Effect of Carbon Fiber Properties on Ablation Performance of Fiber Reinforced Phenolic Composites



By

Ayesha Butt

School of Chemical and Materials Engineering (SCME)

National University of Sciences and Technology (NUST)

2016-2018

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Name: Ayesha Butt

Reg.No: 00000173070

This thesis is submitted as a partial fulfillment of the requirements for the degree of

MS in Materials and Surface Engineering

Supervisor Name: Dr. Muhammad Shahid

School of Chemical and Materials Engineering (SCME)

National University of Sciences and Technology (NUST)

H-12 Islamabad, Pakistan

October, 2018

This thesis is dedicated to my parents

Acknowledgements

All praise to Allah Almighty, who bestows and blesses the mankind with an ability to ponder over forces of nature and carry out scientific researches. In the first place, therefore, I express my utmost thanks to Allah Almighty for helping me accomplish this project.

I express my sincere gratitude to my supervisor Dr. Muhammad Shahid and my cosupervisor Dr. Imran Sadiq for their worthy guidance throughout the project. I consider myself privileged to work under their supervision.

I also thank the entire faculty of Materials Engineering department in SCME (School of Chemical and Materials Engineering) for helping me with the research work. The help from lab engineers and technicians, particularly Mr. Saleem is also noteworthy. I would also like to acknowledge the departments of USPCAS-E and SMME for the help in my experimental work.

Lastly, I would like to thank my family and friends for love, support and sincere prayers.

Abstract

Ablative composites are also called as thermal protection system (TPS) which have a wide application in rocket nozzles, engine throats and leading edges. They are used to protect the metallic structure or component usually from high temperature environments by sacrificing its surface. In this particular case, carbon cloth from three different sources with different properties was used as reinforcement material and phenolic resin was used as matrix. The ablation behavior was observed at SCME, NUST.

For ablation test, a set up was made at workshop of SCME in which Oxy Acetylene flame was used as heating source and back face temperature of the sample was recorded by using data logger. The effect of different densities, carbon content and breaking load of carbon cloths on ablation resistance of the composite was observed while all other factors like weight percentage of reinforcement, orientation of fibers and curing temperature etc. were kept constant. It was observed that the composite with the most dense carbon cloth had the maximum resistance to ablation. The linear and mass ablation rates of the samples were also calculated and compared.

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Chapter 1: Introduction

1.1 Ablation:

Ablation is removal of surface material of a component by erosion and corrosion due to high temperatures, pressures and velocities. This type of process can happen on the surface of the objects entering the Earth's atmosphere at hypersonic speeds for example a spacecraft or a satellite re-entering the atmosphere from outer space or during the mission course of a ballistic missile or in propulsion structures.

Basically ablation is the process in which surface of a material continuously degrades by chemical reactions at high temperature and erosion due to high velocities. Usually, erosion rates increase as the speed of vehicle is increased. Also, heat is directly proportional to the square of speed of the vehicle. Heat transfer to vehicle is also affected by density and composition of gases in atmosphere.

This process can be minimized by changing shapes of entry vehicles and shielding the body by the use of a surface material that has very high heat capacity, good mechanical properties, low thermal conductivity and reactivity to protect inner body or component. These ablative materials are usually used at greater heat loading points like rocket's nozzle, nose cone or leading edges of wings. The purpose of using ablative materials is to keep the structure cool by creating a chemical barrier which reduces heat and thermal transfer from atmosphere to inner component[1].

1.1.1 Mechanism of Ablation:

At ultra-high temperature and velocity conditions, the material goes through two types of processes:

 a) Thermally induced processes: These processes are not affected by the type of chemical species present in the environment; rather they occur due to temperature effects like surface pyrolysis, vaporization and sublimation. b) Chemically induced processes: These processes occur due to chemical interaction between ablator and chemical species in the environment like combustion and chemical erosion.

Processes like vaporization and sublimation occurs in ablators containing Silica and Carbon. The reaction products will effuse to surface and as result some material is lost. Surface pyrolysis occurs in case of non-Charring Ablators while in charring ablators bulk pyrolysis occurs in which surface of ablator reacts with environments for a period of time and forms a carbonaceous layer which is called char. After the formation of char layer decomposition occurs behind the char layer and the products effuse through this char layer. The thickness of char layer depends on the type of ablative material used for example phenolic resins have high char yields than epoxies. Char layer has high porosity which decreases thermal diffusivity resulting in an increase in burn through time. The burn through time is the time required to force the heat vertically from front surface to rear surface through center of the materials with constant velocity and pressure.

Therefore ablation is the process in which a material absorbs heat from the surroundings, degrades and the energy absorbed is dissipated in to different mechanisms[2].



Figure 1: Ablation process in Charring Ablator[3]

1.1.2 Properties of Ablative Material:

Following are the most basic properties of an ablative material to protect inner body or components from high temperature of the environment[2, 4]:

- i. Ablative material should have good insulation properties so the heat is confined to the surface and does not travel beyond ablation zone.
- ii. Low thermal conductivity
- iii. High char yield
- iv. Good thermal and mechanical shock resistant
- v. High heat of Ablation
- vi. Low cost
- vii. Low density
- viii. Good bonding ability to substrate

1.2 Ablative composites:

A composite is a physical combination of two phases or materials which retain their properties to make a new material that has balanced properties of both individual components and can be used for a range of applications.

The continuous soft phase provides toughness and is called "matrix" while the dispersed or discontinuous phase which is hard and provides stiffness is "reinforcement". Matrix and reinforcement are separated by interface.

Classification of composites:

Composites can be classified on the basis of the type of matrix or the type of reinforcements[5].

1. Classification on the basis of matrix:

On the basis of continuous phase, composites can be classified in to three main groups; Metal matrix composites (MMCs) are composed of continuous metallic phase and ceramics or metals are reinforcements, Ceramic matrix composite (CMCs) are composed of continuous ceramic phase and other ceramics as a dispersed phase while Polymer matrix composites (PMCs) has a continuous polymer (thermosets or thermoplastics) phase and fibers as reinforcements.

2. Classification on the basis of reinforcements:

Composites can be particle-reinforced or fiber-reinforced. Particle reinforced composites can have large particles as reinforcements or they can be dispersion strengthened in which crystalline particles are dispersed in matrix.

Fiber-reinforced composites can have continuous and aligned fibers or discontinuous and short fibers. Discontinuous fiber composites are further divided in to aligned and randomly oriented composites. Change in concentration, size, shape, distribution and orientation of fibers may have a large influence on the properties of composites.



Figure 2: Constituent elements and classification of Composites[6]

Ablative composites are the composites which are used to protect a structure, component or a body from high temperature environments by sacrificing its surface. They are also called as thermal protection systems (TPS) materials. The purpose of using TPS materials is to provide a heat shield that has insulating properties and is ablative resistant. Ablative composites are usually used in aerospace industry to shield a component from excessive temperature produced during hypersonic flights[7].

Non-Polymeric (such as metals, ceramics and inorganic polymers) as well as polymeric matrixes composites are being used as ablative composites. Polymeric ablatives (PAs) are widely used as compared to non-polymer matrices due to their properties like low density, high shock resistance and good mechanical properties.

1.3 Types of Ablative composites:

The following figure represents the types of Ablative composites that are being used as protective layer:



Figure 3: Types of Ablative Composites[8]

1.3.1 Inorganic Composites:

Metal matrix composites (for example Rhenium and tungsten composites) are used as TPS materials because of their high performance in high thermal and chemical environments. They are used as heat shield materials in nozzle and nose cone as their erosion rates are very less and throat regions of rocket engines. Major drawbacks include cost and higher densities of the materials which lead to high thermal conduction. If metals are even deposited as a thin layer coating on the other low dense materials like graphite which reduces the cost and thermal conductivity, their purpose is still limited due to high co-efficient of thermal expansion (CTE) at high temperatures[9, 10].

Ceramics coatings and composites (for example Yttrium Y_2O_3 and Zirconium Carbide ZrC) are also used in combustion chambers and exhaust nozzles due to low erosion rates, high specific heat and high melting points[11].

1.3.2 Organic Composites:

Among ablative materials Carbon is considered very important Ablative material due to its high heat capacity, high energy of vaporization and high heat of ablation. Therefore, it can be used as fibers embedded in carbon matrix as well as in polymer matrix.

Carbon-Carbon Composites (CCCs) are formed by combining the mechanical strength of carbon fibers with ablation resistance of carbon matrix. Two and three dimensional carbon fibers in the form of carbon cloth or carbon windings are embedded in carbon matrix to produce CCCs.

Polymeric Ablatives (PAs) are used because their properties can be tuned in bulk. This is considered the most important class for TPS materials because of their versatile properties like low thermal conductivity, low erosion rate, low density, good mechanical properties, high specific heat and high heat of ablation. PAs are mostly used in rocket's thrust chamber and nozzle tip[10].

1.4 Polymer Ablative Composite:

1.4.1 Polymer matrix:

Polymer matrix in ablative composites is favored over metals and ceramics because of their low densities and the tendency to carry effective pay load for atmospheric re-entry vehicles and ballistic missiles.

The polymer matrix can be further divided in to charring and non-charring matrices. Char is a carbon rich layer which is formed due to chemical combustion at high temperature at the surface of the ablator. This layer provides the chemical and thermal barrier to diffusion of gases and heat from atmosphere to inner component or material. Non-charring Polymer matrix is used when ablative is removed leaving a clear surface. For example polytetrafluoroethylene (PTFE) decomposes at high temperature leaving a clean surface and no solid residue.

However, in most ablators carbonaceous solid residue in the form a char layer is required. For example partial combustion takes place on the surface of phenolic resins and silicon rubbers to produce char layer which as reflective surface to heat and chemical gases in atmosphere[10]. Some of the polymer matrices which are used in aerospace industries for high temperature applications are listed in the table:

Polymer Matrix	Pros	Cons
Silicon Rubber	Excellent heat resistant, Resistant to ageing	Moderate mechanical properties, sensitive to oil water and steam
Ethylene Propylene Diene Monomer (EPDM)	Good mechanical properties, resistant to oil and steam, high heat of ablation, Easy processibility	Poor resistance to hydrocarbons, poor adhesion to metals
Styrene Butadiene Rubber (SBR)	Ablation and ageing resistant	Flammable, Poor resistant to oil and gas
Acrylonitrile Butadiene	Good mechanical properties,	Flammable,
Rubber (NBR)	Resistant to chemicals	Limited Shelf life
Phenolic Resins	High thermal stability, Good mechanical Properties, resistant to chemicals	Oxidation of charred layer in Oxygen containing environment

Table 1 : Advantages and disadvantages of different Polymer Matrixes[8]

1.4.2 Reinforcements:

Despite of using high char yielding polymer matrices like phenolic resins, carbonaceous residue tend to erode due to high pressures and temperature. That is why to provide mechanical strength to ablative polymer and to improve or increase heat of ablation reinforcements are used making it fiber reinforced polymeric ablatives (FRPAs). In a polymeric ablative composite, matrix provides ablation resistance by the formation of charred residue while the reinforcements provide mechanical strength to the charred layer[10].

Carbon Fibers, Silica, glass, Kynol and aramid fibers are used as reinforcements in polymer ablatives. In some systems low conducting powders such as Zirconia is also used along with fibers.

In EPDM based ablatives, Kynol, silica and aramid fibers are used resulting in Kynol giving the best ablation resistance and adhesion of charred layer to virgin material while silica fibers giving char layer that do not have good adhesion with virgin material. Aramid fibers produced char which had better quality as compared to silicon/EPDM ablative system[12].

In phenolic based ablative systems, carbon, glass and silica fibers are used. The erosion rate for glass fibers/phenolic system is greatest while the burn through time is least. For carbon fibers, insulation index and burn through time is greatest which leads to improve heat of ablation[13].

1.5 Fabrication of CF Reinforced Phenolic Composites:

Fabrication of CF reinforced Composites is done through many processes. Some of which are listed as under:

- 1. Hand lay-up
- 2. Open mold
- 3. Resin infusion Processes
- 4. Filament winding
- 5. Infusion molding

6. Compression molding

While comparing the aforementioned processes, filament winding is the process which is used for manufacturing of high performance composites. For easy and safe handling of fibers and fabrics, they are first transformed to prepegs. Prepegs are then employed to make composites.

1.5.1 Formation of prepegs:

Prepegs are formed when carbon fibers or fabrics are impregnated in resin for a particular period of time in a certain ratio. Prepegs have certain unique set of properties as they are partially cured under high temperature and pressures. They are formed for easy handling of fibers and can be stored at low temperatures for a month. They are kept at low temperatures to avoid complete curing of resin which is usually done in an autoclave or oven.

After prepegs are formed they are usually tested for RVS (resin, volatile, soluble resin) parameters before storing. The RVS parameters for carbon fabric are given in Table 2:

Parameters	Values
Resin Content	39-55%
Volatile Content	3-8%
Soluble resin content	30-45%

Table 2: RVS values for prepegs[14]

There are two types of processes through which prepegs are formed:

1.5.1.1 Hot melt process:

Unidirectional fibers and fabrics both can be impregnated in resin by this method. A thin film on resin is deposited on to a paper substrate which is then interacted with fibers or fabric in a prepegs machine at high temperature to deposit on to the fabrics.

1.5.1.2 Solvent dip process:

Only fabrics are impregnated in resin by this process. Fabrics are dipped in to a resin solution which is made by dissolving resin in a solvent bath. Then by using a drying oven solvent is evaporated from the fabric forming prepegs[14].

1.5.2 Filament winding process:

This process is used for creating composites by winding pre-impregnated fiber filaments and fabric on to rotating mandrels or tools. The process is controlled by automated machine with well-defined and calculated schematic pattern. Appropriate tension, roller pressure and continuous exposure of heat to prepeg tape ensure wrinkle free, smooth winding of fabric on mandrel. [15]

1.5.3 Hydroclave Curing:

The curing of the composite is done to acquire required density, thermal and mechanical properties for composite parts. These properties are required for application at high temperature conditions. In this process, water is used as medium to transfer heat and pressure to semi cured part for further curing.

Once the composite is formed, it can be tested as required. The physical and mechanical properties for high performance ablative composites are given in Table 3:

S. No.	Property	Unit	Value
1.	Density	g/cm ³	≥1.38
2.	Tensile strength	MPa	≥29
3.	Compression strength	МРа	≥55
4.	Bend strength	MPa	≥44
5.	Shear strength between layers	МРа	≥14

 Table 3: Physical and Mechanical properties for winded Composites[16]

6	Oxyacetylene	mm/s	<0.08
0.	ablation rates	1111/ 5	_0.00

1.6 Ablation Measurement:

Linear and Mass ablation rates are measured by using oxyacetylene flame. Following are some of the factors which are taken in to consideration for ablation rate measurements:

1.6.1 Cylinders:

Oxygen and Acetylene gases are sold in steel cylinders. Oxygen cylinders are usually painted green and contain compressed oxygen at pressures that can be as high as 2200psig. Acetylene cylinders are usually painted black and contain acetylene gas at pressure up to 250psig.

1.6.2 Oxy-Acetylene flame:

Oxyacetylene flame is formed by the reaction of oxygen and acetylene gases. This flame is used due to its high temperature reaching up to 3500°C. There are three distinct types of flame; Neutral flame is created when ratio of oxygen to acetylene is 1:1, carburizing flame is created when ratio of acetylene is greater than oxygen and oxidizing flame is created when ratio of oxygen is greater than acetylene.

Combustion of acetylene in the presence of oxygen is a two-step reaction. In first step, carbon reacts with oxygen to form carbon monoxide and then in the second step carbon monoxide reacts with oxygen from air to form carbon dioxide.



The first reaction occurs at well-defined inner core while the second step occurs at outer boundary. Carburizing flame has a feather like appearance around the inner core while oxidizing flame has shorter inner cone with loud noise.

For this experiment oxidizing flame was used.

1.6.3 Torch:

The torch assembly consists of handle, oxygen, and acetylene gas valve and mixing chamber. Oxygen and acetylene gas flow through tubes inside the handle and blend at tip. It is at tip that these gases ignite.

1.7 Selection of Materials Used:

Due to the properties discussed above phenolic resin is chosen as a matrix due to its high char yield, less amount of formation of volatiles and good elastic properties at low temperatures while carbon fabrics are chosen as reinforcements due to high heat capacity, high heat of ablation and good mechanical properties.

1.7.1 Phenolic or Phenol Formaldehyde Resin:

1.7.1.1 Structure and Formation of phenolic resin:

Phenolic or phenol formaldehyde resin was first produced as a synthetic polymer in 20th century and was known as Bakelite. It is produced by the condensation reaction of phenol and formaldehyde at appropriate reaction conditions and is chemically different from phenols and formaldehyde[17].



Figure 4: Condensation of Phenol and Formaldehyde [18]

1.7.1.2 Types of Phenolic resins:

There are two types of phenolic resins based on manufacturing conditions:

a) Phenolic Novolac resins:

Phenol reacts with small amount of formaldehyde in the presence of acidic environment to produce a thermoplastic having low molecular weight called Phenolic Novolac resin with the evolution of heat.



Figure 5: Formation of Novolac Resin [19]

b) Phenolic Resole resins:

Phenol reacts with excess formaldehyde in the presence of basic medium to produce a thermoset having comparatively high molecular weight than Novolac resins which are called Resole resins.



Figure 6: Formation of Resole resin[19]

1.7.1.3 Properties and Characteristics of phenolic resin:

Some of the properties of phenolic resin are given in Table 4:

Table 4: Propertie	s of Phenolic	Resin[20]
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S. No.	Properties	Unit	Value
1.	Density	g/ml	1.4-1.5
2.	Tensile Strength	MPa	100-140
3.	Tensile Modulus	GPa	5.5-7.5
4.	Flexural Strength	MPa	150-200
5.	Coeff. Of Thermal Expansion	^O C×10 ⁻⁶	10-15
6.	Coeff. Of Thermal Conductivity	W/m/K	0.20-0.24

Some of the characteristics of phenolic resins are as under [21-23]:

- 1. Low flammability
- 2. Ease of processing
- 3. Production of small smoke when burnt
- 4. High char yield
- 5. Low toxicity

- 6. Good thermal stability
- 7. Excellent impact strength
- 8. High stiffness
- 9. Chemically resistant
- 10. Good bonding ability

1.7.2 Carbon Fibers:

1.7.2.1 Manufacturing of Carbon Fibers:

Carbon fibers can be obtained from a number of precursors like Poly acrylonitrile (PAN), pitch and Rayon. The mechanical properties of fibers strongly depend on the type of precursor used. PAN and pitch are mostly used as a precursor for the commercial production of fibers while Rayon does not have commercial importance.

Multi filament carbon fibers (precursor fibers) are first obtained from precursor (pitch or PAN) via a variety of spinning techniques like wet spinning, dry spinning, or dry-jet wet spinning. These precursor fibers are then stabilized at low temperatures (~200°C-400°C) and then carbonized at higher temperatures (~1600°C) to increase the carbon content in fibers. High modulus fibers can be obtained through graphitization by further increasing the temperature (~3000°C). Then the inert surface of fibers is functionalized through a process called sizing to improve its adhesion to composite matrix.[24]



Figure 7: Flowchart of manufacturing of Carbon Fibers[24]

1.7.2.2 Types of Carbon Fibers based on mechanical properties:

Given table shows the different grades of carbon fibers based on mechanical properties:

S. No.	Туре	Elastic Modulus (GPa)	Elastic Strength (MPa)
1	Low Elastic Modulus (LM)	≤200	≤ 3500
2	Standard Elastic Modulus (HT)	200-280	≥2500
3	Intermediate Elastic Modulus(IM)	280-350	≥3500
4	High Elastic Modulus (HM)	350-600	≥2500
5	Ultrahigh Elastic Modulus (UHM)	≥600	≥2500

Table 5: Types of Carbon fibers based on Mechanical Properties[25]

1.7.2.3 Types of Carbon fibers based on final product:

The end products of carbon fibers are divided in to different types based on the shape, length, alignment and type of winding of fibers as they have major impact on the properties of composites.

S. No.	Туре	Specifications	
1	Filament	Yarn consisting of twisted or	
1		untwisted fibers	

Table 6:	Types	of carbon	Fibers	products[26]
1 4010 0.	1) P 0 0	or caroon	1 10010	producto[20]

2	Tow	Untwisted yarn consisting of numerous fibers
3	Staple Yarn	Spinning of staples to make a yarn
4	Woven Fabric	Woven sheet made of filament or staple yarn
5	Braid	Braided yarn made of filament or tow
6	Chopped Fibers	Fibers are chopped to desired length
7	Milled	Powder made by milling fibers in a ball mill
8	Felt Mat	Mat made by stacking layers of staple

1.7.2.4 Properties and Characteristics of carbon fibers:

Table 7 shows some of the properties of Carbon Fibers:

S. No.	Properties	Unit	Value	
1	Density g/cm3		1.70-1.9	
2	Elastic Modulus GPa		≤7	
3	Fracture	$MPa(m)^{1/2}$	<4.8	
C C	Toughness			
4	Thermal	W/m/K	54	
•	Conductivity	,,,, 11 / 1		

Some of the characteristics of carbon fibers are as under which make them ideal reinforcements for ablative composites[28]:

1. Good creep resistance

- 2. Resistant to chemical environment
- 3. High heat of ablation
- 4. High heat capacity
- 5. Fire resistant
- 6. Low co-efficient of thermal expansion
- 7. Rigid

Chapter 2: Literature Review

Cho, et al.[29] observed the microscopic behavior of ablation in carbon phenolic composites. Ablation was done by using Oxygen and acetylene flame in ratio 4:1 and microscopic features were studied by using SEM. The results showed that the matrix in outer surface of sample carbonizes at high temperature and cracks due to thermo mechanical effects but the matrix in subsurface decomposes and forms char which provides structural integrity. The cross section of fibers showed that the shape of the fibers is needle like and the diameter is reduced significantly after ablation. This is because of the reason that micro cracks initiate at most influenced areas and these micro cracks propagates due to thermo mechanical effect and with the passage of time these areas become more sharpened giving fibers needle like shape. The change in fiber diameter depends on the distance from hole that is created by flame. The decrease in fiber diameter will be greater in near hole and smaller away from hole.

Winya, et al.[30] studied the factors that affect the ablation rate. Curing time, curing temperature and amount of matrix (Phenolic resin) were the parameters varied to study the effect on ablation rate. Samples were made by using hand lay-up method with altering wt. % of phenolic resin, curing temperature and curing time. Ablation rate was measured by using oxyacetylene flame. The results showed that as the parameters increase, ablation rate decreases but there is a significant drop in ablation rate if curing time is increased as compared to other factors.

Park and Kang[31] compared the effect of using low temperature carbon fibers (LTCF) and high temperature carbon fibers (HTCF) on ablative properties of carbon fibers/phenolic composites in 2001.LTCFs were produced by "continuous carbonization of PAN fibers at 1100°C. It was learnt that by using LTCFs thermal conductivity is decreased by 35%. Ablation test was also performed using arc plasma touch flame which revealed that the erosion rate increases by 30% if LTCFs are used. Hence thermal insulation was improved but the ablative resistance was decreased by the use of LTCFs in ablative composites.

Similarly, Lee and Jong[32] studied the effect of different pressures on the ablative behavior of carbon fiber reinforced composites (CFRC). The composites were prepared at three pressures: 0.1, 10, 90MPa. SEM results showed that morphology of the samples changed from lamellar to coarse grains structure due to increase in viscosity of matrix as the pressure was increased. Ablation results were also reported by using arc-plasma torch which suggests that the ablation rate decreases as the pressure is increased.

Choi, et al.[33] studied that the mechanical and electrical properties of carbon fiber/phenolic resin composites were altered by the addition of coupling agents in 2000. Glutaric dialdehyde was used as a coupling agent and it was proved that the composite containing the coupling agent had better mechanical properties as compared to other samples with the same amount of carbon fibers. It was also revealed that with the addition of coupling agent the dispersion of short carbon fibers in phenolic resin can be improved which is very effective in compression molding. Also the adhesion of fibers with resin improved due to formation of chemical bonding at the interface. This was also supported by Yuan, et al.[34] in 2012 that surface modification improves the adhesion of carbon fibers with phenolic resin by forming chemical bonds and its mechanical properties are improved.

In 2002, Patton, et al.[35] studied the mechanical and ablation properties of vapor grown carbon fibers/phenolic matrix composite. Three samples were made by using different composition (wt. %) of vapor grown carbon fibers (VGCF) i.e. 30%, 40% and 40% ball milled VGCF and another sample was made which contained 45/5% VGCF and woven Rayon based carbon fibers. Ablation testing was done by using Plasma torch and the results were compared to CF/phenolic composite (MX-4926: NASA's standard nozzle material). The results showed that by using VGCF thermal insulation can be improved but composites formed a very thin char layer due to low carbon content for the case of TGCF which resulted in greater erosion rates.

Firouzmanesh and Azar[36] in 2003 compared the ablative behavior and thermal stability by using different types of carbon fabrics on carbon/epoxy-novolac composites for rocket nozzles. Three types of carbon plain weave with 100cm width were used with 0.16mm, 0.31mm and 0.32mm thickness and 125g/m², 240g/m² and 275g/m² density

respectively. The results showed that the carbon fabric with the density $240g/m^2$ gives the best thermal insulation and are stable as well. This is due to the fact that as the density of the reinforcements is very high thermal conduction is high but if the density is very low, stability of the composite is affected. That is why one has to make a compromise between stability and insulation of ablative material.

Park, et al.[37] compared the effect of spun and filament yarn type carbon fabric on the thermal and ablative properties of carbon/phenolic composites. Carbon fabrics were heat treated to 1100°C and then used as reinforcement in phenolic resin. Shear strength of staple or spun carbon fabric/phenolic composite sample is greater than filament carbon fabric/phenolic composite by 50%. This increase in adhesion strength is due to anchoring of protruded fibers to phenolic matrix in interfacial region and resist against shear force. Thermal conductivity along the laminar plane (longitudinal direction) and perpendicular to laminar plane (transverse direction) was also evaluated. Longitudinal thermal conductivity of staple carbon/phenolic composite is 7% lower than filament carbon/phenolic composite because filament of staple carbon/phenolic composite increases to filament carbon/phenolic composite value. The increased conductivity is because spun carbon/phenolic composite has protruded fibers and lesser matrix rich region. The ablation test was carried by using plasma torch due to which char layer with very good integrity was formed for both samples. The results indicated that the insulation index and time to raise back face temperature of spun fiber/phenolic composite sample was greater than filament fiber/phenolic composite. Also the erosion rate of latter was greater compared to former.

In 2007, Srebrenkoska, et al.[38] compared the mechanical and ablative properties of short carbon fiber/phenolic composites by using different carbon fiber to matrix ratio and length of carbon fibers. Test samples were prepared in 25/75, 45/55, 57/43, 67/33 and 75/25 carbon fibers to matrix ratios with length of 25 and 50mm fibers. Mechanical strength was measured by impact test. The results showed that the samples with longer fibers showed greater impact strength however samples with higher content of fibers become rigid and show lower values of impact strength. The samples with length of 50mm and fiber ration of 57% showed best impact strength. The thermal stability of the

test specimens was calculated by weight loss during thermal degradation. The specimens with higher concentration of fibers showed lower weight loss as compared to samples with lower concentration of fibers. Also samples with longer fibers showed lower weight loss as compared with samples with same ratio of fibers but containing smaller fibers.

In 2008, Sulaiman, et al.[39] studied the effect of adding hardener along with carbon fibers in different percentages on mechanical properties of fiber/phenolic composites. The hardener used was hexamine and was varied from 5-15%. The results showed that composites with 15% hardener showed the best mechanical properties due to increased crosslinking between hardener and matrix.

In 2010, Pulci, et al.[40] manufactured and tested the mechanical and ablative properties of carbon/phenolic composite. Two different composite samples were made using resole type phenolic resin with graphite felt and graphite foam. Four point bent test was performed to check the mechanical properties of carbon/phenolic composites. Felt/resole composite showed much higher flexural strength than foam/resole composite because the felt absorbs a lot of strain before deforming compared to foam which shows brittle failure. Thermo physical properties revealed that conductivity of felt/resole composites was higher than foam/resole composite. Ablation test was done by using oxyacetylene flame. These results also complimented the former results showing better insulation properties of felt/resole composite than foam/resole composite. Also felt/resole composite samples produced char layer with better integrity.

Farhan [41], also in 2010, worked on carbon/carbon composites. He studied the effect of altering density and fiber orientation on ablation behavior of the composites. Three samples were made with same density $(1.77g/cm^3)$ and different orientation of fibers (carbon fiber rods 30° to flame direction, non-woven carbon fibers 0° to flame direction) and non-woven carbon fabric 90° and needled carbon fibers 0° to flame direction) while the other three were made with same orientation (non-woven needled carbon fibers 0° to flame direction) and different densities (1.77, 1.83, 1.85 g/cm³). The samples were exposed to oxyacetylene flame according to E285 standard for ablation test. While comparing the results of the sample with different orientations, the sample with carbon rods 30° to flame direction showed highest erosion rate compared to other two because

the matrix was severely damaged by flame and pits were formed as seen by SEM. The fibers in second sample changed to needle shape and diameter of fibers was significantly reduced. In third sample, for a certain period of time needled fibers became sharper in shape but as micro cracks increased, the cone shape broke and blunt shape was formed due to which erosion rate reduced. While comparing the samples with different densities, the sample with the highest density showed greatest ablation resistance. SEM results also suggested that lower open porosity was formed in sample with density 1.850 g/cm³ and orientation non-woven carbon fabric 90° and needled carbon fibers 0° to flame direction due to which it showed lowest erosion rate i.e. 0.113mm/s.

In 2012, Winya, et al.[42] studied the effects of using fiber glass and synthetic fibers on the mechanical, ablation and thermal properties of phenolic based composite used in rocket motor insulation. It was discovered that the mechanical strength and ablation properties of phenolic/synthetic fiber composite are better than phenolic/fiber glass composite by 50%. The ablation rate of phenolic/synthetic fibers is less than 0.06mm/s while for that of phenolic/fiberglass is 0.14%. It was also suggested that by the use of synthetic fibers the insulation of rocket motor nozzle can be improved.

Hong, et al.[43] also studied the ablation behavior of novel 3-D phenolic impregnated carbon fabric composites in 2012. Phenolic impregnated carbon ablator (PICA) is a low dense composite (density ranging from 0.35 to 0.70 g/cm³) with very high ablation resistance due to high porosity compared to conventional carbon/phenolic composites (density ranging from 1.2 to 1.7g/cm³). Ablation rate was measured by using oxyacetylene flame and the surface and sub-surface temperatures were measured by using thermocouples. The results revealed that temperature was the highest at the surface (around 1800°C) and in-depth temperature was lower and increased gradually as compared to surface temperature. The linear ablation rate (in the range from 0.019 to 0.036mm/s) was also lower than conventional carbon phenolic composites.

Another study was done in 2012 by Natali and Monti[44] to compare the effects of using carbon black and multi walled nanotubes (MWNT) on ablative properties of phenolic composites. Both Nano fillers were used in 50wt. % with phenolic resin. Ablative test was done by using oxyacetylene torch that showed erosion rate of sample containing

MWNT was greater than the sample containing carbon black. The test resulted in formation of thin char layer from which flakes of burnt material could be peeled off for MWNT/phenolic composite samples. While thick char layer with better integrity was produced in case of using carbon black as nano filler.

Park, et al.[45] studied thermal conductivity and ablation properties of carbon/phenolic composites by adding carbon nanotubes and differently oriented carbon fiber reinforcements. Carbon fibers (CF) and carbon nanotubes (CNTs) were used in 30 vol% and 0.5wt% in concentration respectively. Three samples were made: randomly chopped carbon fiber/phenolic composite (RCFPC), woven carbon fiber mat reinforced phenolic composite (MCFPC) and a woven carbon fiber mat with CNTs/reinforced phenolic composite (MCF/CNTPC). Thermal conductivity was measured by thermal conductivity analyzer (Mathis TCi, C-Therm Technologies limited) which showed conductivity values for MCFPC were greater than RCFPC due to the fact that MCFPC had continuous carbon fibers in its structure. While comparing the conductivity for MCFPC and MCF/CNTPC, MCF/CNTPC showed greater values due to large surface area. Ablation test was done by using oxygen-kerosene (1:1) flame torch. The evaluation showed that erosion rate is highest for RCFPC, intermediate for MCFPC and lowest for MCF/CNTPC. The temperature on back wall and time to burn through also supports the above results that it takes longer time to raise the back wall temperature for MCF/CNTPC as compared to other two as the heat energy is transferred and diffused away from the flame.

In 2013 Natali and Rallini[12] also discussed the effects of using different fibrous reinforcements on EPDM based shielding materials which are used for rocket motors. The reinforcements used were aramid fibers and silica fibers. The effects of using Kynol fibers which are phenolic based reinforcements were also evaluated. It was discovered that EPDM/Kynol systems produce char with very small dimensional change and has very good adhesion because its long chains embed in virgin material. EPDM/aramid system produced char of good quality but with high dimensional change over virgin material while EPDM/silica systems produce char layer of very small quantity. Since the

efficiency of ablative system depends on the quality and thickness of char layer EPDM/silica systems are not recommended for rocket motors.

Eslami and Yazdani in 2015,[46] introduced multi walled carbon nanotubes (MWCNTs) in addition to carbon fibers as a reinforcement to phenolic-based composites. The results showed that the thermal stability of the composite increases as the wt. % or the content of MWCNTs is increased (wt. $\% \le 1$) but decreases if wt. % of MWCNTs is increased above 1%. The Ablation rate was reduced by 52% due to the presence of MWCNTs. Morphological characterization revealed that cracks or voids are not formed in char layer due to the presence of MWCNTs if the content is below 1% but as the content increases more than 1% poor dispersion of CNTs in polymer matrix leads to poor thermal insulation.

In 2015, Fonseca, et al.[47] studied the effect of fiber orientation relative to plasma flow direction in ablation process. In their study samples with three different types of fiber orientations were analyzed: 0° , 68° and 48° and 90° to plasma flow direction. The results showed that for sample having fibers parallel to plasma flow direction, the internal conduction of heat was very low because most of the heat transfer was taking place through resin. Also the gases formed during pyrolysis are trapped by closely packed fibers which result in mechanical erosion and results in non-uniform ablation and great amount of mass loss. It was observed that the mass loss was lesser for samples which have fibers oriented at certain angle to plasma flow direction. For samples containing fibers perpendicular to direction of plasma flow, ablation speed is very low which results in an increase in heat of ablation and lower ablation rates. This ablation rate tend to increase as the angle of fibers tend to decrease from being perpendicular (or 90°) to being parallel (or 0°) to plasma flow direction. The plane perpendicular to fibers has limited mechanical properties leading to material removal and ultimately failure due to stress concentration during ablation.

Chapter 3: Experimental Procedures

3.1 Material and composite:

As mentioned before, phenolic resin is chosen as a matrix due to its high char yield, less amount of formation of volatiles and good elastic properties at low temperatures while carbon fibers are chosen as reinforcements due to high heat capacity, high heat of ablation and good mechanical properties.

3.1.1 Fibers used:

Several types of PAN based carbon fibers were used from different sources for the formation of different types of composite samples. The information about the type along with some properties is given below:

3.1.1.1 Type A:

3K PAN carbon fabric was obtained from this source with the following properties:

S. No.	Properties		Unit	Value
1.	Density		g/cm ³	1.83
2.	Carbon content		%	98%
3	Breaking	Warp	N	2468
	load	Weft		2058

Table 8: Fiber Properties of Type A

3.1.1.2 Type B:

Two different batches (B and C) of 3K PAN based carbon fabric were obtained from the same source with the properties given in Table 9:

S. No.	Properties		Unit	Value
1.	Density		g/cm ³	1.78
2.	Carbon content		%	96
3	Breaking	Warp	N	1993
	load	Weft		2257

Table 9: Fiber properties of Type B and C

3.1.1.3 Type D:

3K carbon cloth with the following properties was obtained from this source:

S. No.	Properties		Unit	Value
1.	Density		g/cm ³	1.68
2.	Carbon content		%	92%
3.	Breaking	Warp	Ν	1853
5.	load	Weft	- 1	1497

Table 10: Fiber properties of Type D

3.1.2 Resin used:

Phenolic Resin was obtained from Polymer Industry Gujranwala and was used due to aforementioned properties.

3.1.3 Composite manufacturing:

Firstly prepregs are formed through impregnation process in which reinforcing cloths or fibers are impregnated or wet in phenolic resin in an impregnation plant. Carbon cloth or fibers are preheated in a furnace to about 150°C for 10 min, then impregnated in phenolic resin for a time greater than 30 seconds and the wet cloth is precured in a precuring furnace to a temperature between 80-120 °C for 5-15 minutes. Prepregs can be stored below room temperature for a month. Prepegs for this research were made using Figure 8:



Figure 8: Method for prepeg formation for research

Prepeg tape is then winded on to specific rotating mandrels for manufacturing of various ablative composite products. Prepreg tape is wrapped tightly with appropriate tension and roller pressure to ensure wrinkle free, straight and smooth winding. Parameters used for tape winding for this research are as follow:

S. No.	Parameters	Values
1.	Tape tension	For $40mm = 200 \pm 30$ N
2.	Roller Temperature	35-80°C
3.	Roller pressure	1000-18000Pa
4.	Spindle speed	7-15rpm

Table 11: Parameters used for tape winding for research

Cross-linking takes place during a chemical process which is called curing. Hydroclave curing process uses water as medium to transmit heat and pressure to cure the semi cured parts. The composite parts attain required density, mechanical and thermal properties through curing process. Parameters used during curing and curing sequence for carbon fiber reinforced phenolic composite used for this research is shown in Table 12 and Figure 9 respectively:

Table 12: Parameters for Curing of Samples for this research

S. No.	Parameters	Values
1.	Working Pressure	4MPa
2.	Vacuum pressure	-0.094MPa
3.	Temperature Rising Rate	12°C/hr



Figure 9: Curing Sequence of CF reinforced phenolic composite used for the research Cure sequence is followed to allow for viscosity changes and the forces created in composite. Pressure is applied for consolidation of carbon fabrics. Temperature is raised to a level below gelation point steadily. The sample is held for two hour at this temperature for gelation. When the gelation process is completed the sample is rigid but has not developed any strength. The strength of the sample is improved due to additional cross linking by holding it at curing temperature. However the rate of crosslinking is slow at cure temperature, temperature is further raised to advance the cure. After holding the sample again at this temperature, the part is cooled.[48]

3.2 Scanning Electron microcopy (SEM):

Tescan Vega3 was used to scan carbon fibers for any defects, porosity, sizing and adhesion of fibers to phenolic matrix.



Figure 10: SEM used

3.3 Ablation Measurements:

Chinese standard GJB323-96A was followed to measure linear and mass ablation rates of the sample. According to this standard, sample of size 30mm diameter and 10mm thickness is heated by oxy acetylene torch at one face and temperature change is measured from the back face of the sample.



Figure 11: Size of sample as per standard GJB323-96A

3.4 Data recording:

Back face temperature was measured by using K type thermo couple and recorded manually by four channel input TECPEL thermometer data logger (RS-232 Thermo log)

which records with the accuracy of $\pm 1^{\circ}$ C and has a sampling rate of 3 seconds per testing circle.



Figure 12: Data logger used

3.5 Procedure:

To test CF reinforced phenolic composite samples for back face temperature and erosion rate measurement, a fire brick was designed to place the ablator in the center of the brick. A hole was formed in brick through which thermocouple can pass to sense the temperature. The brick with then placed on the stand. Oxy acetylene torch was held in a stand in such a way that the distance between the tip of torch and sample is 10mm and torch tip is exactly 90° to surface of the sample.



Figure 13: Ablation Set-up designed for experimentation



Figure 14: Fire brick for holding Sample



Figure 15: Setup for Ablation Test

The pressure of Oxygen is kept 0.4MPa (58psi) and that of acetylene is 0.095MPa (13.78psi). The mixture of gases is ignited and the flame is exposed to the sample for 60 seconds. Back face temperature readings were recorded manually and graphs were made using Microsoft excel representing the transition of back face temperature as a function of time.



Figure 16: Experiment in Progress

Linear and mass ablation rates of the samples were also calculated by use of following relations:

Mass ablation rate M (mg/s) = $(m_0-m_t)/t$

Where,

 $m_o = mass of sample before ablation$

 $m_t = mass of sample after ablation$

t = time of test

Linear Ablation rate L (mm/s) = $(l_0-l_t)/t$

Where,

 $l_o =$ thickness of sample before ablation

 l_t = thickness of sample after ablation

t = time of test

Chapter 4: Results and Analysis:

4.1 SEM:

SEM results of fibers show layer of sizing on fibers but no evident defects on the surface of fibers. Elemental Dispersion Spectroscopy (EDS) analysis showed that type A has 98% Carbon; type B has 96% C while type C has 92% carbon. Trace amount of oxygen was also present.



(a) Figure 17: SEM images of fibers (a) Type A (b) Type B (c) Type D

4.2 Ablation Test:

The back face temperature readings were collected using data logger. Back face temperature for every composite, two samples each, was recorded and the readings along with graphs are given:

4.2.1 Ablation test for Composite A

Time (a)	Back face temperature of	Back face temperature of	
Time (s)	Sample 1 (°C)	Sample 2 (°C)	
0	31.3	32.3	

Table 13: Back face temperature readings for Composite A

31.3	32.3
31.3	32.3
31.3	32.3
31.4	32.3
31.4	32.4
31.4	32.4
31.4	32.4
31.4	32.5
31.6	32.7
31.6	32.7
31.6	33.1
32	33.6
32	34.3
32.7	34.3
32.7	34.3
33.6	35.3
35	35.3
35	36.4
36.8	36.4
39	38
39	39.8
41.6	39.8
41.6	39.8
44.5	41.8
44.5	44
47.8	46.4
51.4	49
55.6	51.9
58.9	55.1
63.6	58.6
	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$

Graph representing the data given in Table 13 is shown below:



Figure 18: Graph representing Temperature-Time plot for Composite A

The surface of the samples before and after ablation is shown in Figure 19. A depression on the surface of the sample can be seen on the sample after ablation which represents mass loss during experiment.



Figure 19: Surface of Composite A before and after ablation

4.2.2 Ablation test for composite B

Time (g)	Back face temperature of	Back face Temperature of	
Time (s)	Sample 1 (°C)	Sample 2 (°C)	
0	30.4	28.9	
2	30.4	28.9	
4	30.4	28.9	
6	30.4	28.9	
8	30.4	29.1	
10	30.4	29.1	
12	30.4	29.1	
14	30.4	29.4	
16	30.7	29.4	
18	30.7	29.4	
20	30.9	29.8	
22	30.9	29.8	
24	30.9	30.5	
26	31.4	30.5	
28	31.4	31.4	
30	32.1	31.4	
32	32.1	32.5	
34	32.1	32.5	
36	32.9	33.9	
38	32.9	35.9	
40	34	37.4	
42	35.4	37.4	
44	37	39.5	
46	38.9	39.5	

Table 14: Back face temperature readings for Composite B

48	40.7	41.7
50	42.5	41.7
52	44.5	43.6
54	46.4	44.8
56	-	-
58	-	-
60	-	-

Graph for Table 14 is given in Figure 20:



Figure 20: Graph representing Temperature-Time plot for Composite B Table 14 shows that the experiment did not complete its course and the samples of composite B were destroyed after 54 seconds. It can be seen in Figure 21 that the layers of the sample separated after 54 seconds.



Figure 21: Surface of composite B before and after Ablation

4.2.3 Ablation test for Composite C

T:	Back Face Temperature of	Back Face temperature of
Time (s)	Sample 1 (°C)	Sample 2(°C)
0	32.7	33.8
2	32.7	33.8
4	32.7	33.8
6	32.7	33.8
8	32.7	33.8
10	32.7	34.2
12	32.8	34.2
14	32.9	34.2
16	33.1	34.5
18	33.1	34.5
20	33.5	35
22	34.1	35
24	34.1	35.5
26	34.9	35.5

Table 15: Back face temperature readings for Composite C

28	34.9	37.5
30	36.1	37.5
32	36.1	38.9
34	37.8	38.9
36	39.8	40.5
38	41.8	40.5
40	44.3	44
42	47	46.8
44	47	46.8
46	50.1	49.8
48	50.1	50.2
50	53.3	54.2
52	53.3	54.2
54	57	56
56	62.8	61.9
58	66.2	66.2
60	75.4	73.4
1		1

Graph representing data of Table 15 is shown in Figure 22:



Figure 22: Graph representing Temperature-Time plot for Composite C

4.2.4 Ablation test for Composite D

	Back Face Temperature of	Back Face temperature of
Time (s)	Sample 1 (°C)	Sample 2(°C)
0	33.3	31.4
2	33.3	31.4
4	33.3	31.4
6	33.3	31.4
8	33.9	33.3
10	33.9	33.3
12	33.9	33.3
14	34.8	34.1
16	34.8	34.1
18	34.8	34.5
20	36.9	34.5
22	36.9	34.5

Table 16: Back Face temperature readings for Composite D

24	38.1	35.2
26	38.1	36.4
28	39	38.3
30	39	40.8
32	40.1	40.8
34	40.1	42.6
36	41.9	42.6
38	41.9	45.6
40	44	45.8
42	44	47.4
44	44	47.4
46	46.5	50.6
48	46.5	56.5
50	49.5	61.2
52	53.1	65.6
54	57.3	66.7
56	62	69.7
58	67.9	73.7
60	75.2	79.9

Graph representing Table 16 is given in Figure 23:



Figure 23: Graph representing Temperature-Time plot for Composite D

4.3 Comparison and analysis of Ablation test:

The Table 17 represents the comparison in average of change in back face temperature of composites A through D for 60 seconds with two second interval.

Time (s)	Composite	Composite B	Composite C	Composite
	A(°C)	(°C)	(°C)	D(°C)
0	31.8	29.65	33.25	32.35
2	31.8	29.65	33.25	32.35
4	31.8	29.65	33.25	32.35
6	31.8	29.65	33.25	32.35
8	31.85	29.75	33.25	33.6
10	31.85	29.75	33.45	33.6
12	31.9	29.75	33.5	33.6
14	31.9	29.9	33.55	34.45
16	31.95	30.05	33.8	34.45

Table 17: Comparison in average readings of back face Temperature

18	32.15	30.05	33.8	34.65
20	32.15	30.35	34.25	35.7
22	32.35	30.35	34.55	35.7
24	32.8	30.7	34.8	36.65
26	33.15	30.95	35.2	37.25
28	33.5	31.4	36.2	38.65
30	33.5	31.75	36.8	39.9
32	34.45	32.3	37.5	40.45
34	35.15	32.3	38.35	41.35
36	35.7	33.4	40.15	42.25
38	36.6	34.4	41.15	43.75
40	38.5	35.7	44.15	44.9
42	39.4	36.4	46.9	45.7
44	40.7	38.25	46.9	45.7
46	40.7	39.2	49.95	48.55
48	43.15	41.2	50.15	51.5
50	44.25	42.1	53.75	55.35
52	47.1	44.05	53.75	59.35
54	50.2	45.6	56.5	62
56	53.75	-	62.35	65.85
58	57	-	66.2	70.8
60	61.1	-	74.4	77.55



The graph comparing the results of ablation test shown in Table 17 is given below:

Figure 24: Graph representing comparison of Back face temperature readings

The results show that the back face temperature of every sample increases with time. At the start of the experiment, the change in the back face temperature readings is very small. This is due to the fact that heat is not effectively transferred across the thickness of fibers. It dissipates along the direction of the fibers and do not concentrate on a source. This region where the slope of the curve is very small is called the safe zone for an ablator. The thickness of the ablation composite is varied according to this zone for application purposes.

While comparing the results of composite B and C, it can be seen that although they have same properties but their results differ a lot. Moreover, layers of samples of composite B were separated after 54 seconds as can be seen in Figure 21. The separation of layers shows poor stability of composite B which may happen due to un-noticed deviation from processing parameters. The manufacturing process of high performance fiber reinforced composite is a complex process as it has many variable parameters which are constantly checked and recorded at each step. Difference from a single parameter can alter the results. Following is the list of deviations that may have happened that could result in poor stability of Composite B:

- 1. Prepegs are required to store at 25°C. If the temperature is altered during storage that may result in poor stability of composite B.
- 2. Winding of carbon fabric from spools on to mandrels under specific tension and pressure is a continuous process. If the spool ends during winding, tension and pressure alters which may result in poor stability.
- 3. If the curing of the composite is not ensured to its core, which may result in poor stability of Composite B.

While comparing the results of composites A, C and D, slope of the curve for composite A is very small which shows it has greater ablation resistance. This can be explained by carbon content of carbon fibers. Table 8, Table 9 and Table 10 show Type A fibers have the largest amount of carbon content which means composite A will have maximum ablation resistance because of high heat capacity of carbon.

4.4 Comparison and analysis of Ablation rates:

Ablation rate was calculated by using the relation given at the end of chapter 3. The results are as follow:

Composite Type	Measurements	Sample 1	Sample 2	Average Mass Ablation Rate (mgs ⁻¹)
	m _o (mg)	10.656	10.935	
Composite A	m _t (mg)	7.773	8.364	0.045
	M (mgs ⁻¹)	0.048	0.043	
Composite B	m _o (mg)	10.581	10.559	
	m _t (mg)	7.04	7.38	0.056
	M (mgs ⁻¹)	0.059	0.053	
Composite C	m _o (mg)	10.667	10.704	
	m _t (mg)	6.123	7.524	0.052
	M (mgs ⁻¹)	0.050	0.053	

Table 18: Comparison between Mass Ablation Rates

	m _o (mg)	10.573	10.627	
Composite D	m _t (mg)	4.198	6.849	0.062
	M (mgs ⁻¹)	0.061	0.063	

Linear ablation rate was also calculated by using the relation given at the end of chapter 3.

Composite Type	Measurements	Sample 1	Sample 2	Average Linear Ablation Rate (mms ⁻¹)
	l _o (mm)	10.24	10.11	
Composite A	l _t (mm)	7.24	6.93	0.052
	L (mms ⁻¹)	0.050	0.053	
	l _o (mm)	10.30	10.33	
Composite B	l _t (mm)	7.24	7.09	0.0525
	L (mms ⁻¹)	0.051	0.054	
	l _o (mm)	10.28	10.32	
Composite C	l _t (mm)	5.25	6.90	0.0565
	L (mms ⁻¹)	0.056	0.057	
Composite D	l _o (mm)	10.28	10.27	
	l _t (mm)	3.56	6.35	0.0645
	L (mms ⁻¹)	0.064	0.065	

Table 19: Comparison between Linear Ablation Rates

A comparison between the results is shown in Figure 25:



Figure 25: Graph comparing the ablation rates of composites

The results show that the linear and mass ablation rates of the samples are equal. While comparing the ablation rates of A, B, C and D, ablation rate of composite A is less than Composite B, C and D. This can be explained by the difference in densities between composite A, B, C and D. The density of type A carbon fabric is greater (1.83g/cm³) comparing to fiber type B and D (Density= 1.70, 1.68 g/cm³ respectively). Greater density of carbon fibers will mean that open porosity is lower which will result in retention of char layer resulting lower ablation rates which can be seen in Figure 25.[41]

Conclusions:

- 1. SEM of the fibers was done and the images showed a layer of sizing on the fibers while no defects on the surface were observed.
- 2. Ablation test was carried by using standard GJB323-96A. It was observed that composite A had more resistance to ablation as compared to composite B, C and D because it had higher carbon content as compared to other three types.
- 3. Composite B showed results similar to composite A but it was unstable and did not complete the test. This difference from the trend aroused due to change from processing parameters.
- 4. Ablation rate of composite A was 11% less than composite C and 28% less than composite D because of higher density of type A fibers. Composite B had 19% lower ablation rate than composite A, however, was unstable and its laminates separated before completion of the test.
- 5. The results were compared to the results of samples test carried at another facility which proved the reliability of the locally developed set-up.

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