

Experimental Study on Pan Based Composites with Multi Filler Material

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ABSTRACT

The experimental work done on studying the characterization of PAN based composites with different types of fillers is investigated. Phenolic composite materials are used into wide range of components to supply a diverse and fragmented commercial base that includes customers in aerospace, aircraft, defense, marine etc. The PAN based laminates are prepared with phenolic resin by hand layup process cured under temperature and pressure. The test samples are prepared by ASTM standard and subjected to testing. Comparison of Mechanical properties of the laminates is tabulated. Generally PAN composites are used for high thermal stability used in aerospace industry. The experimental work carried out to study back wall Temperature through Oxy-acetylene Torch test. The study result reveals that PAN based composite laminates with multi fillers exhibits better thermal protection than single filler laminate.

INTRODUCTION

Composite materials are engineered systems made up of two or more distinct components that are combined in a design that imparts complimentary properties to the compounded product^[1]. Phenolic composites are commonly used in high temperature environments and wherever they can be applied to protect high temperature environments^[2]. These crucial applications generally require extensive development and testing.

Carbon phenolic composites have one of the most distinctive features of carbon based composites is the capability to withstand extremely high temperatures for relatively long period of time. Phenolics are selected as the resin of choice for thermal applications because of their high carbon yield with organic resin system^[2,3]. The carbonized such as Rayon, Polyacrylonitrile (PAN) prepegs that are made with these components can be thermally converted into a carbon phenolic composite depending on the process.

Generally a re-entry vehicle structure in aerospace is made up of Carbon Epoxy (CE) and Carbon Phenolic (CP) shells. The CE shells withstand for structural integrity and CP shells withstand for high thermal applications^[4,5]. The phenolic composites quality offers the capability to provide thermal protection, light weight composition. The phenolic resins can withstand high temperature for short duration^[5].

The major objective of this research is to develop the PAN/ phenolic based laminates and to study the mechanical properties and transition or back wall temperature along with ablation properties^[6]. The process involves PAN fabric impregnated with phenolic resin, and the laminates are prepared by hand layup process. The laminates are cured in autoclave under temperature, pressure and vacuum. Test samples are prepared as per ASTM standard for testing mechanical properties.

Selection of materials

The following materials are selected for this study. In the present work, the mechanical and thermal characteristic properties of Phenolic based composites with zirconium diboride & silicon carbide fillers and carbon as reinforcement will be studied (**Table 1**).

Table 1: Details of raw materials.

Sl. No	Raw material	Grade
A	Reinforcement	PAN Carbon fiber T-300

B	Phenolic Resin	ABRON-PR 100 (WS)
C	Filler material-I	Silicon Carbide Powder
D	Filler material-II	Zirconium diboride

Carbon fabric

Poly Acrylo Nitrile (PAN) based carbon fibers and their composites, particularly those with Polymeric matrices, have become the dominant advanced composite materials for aerospace application due to their high specific strength, stiffness and low weight. PAN carbon fabric is very expensive, it is amorphous material for ablative purpose and is having vast applications in aerospace industry and hence it is selected.

Phenolic resin

Phenolic resin is the conventional matrix material which is used for aerospace applications to withstand high temperatures. Phenolic resin is the oldest synthetic polymers used commercially available of ABRON-PR100 (WS) Phenolic resin to meet the requirement for low smoke and toxicity^[2,4,5]. Hence it is selected.

Filler material-I

Generally fillers in the matrix gives rise to increase in load withstanding capability, reduce coefficient of friction, improved wear resistance and improved thermal properties. The Silicon Carbide (SiC) of 220 mesh as filler material to increase the properties of the laminate. The SiC filler is having the average particle size of 0.5 microns. Grains of silicon carbide can be bonded together by sintering to form very hard ceramics that are widely used in aerospace applications. Hence it is selected.

Filler material-II

Zirconium diboride (ZrB_2) is a highly covalent refractory ceramic material with a hexagonal crystal structure. Zirconium diboride is having the average particle size of 1.5 microns. Zirconium diboride is an ultra-high temperature ceramic with a high melting point along with its relatively low density and good high temperature strength used in aerospace applications. Hence it is selected.

EXPERIMENTAL PROCEDURE

In this experimental study, the following types of laminates were considered, there are (a) Laminate 1 is Carbon-Phenolic without filler, (b) Laminate 2 is Carbon-Phenolic with 20% SiC filler, (c) Laminate 3 is Carbon-Phenolic with 15%Zrb2-Sic filler.

Preparation of laminate

PAN Carbon fabric plane wave bi-woven were used as fiber reinforcement. Phenolic resin with specification mentioned as above was used as the matrix material. The composite laminate was prepared by using the hand layup technique. A mould can be used for this process. The mould surface is to be cleaned with some solution such as acetone and release agent like wax. Petroleum jelly is applied for easy removal of laminate from the mould. The reinforcing material wave bi-woven PAN carbon fabric cut into required size (250 x 250 x 3 mm) and laid on flat surface of the mould. The Phenolic resin is mixed as per weight proportions with PAN carbon fabric material varied fiber volume fraction is spread evenly on the reinforcing fiber surface. The resin is squeezed evenly on the surface by roller and compresses the PAN carbon fabric thoroughly with the help of roller. The reinforcing laminates are stacked one above the other to the required thickness of the laminate. The laminates are cured in autoclave by a cure cycle of Temperature Vs Time under vacuum and pressure.

Designation of laminates

Type 1 Laminate

Carbon-Phenolic without filler designated as L1

Type 2 Laminate

Carbon-Phenolic with 20% SiC filler designated as L2

Type 3 Laminate

Carbon-Phenolic with 15% Zrb2-Sic filler designated as L3

Preparation of test specimen

The laminates are taken out of the autoclave and are to be cut into required sizes as per standard for Mechanical testing and others laminates for oxy-acetylene test were cut by a tile saw. Two specimens were cut in each category stated with and without fillers. The J type thermocouple (Fe-Cu) is bonded in center of the test specimen from the rear surface with help of the high temperature adhesive cerma bond and subjected to Oxy-acetylene flame test.

Test methods

The Mechanical properties include the determination of Flexural Strength and Inter Laminar Shear Strength (ILSS).

The tensile strength is performed on the flat surfaces. The tested specimens have dimensions of length 250mm width 25mm and thickness 3.0 mm. These specimens are tested under Universal Testing machine (UTM) (Instron) as per ASTM D3039 Test standard.

Flexural and inter laminar strength

The short beam shear (SBS) test performed on the prepared laminates at room temperature to evaluate the value of flexural strength. The flexural strength has been conducted as per ASTM D790. The dimensions of each test specimen were 130mm length 25mm width and 3.0mm thickness as shown in **Figure 1**. Same identical test samples were tested for calculating Inter laminar shear strength as per test method ASTM specification D2344 as shown in **Figure 2**.



Figure 1: Flexural strength specimen.



Figure 2: ILSS specimen.

The flexural strength of any composite specimen is determined by the following “equation-1”

$$\sigma_f = 3PL / (2bh^2) \quad (1)$$

Where, σ_f -Stress in the outer fibers at mid point (Mpa), P -Maximum load (N), L-Span length of specimen (mm), b-Width of the specimen (mm), h-Thickness of the specimen (mm).

The ILSS were calculated by following “equation-2”

$$ILSS = 3F/4bt$$

(2)

Where, ILSS Inter laminar shear strength (MPa), F –Maximum load (N), b –Width of the specimen (mm), t –Thickness of the specimen (mm).

Testing of specimen on oxy-acetylene test bed

The oxyacetylene test bed (OTB) is a small scale experimental setup to study back wall temperature of the said test laminates. The oxy-acetylene flame capable of producing a flame temperature up to 3000°C using a calibrated oxyacetylene welding torch. This type of experimental setup is used for testing the composite materials at relatively low costs while still simulating extreme conditions in real time applications ^[4,5]. OTB setup contains a data acquisition system to measure the in situ temperature of the test specimens using embedded J type (Fe-Cu) thermocouples. Test sample of 4' X 4' is held on the fixture, and oxy acetylene torch is held at a predetermined distance (d=30cm) in front of the laminate focusing at the center as shown in **Figure 3**. The torch is lit and the sample is subjected to exposure for more than 1 minute and the back wall temperature recorded for the said laminates refer the Figures (**Figures 4 and 5**).

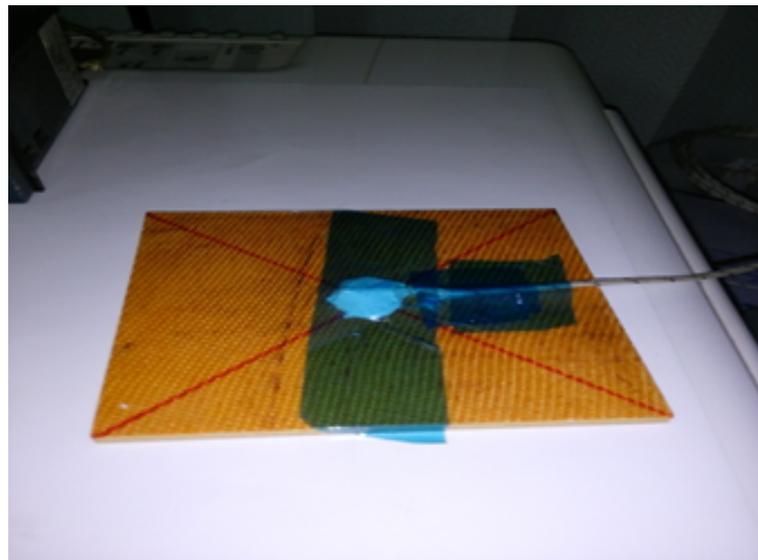


Figure 3: Thermocouple bonding



Figure 5: Laminate fixing.

TEST RESULTS

The experimental test results for the Mechanical test sample are as follows (**Table 2**). The oxy-acetylene test results revealed the back wall temperature are as follows (**Table 3**).

Table 2: Mechanical properties.

SI. No	Laminates	Tensile Strength MPa	Tensile Modulus GPa	% of Elongation	Flexural Strength MPa	Flexural Modulus GPa	ILSS MPa
1	L1	348.49	86.43	2.80%	393.14	58.69	21.45
2	L2	231.22	63.71	2.39%	236.86	40.91	13.50
3	L3	408.6	94.35	2.25%	416.37	60.28	23.44

Table 3: Back wall temperature results.

SI. No	Time taken in Seconds	Laminate 1	Laminate 2	Laminate 3
1	15	34	52	45
2	30	57	88	80
3	45	118	96	94
4	60	192	100	98

CONCLUSION

The mechanical properties of laminates exhibit the enhanced in Flexural strength and ILSS in L3 test sample i.e Carbon Phenolic laminate with 15%Zrb2-Sic filler compared with other laminates due to good compatibility between filler and laminate. High tensile strength achieved in the laminate L3 and moderate properties were achieved in Laminate L1. The properties of laminate L2 were low compared to one and three due to bigger grain size which led to internal fracture of laminate. There was sudden incrimination of temperature seen in second and third stage interval in which 50% temperature increment have been recorded in Laminate L1. Whereas laminate L3 was least with temperature of 80°C in 30 seconds. The temperature recorded for laminate L2 was quire moderate with 88°C in 30 seconds. At overall time interval for 60 seconds ablation was higher and extreme in laminate L1 with a record temperature of 192°C in 60 seconds. To conclude the experimental result shows the Laminate L3 with Zrb2-Sic (with 15%) filler shows that the improved performance in mechanical properties like tensile, flexural and ILSS and the oxy-acetylene flame test shows the less back wall temperature at 60 sec 90°C comparing 192°C, 100°C in laminate L1 and L2 respectively. Hence, PAN based carbon/phenolic based composites with Zrb2-Sic filler exhibits better thermal protection resistance than the carbon/phenolic laminate without filler.

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