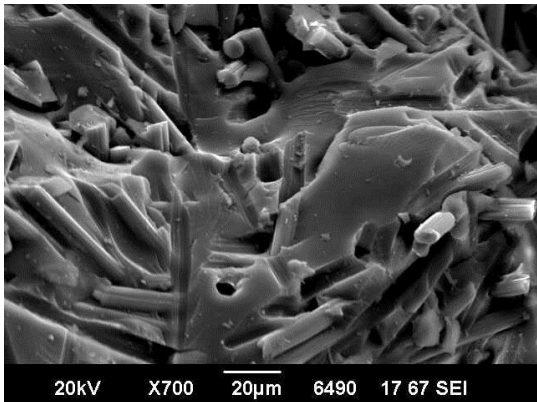
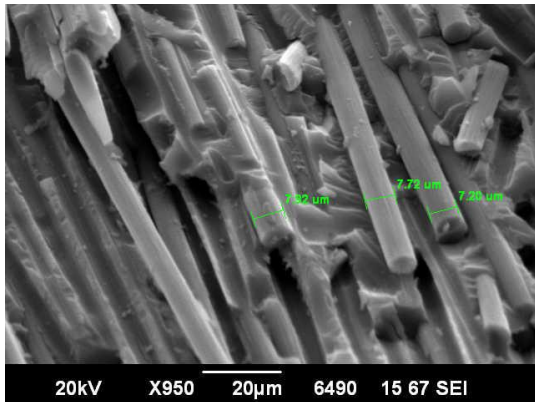
Sample No 01 40 wt% Fiber	
<p>Image # 01</p>  <p>20kV X150 100µm 6490 15 67 SEI</p> <p>Image showing random orientation of carbon Fibers in Phenolic Matrix</p>	<p>Image # 02</p>  <p>20kV X700 20µm 6490 15 67 SEI</p> <p>Resin concentration is obvious in sample. Fractured surface showing typical cleavage/river pattern pointing towards brittle behavior of phenolic matrix</p>
<p>Image # 03</p>  <p>20kV X700 20µm 6490 17 67 SEI</p> <p>Fiber pull out from matrix is observed, which may attributed to poor adhesion of Carbon fibers to phenolic matrix</p>	<p>Image # 04</p>  <p>20kV X950 20µm 6490 15 67 SEI</p> <p>Fiber diameter has been measured and found as 7µm.</p>

Table 10 SEM Analysis of 45 wt% Sample

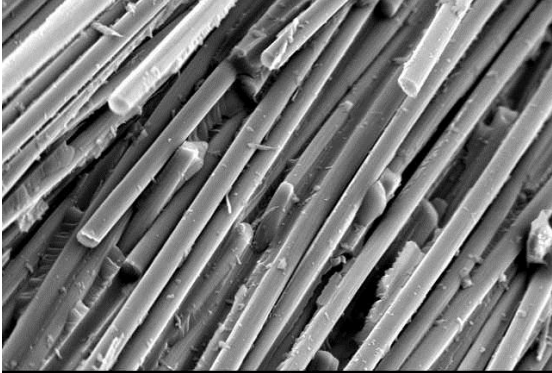
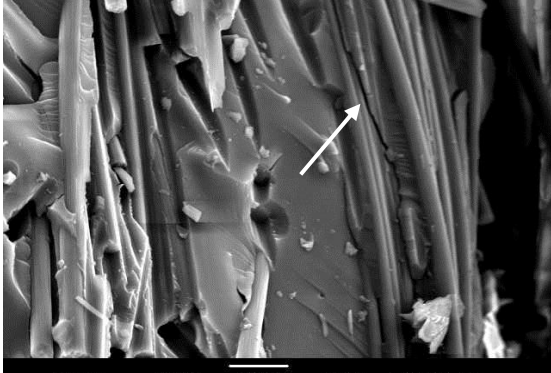
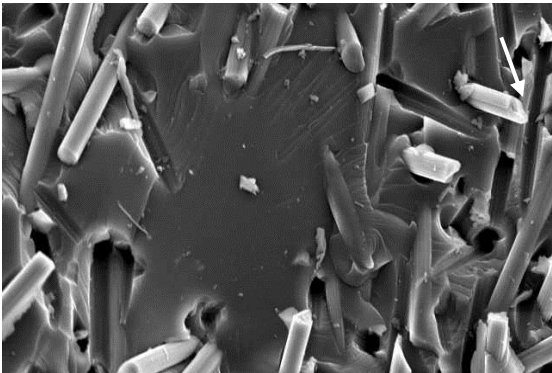
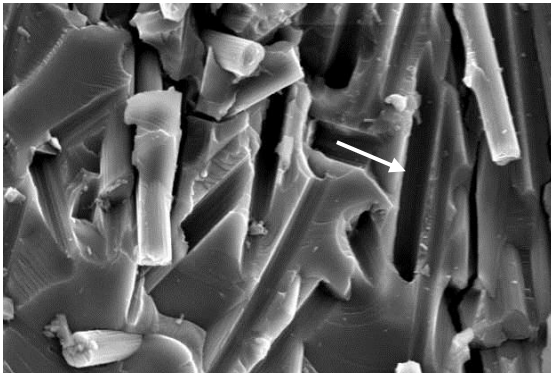
Sample No 2 45 wt% Fiber	
<p>Image # 01</p>  <p>20kV X700 20µm 6490 16 67 SEI</p> <p>Randomly orientated carbon fibers are observed, embedded in phenolic matrix</p>	<p>Image # 02</p>  <p>20kV X700 20µm 6490 16 67 SEI</p> <p>Crack is observed in resin matrix</p>
<p>Image # 03</p>  <p>20kV X700 20µm 6490 16 67 SEI</p> <p>Fracture of carbon fiber is observed along with brittle fracture of phenolic matrix</p>	<p>Image # 04</p>  <p>20kV X1,000 10µm 6490 16 67 SEI</p> <p>Fiber pull out from matrix and cleavage type fracture is observed in phenolic matrix</p>

Table 11 SEM Analysis of 50 wt% Sample

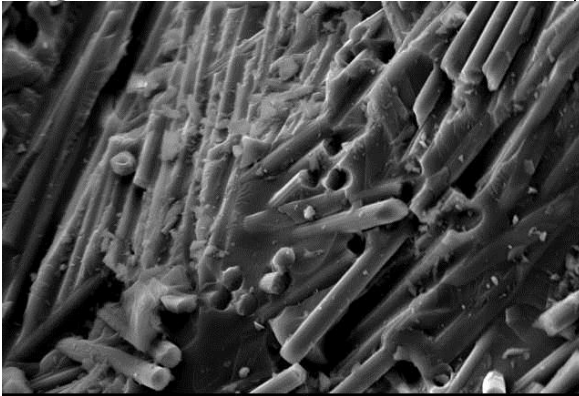
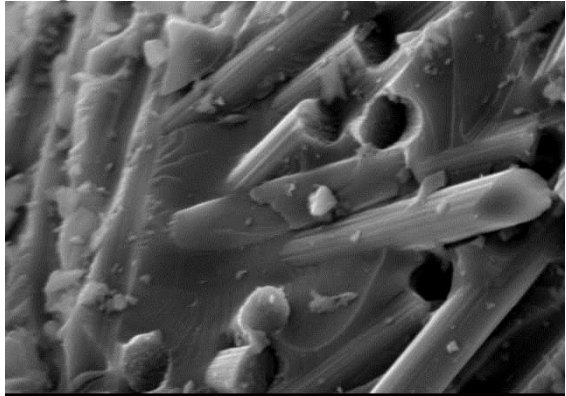
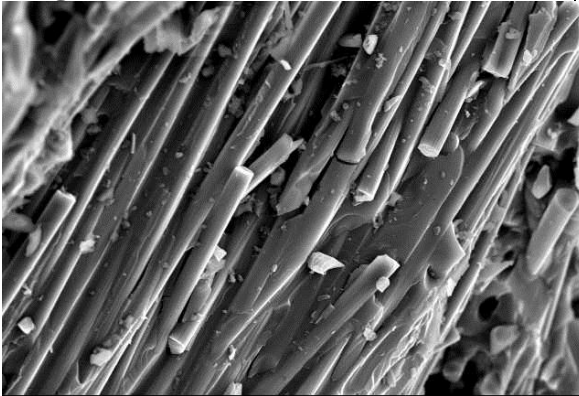
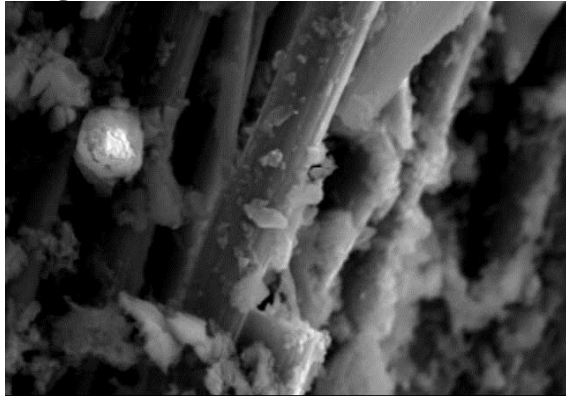
Sample No 3 50 wt% Fiber	
Image # 01	Image # 02
	
<p>Randomly oriented Carbon fibers observed. As fiber wt% is increasing, more fibers are observed at 700X. Fiber & resin brittle fracture is obvious.</p>	<p>Resin fragments observed on fractured surface of matrix.</p>

Table 12 SEM Analysis of 55 wt% Sample

Sample No 04 55 wt% Fiber	
Image # 01	Image # o2
	
<p>Fiber concentration is increased if compared with Image # 01 of Sample No 03 at same magnification i.e. 700X</p>	<p>Partial Phenolic resin adherence to surface of carbon fibers observed at 2500X.</p>

4.2 Density

4.2.1 Density of Pure materials

Density of Pure materials i.e. Carbon fibers and Phenolic resin was measured and is shown in Table 13

Table 13 Density Measurement of Pure samples

S No	Nomenclature	Unit	Value
i	Carbon Fibers	g/cm ³	1.69
ii	Phenolic Resin (Uncured Sample)		1.089
iii	Phenolic Resin (Cured Sample)		1.2

4.2.2 Density of Composite Samples

Density of 04 x Composite samples was measured and is shown is Table 14. For each mixing ratio, 06 x specimens were prepared and an average of these 06 specimens has been accounted for.

Table 14 Density Measurement of Composite Samples

Sample ID	Sample # 01	Sample # 02	Sample # 03	Sample # 04
Fiber wt%	40 wt % Fiber	45 wt % Fiber	50 wt % Fiber	55 wt % Fiber
Unit	g/cm ³	g/cm ³	g/cm ³	g/cm ³
Specimen # 01	1.44	1.46	1.52	1.51
Specimen # 02	1.46	1.46	1.52	1.48
Specimen # 03	1.46	1.47	1.53	1.55
Specimen # 04	1.44	1.47	1.5	1.54
Specimen # 05	1.47	1.47	1.49	1.55
Specimen # 06	1.45	1.47	1.51	1.54
Average	1.45	1.47	1.51	1.53

4.2.3 Result Deduction

It may be concluded from the experimental results in Table 14 that density of carbon phenolic composite is gradually increasing by Increasing Wt % of Carbon fibers from 1.45 g/cm³ to 1.53 g/cm³, and it almost in mid range of Pure materials i.e. Carbon Fibers (1.69 g/cm³) and Phenolic Resin (1.2 g/cm³), as expected from composite materials. In other words, the density of pure resin is increased 1.25 times by addition of carbon fibers, which is a depiction of improvement of mechanical properties [14]. Fig 27 showing the graphical representation of increase in density with increasing Fiber wt% in composite samples.

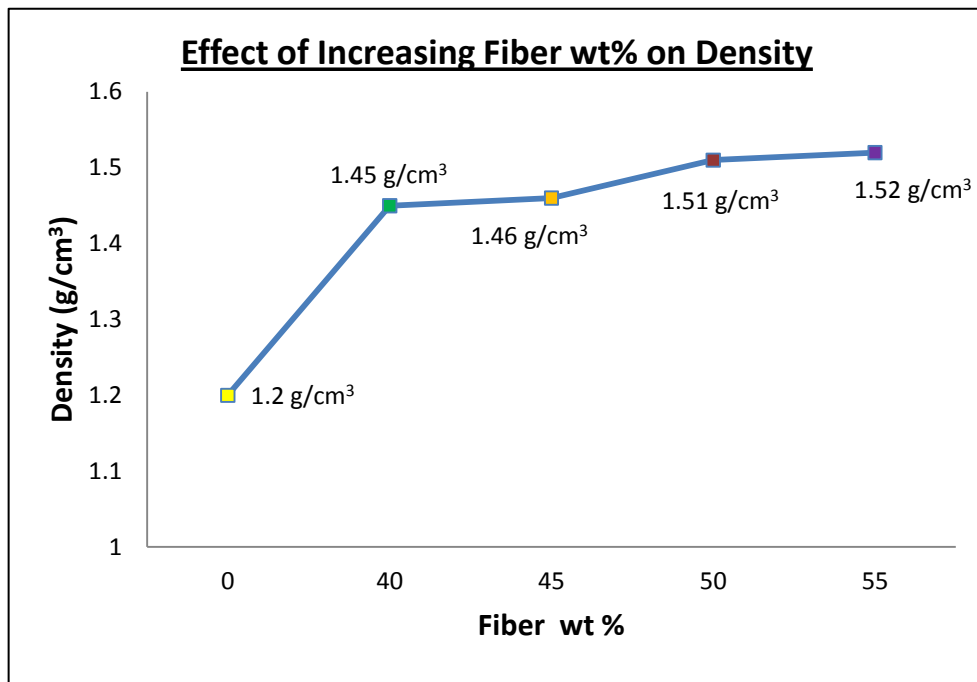


Figure 25 Effect of Increasing Fiber wt% on Density

4.3 Tensile Test

Tensile testing is an essential mechanical test determining load bearing capability of various materials such as metals, polymers, composites, and fabrics. During tensile test an axial load is applied to samples till fracture. This load induces mechanical deformation which in turn puts energy into a material. The energy is stored elastically

or dissipated plastically. The way a material stores this energy is summarized in stress-strain curves. Using data obtained from Stress Stain curve, tensile strength, elasticity modulus, elongation at break are evaluated.

4.3.1 Tensile Strength

Tensile strength of pure samples was measured at room temperature and is mentioned in Table 15

Table 15 Tensile Strength of Pure Materials

S No	Nomenclature	Unit	Value
i	Carbon Fiber	MPa	2600 ~ 3600 [20]
ii	Phenolic Resin (Cured Sample)		5.13

Tensile test of composite samples was performed at room temperature for 04 x samples with increasing wt% Fiber as mentioned above in Table 6. 06 x dumb bell shape specimens as shown in Fig 25 were machined out of each composite sample as shown in Fig 22. The values for Tensile strength, modulus of elasticity and elongation at break were measured for 06 x specimens from each sample and average value of 06 x specimens is accounted for as shown in Table 16

Table 16 Tensile Test of Composite Samples

Sample ID	Sample # 01			Sample # 02			Sample # 03			Sample # 04		
Fiber wt%	40 wt % Fiber			45 wt % Fiber			50 wt % Fiber			55 wt % Fiber		
Test ID	σ	ϵ	E	σ	ϵ	E	σ	ϵ	E	σ	ϵ	E
Unit	MP a	%	GPa	MP a	%	GPa	MP a	%	GPa	MP a	%	GPa
Specimen # 1	53	4.84	2.23	34	4.53	1.58	56	5.52	2.39	66	5.07	2.04
Specimen # 2	29	4.71	1.22	93	5.05	3.19	109	6.02	3.77	63	4.6	2.66
Specimen # 3	32	4.09	1.39	88	5.79	3.1	81	6.52	2.98	71	4.89	2.68
Specimen # 4	31	4.68	1.47	84	4.61	3.08	86	5.9	2.46	76	5.05	2.9

Specimen # 5	59	4.71	2.33	89	5.01	3.24	51	4.91	2.3	42	4.83	1.79
Specimen # 6	59	3.94	2.82	74	4.61	2.8	43	5.05	1.53	38	5.19	1.71
Average	44	4.49	1.91	77	4.94	2.82	71	5.65	2.57	59	4.94	2.29

σ : Tensile Strength , ϵ : Elongation , E : Modulus of Elasticity

The results of tensile test (Table 16) reveal that tensile strength of phenolic thermoset increases by addition of carbon fiber, i.e increased from 5.13 MPa (Pure resin sample) to 44 MPa (Sample No 01 40 wt% fiber addition). With further increase in fiber weight to 45% , it further increases to 77 MPa, which is about 06 times increase in the tensile strength of pure phenolic resin. By adding further carbon fibers upto 50% by weight, it decreased to 71 MPa, which is still higher than phenolic thermoset. Further increase in fiber weight percent to 55%, strength is decreased as shown in graphical representation Fig 28.

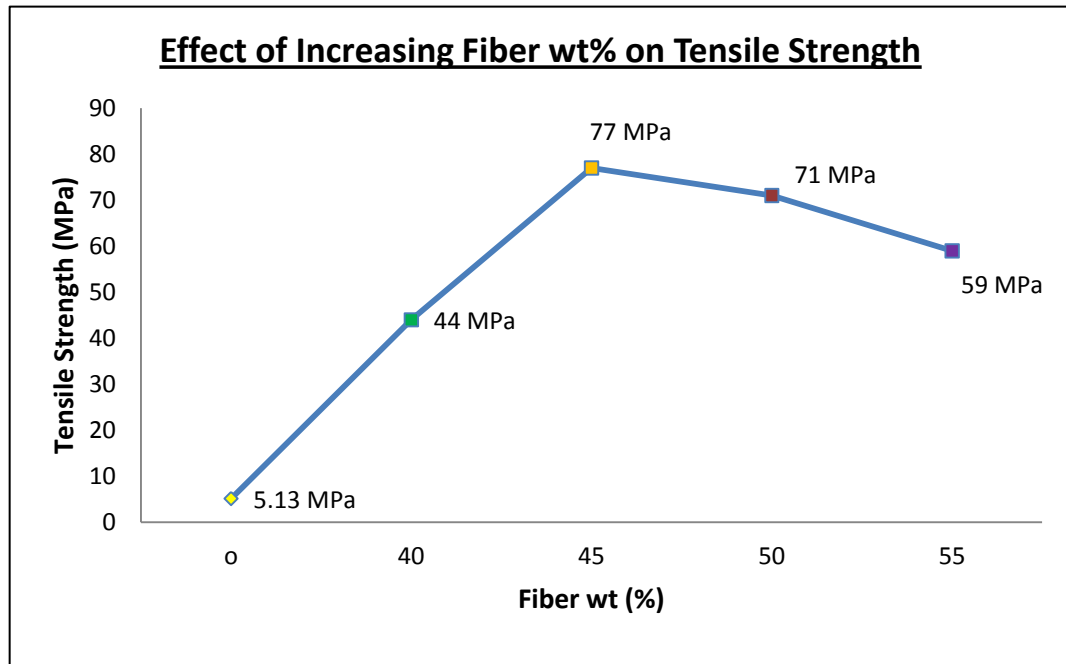


Figure 26 Effect of Increasing Fiber wt% on Tensile Strength

4.3.2 Stress-Strain Behaviour

Stress-Strain behaviour of pure phenolic and 04 x representative specimens of 04 x composite samples is elaborated in Fig 29 showing that maximum strength is obtained at 45 wt% fiber.

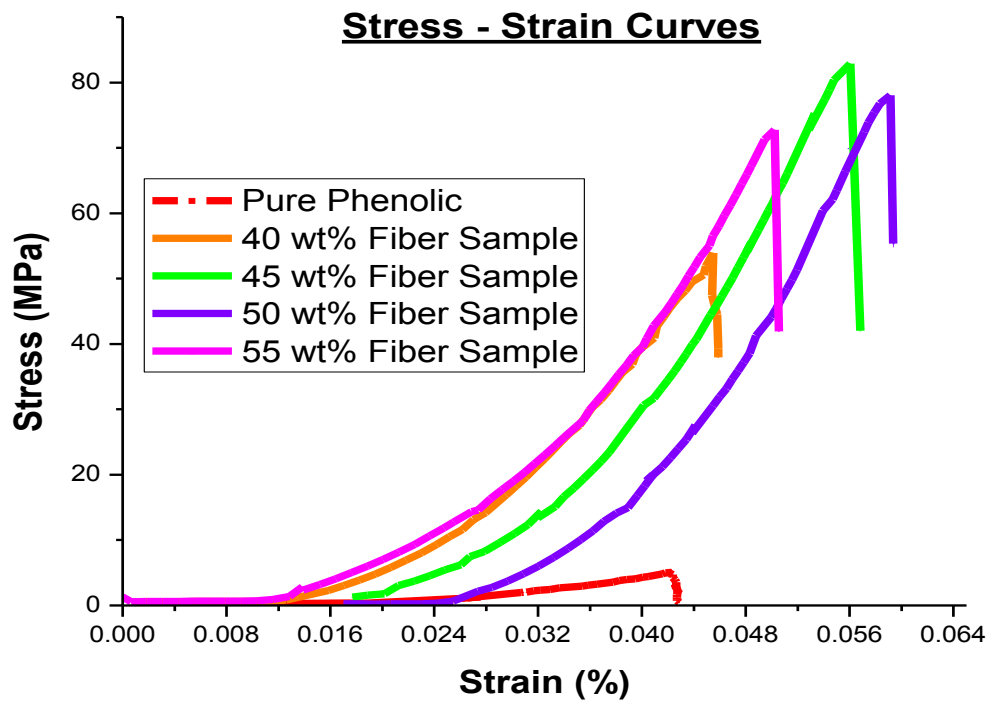


Figure 27 Stress Strain Curves

4.3.3 Modulus of Elasticity

Stiffness of a material is described by Modulus of elasticity. It is an important factor determining the strength and ductility of a material. It is determined in elastic region of stress strain curve by measuring ratio of stress to strain.

$$\text{Modulus of Elasticity} = \frac{\text{Stress}}{\text{Strain}}$$

$$E = \frac{\sigma}{\epsilon}$$

Slope of stress strain curve in elastic region gives an idea of modulus of elasticity

$$E = m = \frac{y_2 - y_1}{x_2 - x_1}$$

Modulus of elasticity of pure sample was found to be 0.3 GPa and results of modulus of elasticity for all specimens were measured as explained in section 4.3.1 and results are elaborated in Table 16. Graphical comparison for modulus of all samples is shown in Fig 30. It was observed that likewise tensile strength, modulus for sample No 2 i.e. 45 wt% fiber is highest (2.82 GPa) as obvious from graph shown below. Overall, modulus of pure phenolic is increased up to 9 times by addition of carbon fibers.

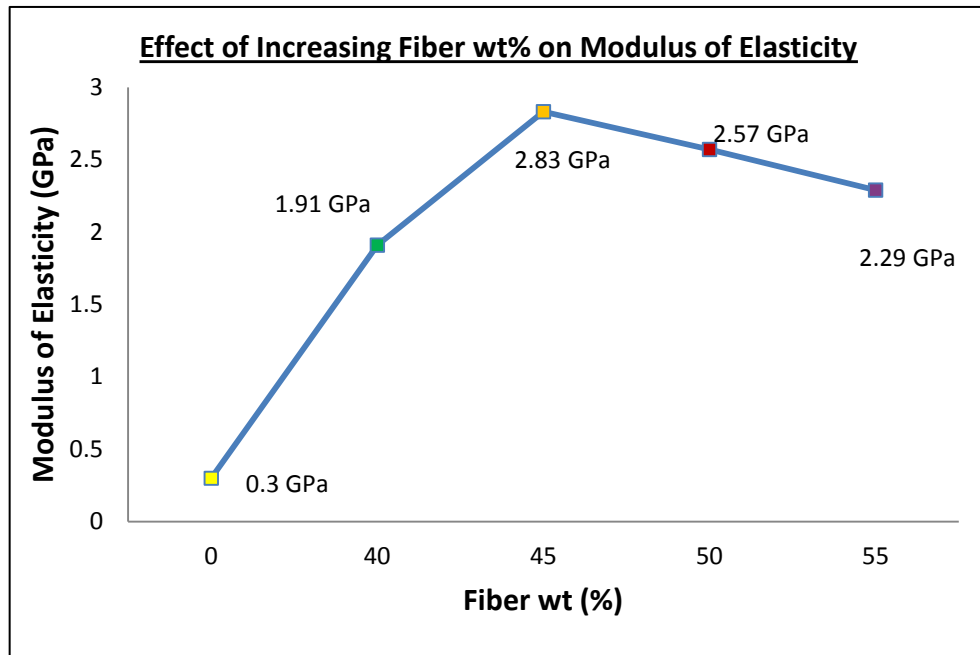


Figure 28 Effect of Increasing Fiber wt% on Modulus of Elasticity

4.3.4 Elongation at break

Elongation at break is the ability of a material to resist changes of shape without crack formation. Mathematically, it is the ratio between changed length and initial length after breakage of the test specimen. Plastics, fiber reinforced composites have values < 5%. High toughness is obtained with combination of high tensile strength and high elongation [16]. Elongation at break was measured using following formula:

$$\text{Elongation at Break (\%)} = \frac{\text{Max Extension}}{\text{Guage Length}} \times 100$$

Elongation at break of pure sample was found to be 4.3 % and results of modulus of elasticity for all specimens were measured as explained in section 4.3.1 and results are elaborated in Table 16. If results are analyzed, it would be noticed that Sample No 3 showing better elongation at break as compared to other samples i.e. 5.56 %. Overall it was observed that elongation at break of pure phenolic sample is increased up to 1.2 times by addition of carbon fibers. Graphical comparison of all samples is as shown in Fig 31.

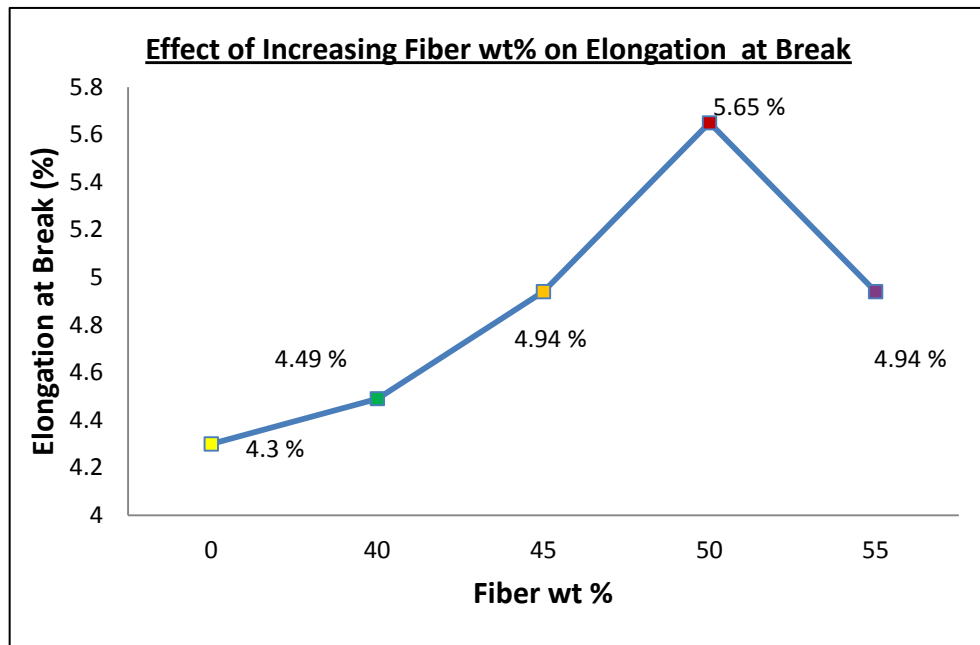


Figure 29 Effect of Increasing Fiber wt% on Elongation at Break

4.3.5 Discussion on Tensile Test Results

Literature study reveals that strength of composite materials generally increases by addition of reinforcement [29]. This fact has been proved by the results that tensile strength, modulus and elongation of pure phenolic is increased by addition of carbon fibers. Fibers have a constraining effect on movement of chains as shown in schematic Fig 32. However if results of 04 x composite samples (Table 6) are compared, a different behaviour is observed in Sample 03 & 04.

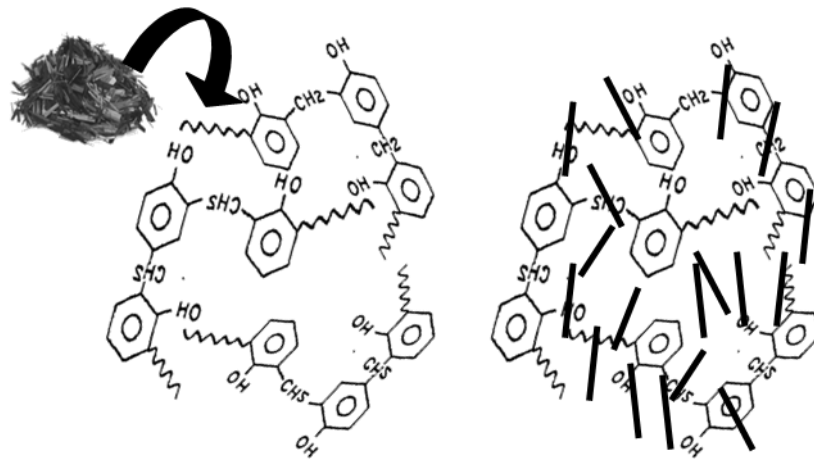


Figure 30 Chain restraining effect by addition of chopped carbon fibers

The reason for this gradual decrease in mechanical properties after certain wt % addition of carbon fibers may be attributed to behavior of fiber/resin in a composite, interfacial strength [2] and void volume [3]. Carbon fiber is inherently brittle [2] [4] and it does not yield if compared with other synthetic fibers as demonstrated in Fig 33.

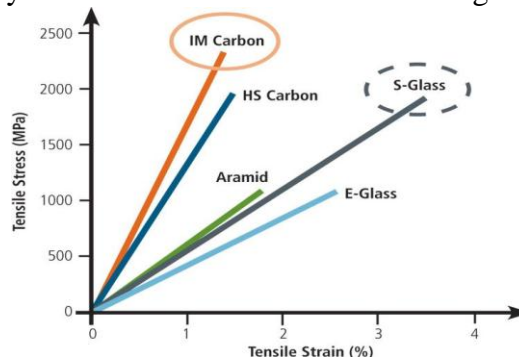


Figure 31 Stress Strain Behavior of Various Synthetic Fibers

Carbon fiber bends under stress. When stress passes beyond tensile strength of fibers they fail suddenly [19], whereas glass fiber shows deformation under load even after breaking as shown in figure 34

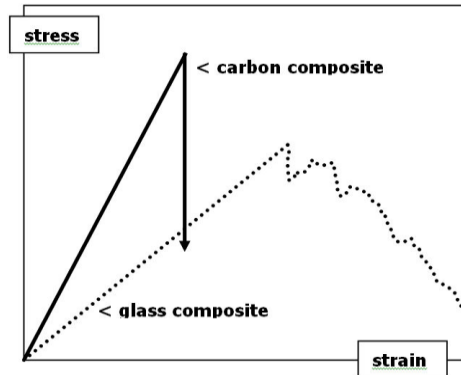


Figure 32 Stress Strain Behavior of Carbon and Glass Fiber Composite

Similarly, phenolic resin tends to be brittle as well [33]. Condensation nature of these resins during curing process leads to certain drawbacks as inclusion of voids and surface defects and poor mechanical properties [4].

Therefore increasing fiber weight % to a certain amount leads to drop in tensile strength.

Another reason for decline in mechanical properties after certain wt% addition of carbon fibers in phenolic matrix is fiber matrix interfacial strength. Mechanical properties depend on quality of fiber-matrix interface as load from fiber is transferred to matrix at the interface [2]. Adhesion at interface is composed of Vander Waals and electrostatic forces. Maximum interfacial strength is achieved when fibers are properly wet by resin, therefore when weight % of carbon fiber increases in phenolic matrix, resin is not sufficient to infiltrate and properly wet the fibers. Carbon fibers are hydrophobic in nature [30] so enough resin is required to wet the fibers. It has been noticed during experimentation that carbon fibers require resin twice the quantity of fibers for optimum wetting required for processing. However, increasing resin has also some drawbacks such as decrease in strength [33] and ablation rate. Another

reason for decrease in strength may also be the agglomeration of fibers at high concentration, when sufficient resin is not available in matrix to wet the fibers properly. This agglomeration behavior is responsible for maximum fiber-fiber interaction than fiber-matrix interaction. Therefore an optimum fiber weight percent evenly dispersed in phenolic matrix is required to achieve the best results. This optimum fiber wt % is found to be 45 %, i.e. Sample No 2, as depicted from Figures 28 ~ 31.

4.4 Impact Test

Impact test determines energy absorbed by a material during fracture when subjected to high strain rate.

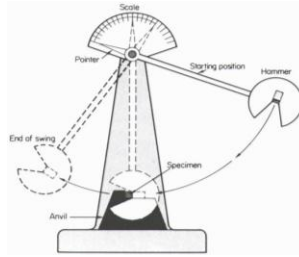


Figure 33 Impact Testing Machine

4.4.1 Results and Deduction

Impact test was performed at room temperature for 04 x samples with increasing wt% Fiber as mentioned above in Table 6. 06 x specimens from each sample were machined and average value for 03 specimens with valid results has been accounted for as shown in Table 17

Table 17 Impact Test Results of Composite Samples

Sample ID	Sample # 01	Sample # 02	Sample # 03	Sample # 04
Fiber wt%	40 wt % Fiber	45 wt % Fiber	50 wt % Fiber	55 wt % Fiber
Unit	J/m ²	J/m ²	J/m ²	J/m ²
Specimen # 01	39	30	30	23
Specimen # 02	33	43	28	23
Specimen # 03	15	27	23	22

Average	29	33	27	23
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It may be observed from impact test results in Table 17 that impact strength of pure phenolic is increased by addition of carbon fibers, i.e. from 1.9 J/m² (Pure phenolic) to 29 J/m² (addition of 40 wt% fibers) which is about 15 times greater than impact strength of pure sample. Impact strength further increases up to addition of 45 wt% fiber and then drops as fiber weight % is further increased.

Reason for this drop is brittle behavior of carbon fiber [19] and phenolic resin [33], As explained earlier in Section 4.3.3, that carbon fiber phenolic composite shows no deformation under load and tend to fail catastrophically Fig 34. Moreover The composite with a more fiber wt% becomes more rigid, resulting in lower impact strength. [14]. Graphical comparison is shown in Fig 36.

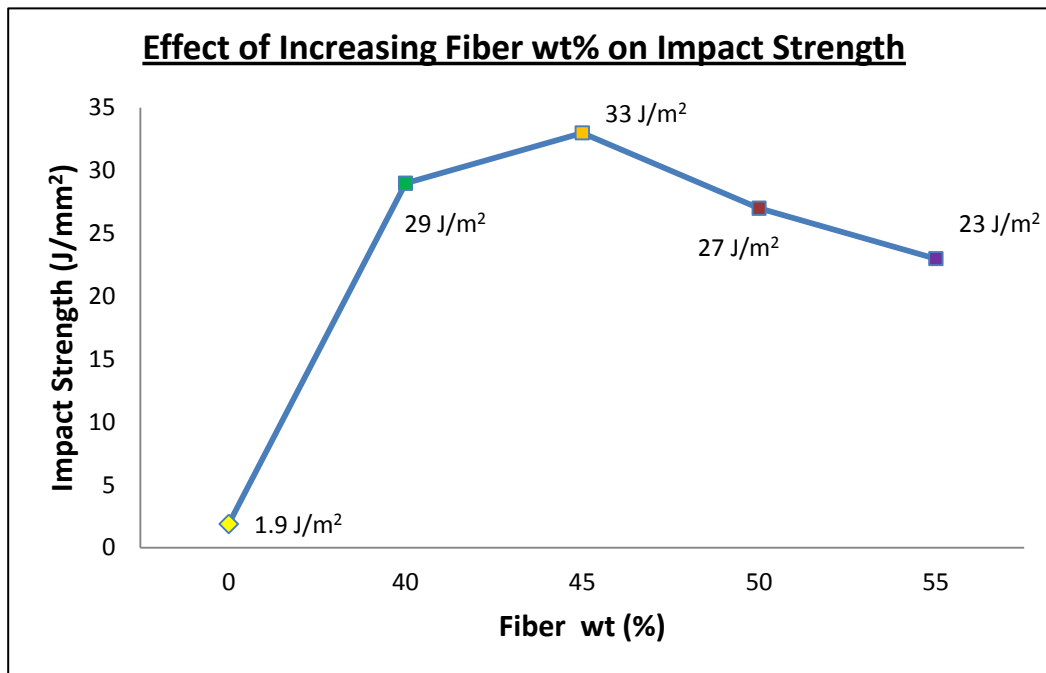


Figure 34 Effect of Increasing Fiber wt% on Impact Strength

4.5 Ablation Rate

Ablation involves material from surface by chipping, vaporization or any other erosive processes. Ablation test is performed for composite which are to be employed at high temperature and high fluid velocities. Important test parameters used for ablation rate testing are mentioned in Table 18.

Table 18 Test Conditions for Ablation Rate Test

S No	Test Conditions	
1	Sample Size	ϕ 30 mm δ 10 mm
2	Flame Temperature	3000 °C
3	Exposure Time	20 sec
4	Total flow rate of acetylene and oxygen	2628 L/h
5	Angle of flame exposure	90°
6	Distance between flame tip and sample	10 mm
7	Calculation Formula	$R_L = \frac{\Delta L}{T} = \frac{L_1 - L_2}{T}$ <p> R_L = Linear Ablation Rate , mm/s L_1 = Initial Thickness , mm L_2 = Residual Thickness after ablation , mm T = Ablation Time , sec </p>



Figure 36 Ablation Rate measuring apparatus

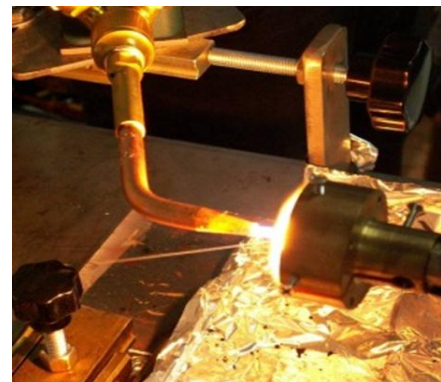


Figure 35 Ablation Rate Test

4.5.1 Results and Deductions

Ablation rate was performed on 04 x samples as per Table 6 and an average of 06 specimens from each composite sample is accounted for. Results are recorded in Table 19

Table 19 Ablation Test Results of Composite Samples

Sample ID	Sample # 01	Sample # 02	Sample # 03	Sample # 04
Fiber wt%	40 wt % Fiber	45 wt % Fiber	50 wt % Fiber	55 wt % Fiber
Unit	mm/sec	mm/sec	mm/sec	mm/sec
Specimen # 01	0.02	0.03	0.02	0.04
Specimen # 02	0.04	0.03	0.02	0.03
Specimen # 03	0.03	0.04	0.02	0.02
Specimen # 04	0.03	0.03	0.02	0.01
Specimen # 05	0.03	0.01	0.02	0.00
Specimen # 06	0.02	0.04	0.02	0.00
Average	0.028	0.03	0.02	0.016

It is obvious from the test results in Table 19 that by increasing fiber weight % in phenolic matrix, ablation rate is decreased from 0.028 mm/sec (40 wt% fiber) to 0.016 mm/sec (55 wt% fiber).

Decrease in ablation rate may be attributed to excellent fire resistant and chemically inert properties of Carbon fiber [19]. Similarly phenolic resin chars when heated to temperatures $>500^{\circ}\text{C}$ and converts to amorphous carbon. This property gives unique ablative phenomena to phenolic resins [8]. Combined effect of these unique properties makes phenolic resin composites ideal to be used at high temperature environments such as rocket nozzles [9] where strength is not the only design requirement. Moreover heat flow in phenolic/Carbon composites is restricted by randomly orientated Carbon fibers as shown in schematic Fig 39

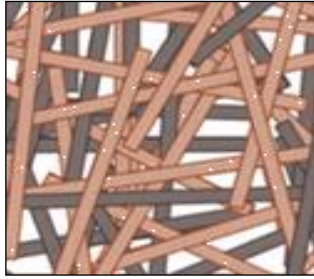


Figure 37 Discontinuous and Randomly Orientated Carbon Fibers

Graphical representation of test results are shown in Figure 40

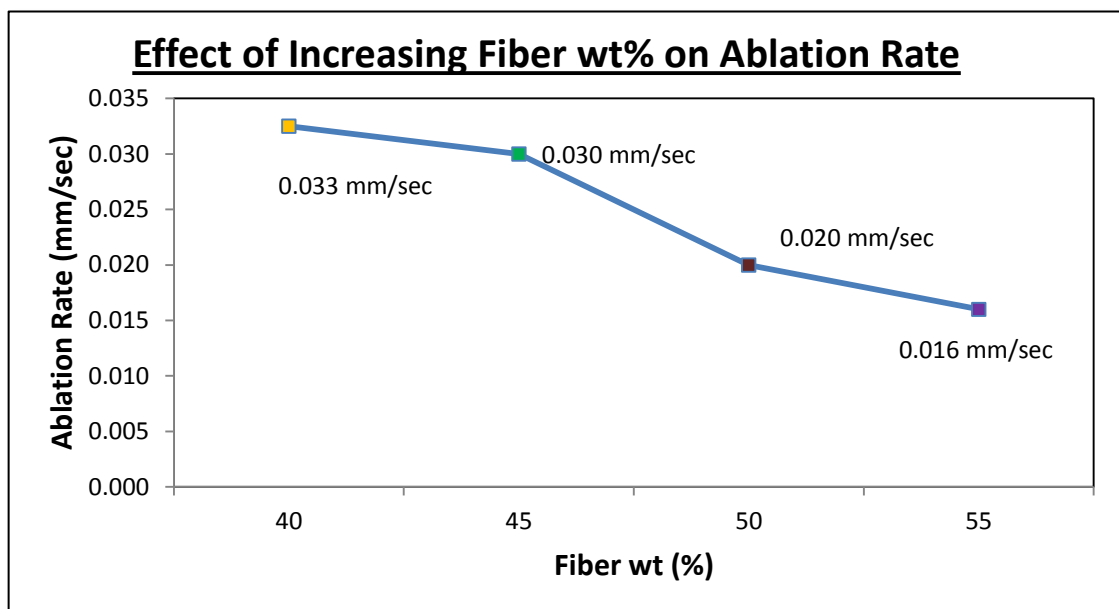


Figure 38 Effect of Increasing Fiber wt% on Ablation Rate

4.6 Thermogravimetric analysis

Thermogravimetric analysis (TGA) is a thermal analysis techniques used to characterize materials by measuring rate of change of sample mass as a function of time or temperature in controlled environment. This technique is employed to determine the thermal stability and compositional properties of various materials.

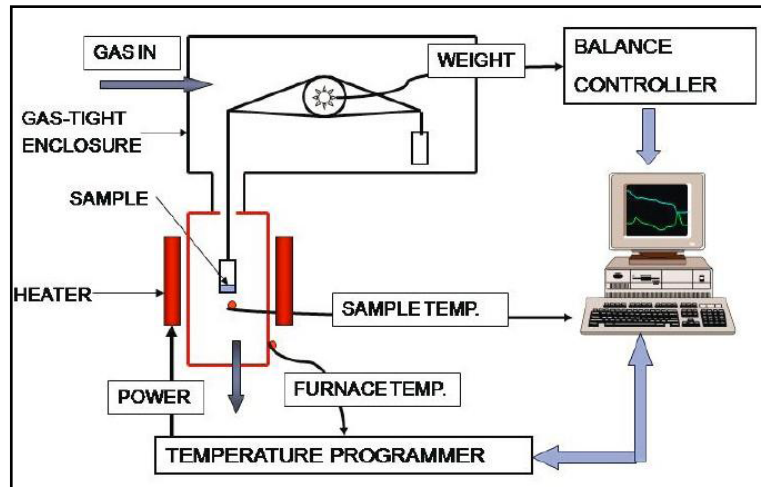


Figure 39 Schematic of TGA Apparatus

4.6.1 Experimentation

TGA was performed on pure phenolic sample and 04 in samples per Table 6 .Test conditions are as under:

- Test Temperature: 0 ~ 900 °C
- Heating Rate: 10 °C / min
- Environment: Nitrogen Gas

4.6.2 Results and Deductions

Results are summarized in Table 20

Table 20 Test Results of TGA

S No	Sample ID	Decomposition Onset
1	Pure Phenolic Sample	380 °C
2	40 wt% Fiber Sample	400 °C
3	45 wt% Fiber Sample	440 °C
4	50 wt% Fiber Sample	455 °C
5	55 wt% Fiber Sample	470 °C

Test results in Table 20 are revealing that thermal degradation temperature of pure phenolic is increased 90 °C with addition of Carbon fibers, which is a depiction that fiber content has influence on the thermal degradation: the composites with the highest fiber content degraded at a higher temperature. Thermal properties of Carbon fibers add to that of Phenolic Resin indicative of High temperature stability of Carbon Phenolic Composites. TGA curves have been shown in Fig 42

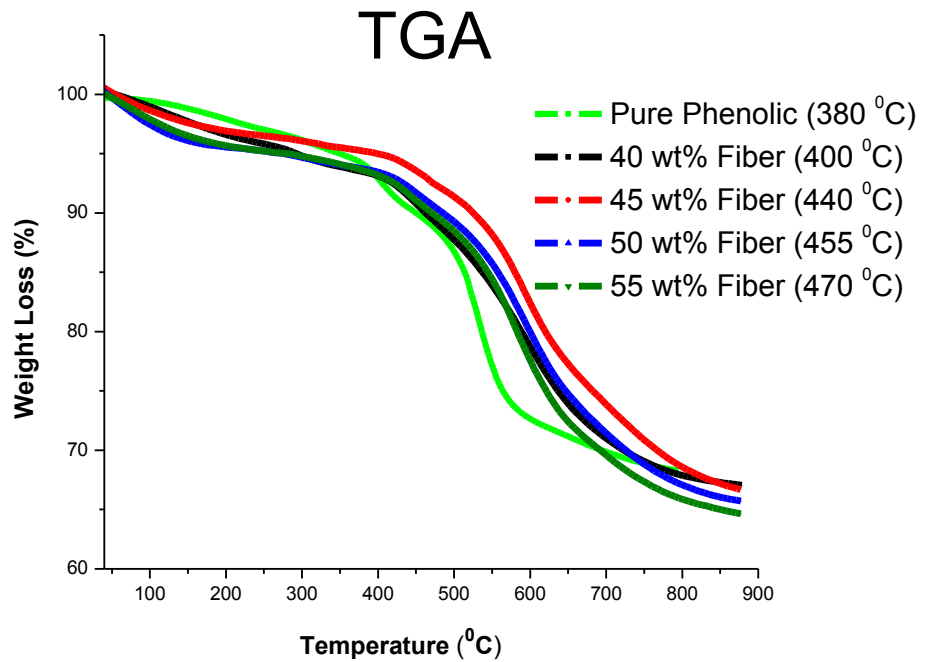


Figure 40 TGA Curves

4.7 Differential Scanning Calorimetry

In a DSC experiment the difference in energy input to a sample and a reference material is measured while the sample and reference are subjected to a controlled temperature program. In DSC, $DT = 0$ maintain between sample and reference .DSC measures the Energy necessary to measure or to maintain zero temperature difference between reference and the sample .DSC employs two compartments having furnace, thermocouples, gas controller , and recorder as shown in Fig 43.

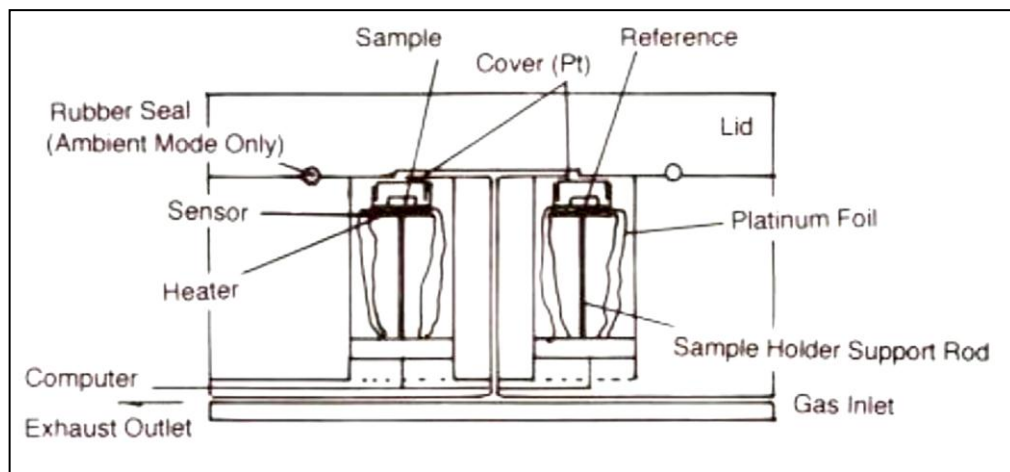


Figure 41 Schematic of DSC

4.7.1 Experimentation

DSC was performed on pure phenolic sample and 04 in samples per Table 6 .Test conditions are as under:

- a. Test Temperature: 20 ~ 200 °C
- b. Heating Rate: 10 °C / min
- c. Environment: Nitrogen Gas

4.7.2 Results and Deductions

Results are recorded in Table 21

Table 21 Test Results of DSC

S No	Sample ID	T _g
1	Pure Phenolic Sample	100
2	40 wt% Fiber Sample	102
3	45 wt% Fiber Sample	84
4	50 wt% Fiber Sample	83
5	55 wt% Fiber Sample	82

The glass transition temperature (T_g) of pure phenolic sample was found to be 100 °C, it increased with addition of Carbon fiber (40 wt%) to 102 °C but then reduced with further addition of carbon fibers (45 wt%) to 84 °C. Further addition of carbon fibers has shown similar tendency to drop as shown in Fig 44

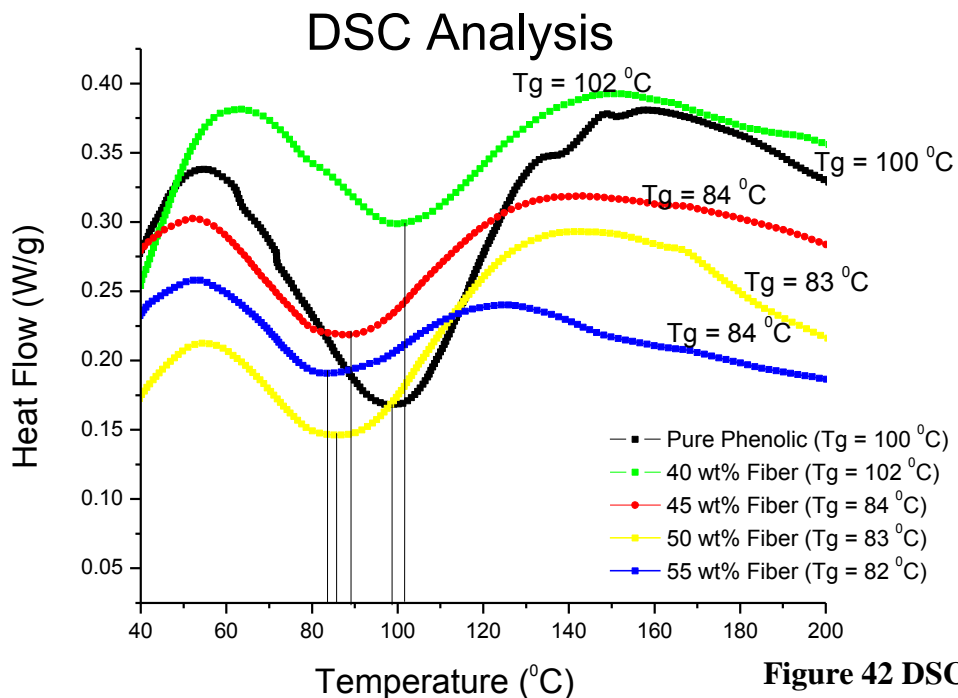


Figure 42 DSC Curves

The inclusion of carbon fibers to phenolic matrix shifted the T_g to lower temperatures. Lowering in T_g shows that chain mobility of phenolic matrix has increases when restricted in the narrow spaces of carbon inter galleries. [27] It has been reported stable constrained region in the polymer segments have been imposed by reinforcement [28] .Pure resin is well entangled & Cross linked and chains tend to arrange around fibers according to interaction at interface with fiber. Entanglement density decreases during attaching/aligning of chains around fibers as shown in schematic Fig 45. With increase in temperature chain mobility occurs due to increase in localized free volume around fiber leading to lowering of T_g with addition of Carbon Fibers

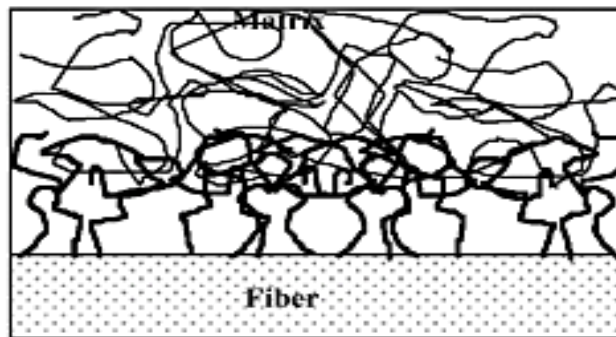


Figure 43 Matrix chain alignment after interaction with fiber

4.8 Conclusion

Introduction of carbon fibers was found to increase the density, tensile strength, modulus and impact strength of phenolic resin matrix. Ablation rate was however reduced as expected due to heat resistant properties of Carbon fibers and phenolic resin. In order to find best mixing ratio satisfying the strength requirements as well as thermal properties for any given aerospace applications, a comparison of various test results has been made.

4.8.1 Comparison between Density and Ablation rate

If test results for density and ablation rate of 04 x samples are compared, general trend is observed that density is increased by addition of carbon fibers and ablation rate is decreased. Best results are achieved in Sample No 04 i.e. 55 wt% fibers as shown in Fig 46

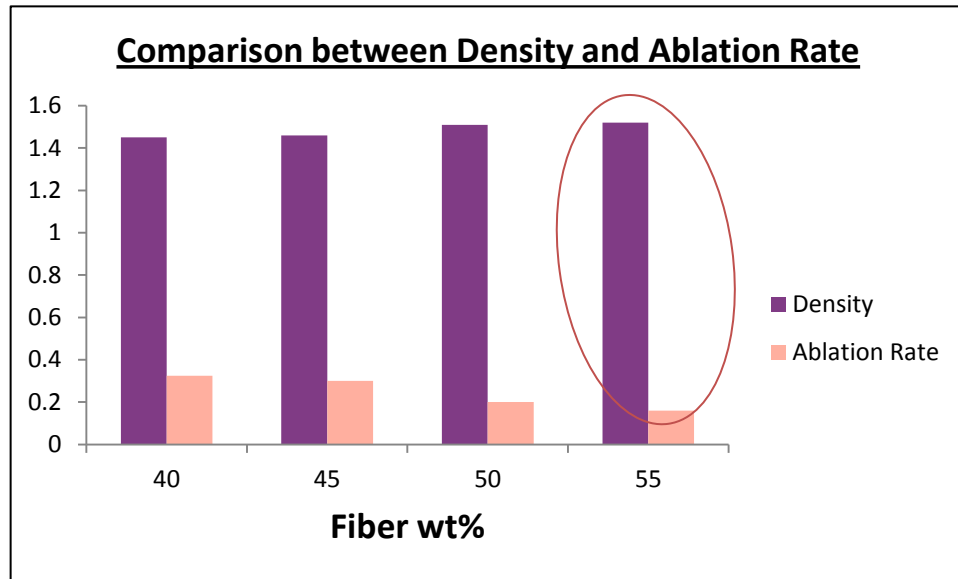


Figure 44 Comparison between Density and Ablation Rate

4.8.2 Comparison between Tensile Strength and Impact strength

If the test results of tensile strength and impact strength of all 04 x composite samples are compared, different behavior is observed. It is obvious from Fig 47 that tensile strength and impact strength of 45 wt% Fiber sample is showing comparatively better results.

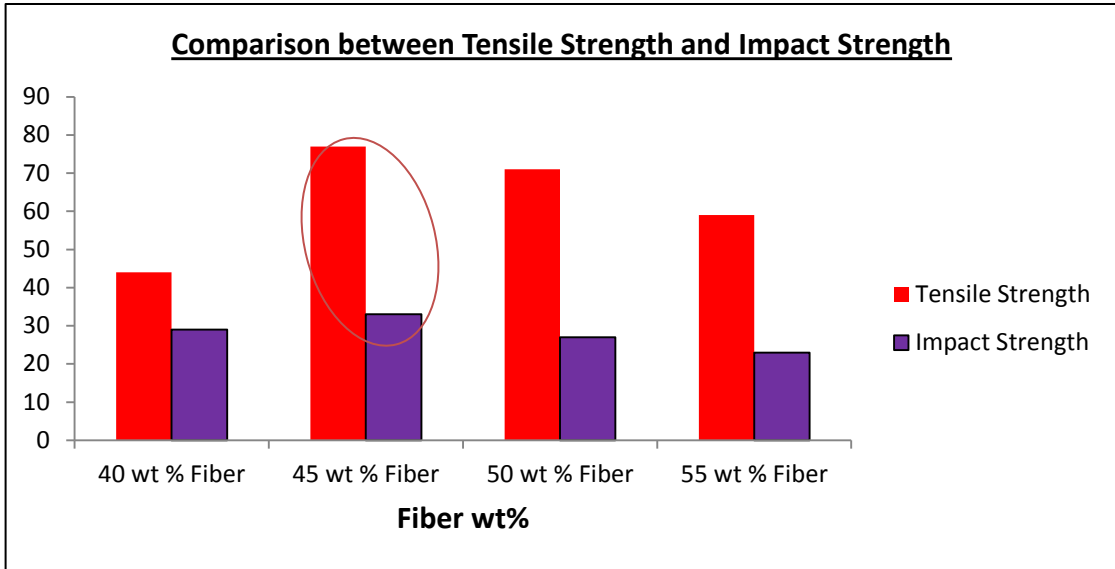


Figure 45 Comparison between Tensile Strength and Impact Strength

4.8.3 Comparison between Modulus and Elongation at Break

Comparison drawn between 04 x composite samples in Fig 48 shows that modulus of elasticity for 45 wt% fiber sample is highest, however elongation at break is relatively less than 50 wt% fiber sample

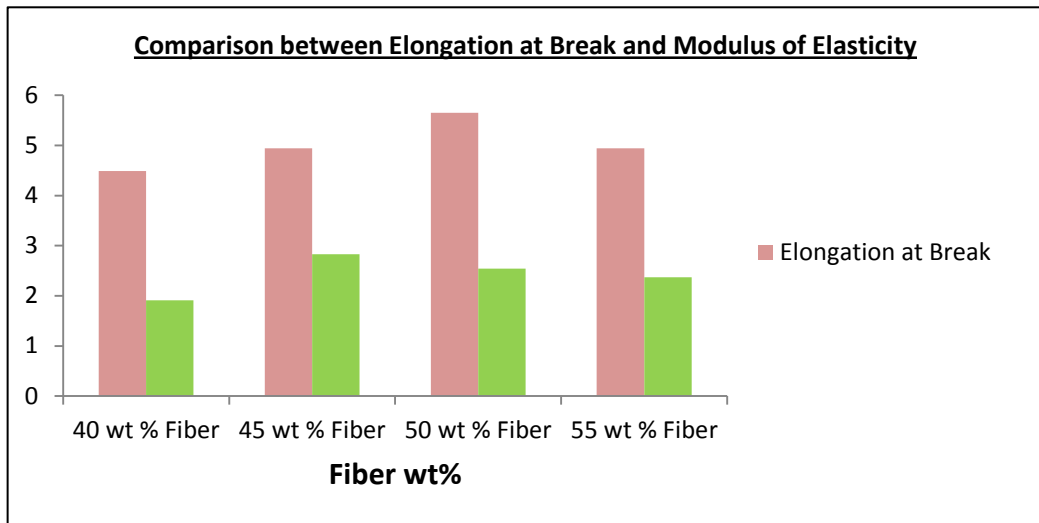


Figure 46 Comparison between Elongation at Break and Modulus of Elasticity

4.8.4 Comparison between TGA and DSC Results

Results of TGA are showing that decomposition temperature is increasing by increasing carbon wt% in phenolic resin whereas , DSC results have shown that glass transition temperature has decreased in general by addition of carbon fibers to phenolic resin .

4.8.5 Conclusion

It may be concluded from above mentioned comparison that Sample No 02 i.e. 45 wt% fiber composite is showing optimum properties with better tensile strength, impact strength and modulus of elasticity, good ablation rate and acceptable thermal degradation behavior for an application where strength as well as temperature stability is important design parameter.

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