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Investigation Study on Silica Filler Pan Based Composite Laminates

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Abstract: **-- The aim of this work is to give more information on the PAN/carbon phenolic laminate with and without silica filler material. The experimental study of the mechanical and thermal characteristic properties of Phenolic resin based composites with Silica filler and Carbon as reinforcement will be studied. Laminates will be made by PAN based Carbon with and without Silica filler, sample pieces are tested as per ASTM standards. The quality of laminates is verified and further tested for their mechanical properties like tensile, compression, flexure & ILSS and thermal properties like ablation rate. Fibre volume fraction is also determined from resin content and density as a part of physical properties. The test results are reveals that CP laminate with 5% Silicon filler exhibits the better physical and mechanical properties compared without silica filler laminate.**

Keywords: **-- PAN Carbon fabric, Phenolic Resin, Silica, hand layup process, Oxy-acetylene torch flame test.**

I. INTRODUCTION

Generally composite materials are combination two or more materials and resulting in enhancing the properties of end product. In this experimental study the PAN/Carbon is used as reinforcement and phenolic resin as matrix, but in phenolic resin mixed with Silica filler the predominate changes are noticed in physical and mechanical properties.
The airframe structure in aerospace is made up of carbon phenolic due to with stand high thermal application [1,2]. The ablation properties are enhanced with adding of silica filler material [3]. The experimental process for this study is to evaluate the physical and mechanical properties and to know the back wall temperature with help of oxy acetylene test bed [4,5].

This work is focussed on the study of the silicon filler involved in the development of carbon/PAN phenolic laminate and how variables are affected by the incorporation of adding the silica filler. Test laminates are prepared with and without silica filler in varying the percentage of silica with phenolic resin mix. The three laminates are prepared by hand layup method and followed by cured cycle. The cured laminates quality was ensured and subjected to testing as per ASTM standards. The physical and mechanical test results are tabulated. To know the back wall temperature of the laminate to evaluate the ablation rate by other small experiment known as oxy acetylene test method.

II. RAW MATERIAL SELECTION

In the present work, to evaluate the physical and mechanical characterisation of the laminate with adding the silica filler, PAN as reinforcement and phenolic resin as matrix. The following raw materials are selected for this experimental study.

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	Silica Powder

2.1 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[6,7]. 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.

2.2 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

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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.

2.3 Filler material:

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 Silica powder procured from Allied Agencies, Hyderabad. The Silica Content is Min 94% and Moisture content is Max. 0.5% by weight, the Particle size is ≤ 50µ equal to 80% Minimum and 50 to 70µ is equal to 0 to 20 %. Grains of silica can be bonded together by sintering to form very hard material that is widely used in aerospace applications. Hence it is selected.

III. EXPERIMENTAL PROCEDURE

In this experimental study, the three types of laminates were considered, there are Laminate 1 (L_1) is Carbon-Phenolic without adding filler, Laminate 2 (L_2) is Carbon-Phenolic with 5% Silica filler, and Laminate 3 (L_3) is Carbon-Phenolic with 10% Silica filler.

3.1 Preparation of Laminate:

The lay-up of pre impregnated material by hand is the oldest and most common fabrication method for advanced composite structures. Furthermore, the basic features of the method remain unchanged. Various steps are involved in the hand lay-up of a flat composite laminate. Each step must follow in successive fashion in order to obtain a high-quality composite laminate after final processing. Hand layup process is described in the following steps:-

- **Step 1.** The surface of the tool is cleaned and a release agent is applied.
- **Step 2.** An optional sacrificial layer is laid up on the tool surface.
- **Step 3.** A peel ply is placed on top of the sacrificial layer.
- **Step 4.**The pre impregnated plies are cut according to size.
- **Step 5.** The prepreg ply is oriented and placed upon the tool or mold.
- **Step 6.** A flexible resin dam is anchored to the sacrificial layer.
- **Step7.**Another peel ply is placed on top of the laminate to protect the laminate surface.
- **Step 8.** A sheet of porous release film is laid over the dam and the laminate.
- **Step 9.** Next, bleeder plies are laid up over the release film.
- **Step 10.** The vacuum bag is made; the mould is kept inside the bag and sealed, and kept inside the autoclave.

Fig (a,b,c) represents the hand layup process

3.2 Preparation of Test Specimen:

The laminates are taken out of the autoclave are to be cut into required sizes as per ASTM standard. Specimen subject to as per ASTM D792 standard describes the determination of the specific gravity / relative density. Some more specimen subject to as per ASTM D 2584 standard describe the determination of the ignition loss of cured reinforced resins i.e. resin content and ensured remaining portion is fiber content. The tested specimens having dimensions of length 250mm width 25mm and thickness 3.0 mm. To evaluate the mechanical properties as per ASTM D 2344 determine the short beam strength of high modulus fiber reinforced composite material i.e. Inter Laminar Shear Stress (ILSS) at room temperature. ILSS is usually limiting design characteristics because conventional manufacturing techniques do not reveal reinforcing fibers oriented in the thickness direction to sustain load. As per ASTM D 790 standard determination of flexural properties of laminate by 3-point bend test. Six specimens were tested and average value is considered for flexural strength and the test specimens were examined through visual inspection for failure of fiber and matrix. Finally as per ASTM D 3039 determines the in plane tensile properties of the laminate at room temperature by using INSTRON Universal Testing Machine (UTM), model No.1185 with load cell capacity of 100 KN made by UK. The specimen was loaded between two adjustable grips of UTM. Each test was repeated three times and the average value was taken to calculate the tensile strength of the laminate.

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Fig(d) Test Samples Fig(e) Flexural Test Fig (f) ILSS Test

***3.3 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 Fig (g) & (h). 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 Fig (i).

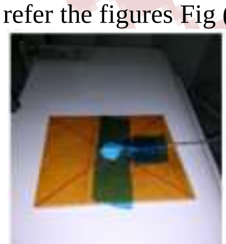 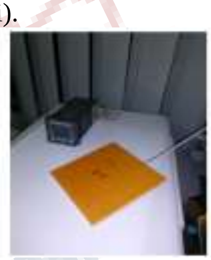 

Fig (g) Thermocouple bonding Fig (h) OTB Sample
Fig (i) OTB Test Setup

IV. TEST RESULTS

The comparative study has been carried out between Physical properties and Mechanical properties. Physical properties like Density, Resin content and fiber content were measured. Mechanical properties like Flexural strength and Inter Laminate Shear Stress (ILSS) were measured. The physical properties are achieved by using the above said test samples are given Table 2.

Sl. No	Test sample	Density (gm/cc)	Resin content (% of wt.)	Fiber Content (% of wt.)
1	L_1	1.63	40.15	59.85
2	L_2	1.72	37.93	62.07
3	L_3	1.58	35.62	57.93

Table 2: Physical properties

The mechanical properties are achieved by using the above said test samples are given Table 3.

Sl. No	Test sample	Flexural (MPa)	ILSS (MPa)
1	L_1	101.46	9.435
2	L_2	104.89	11.02
3	L_3	83.10	7.13

Table 3: Mechanical properties

The back wall temperature properties are achieved by using the Oxy-acetylene test flame are given Table 4.

Sl. No	Time taken in Seconds	Temperature achieved for Laminate L1 in Degrees	Temperature achieved for Laminate L2 in Degrees	Temperature achieved for Laminate L3 in Degrees
1.	0	38	37	37
2.	10	48	46	43
3.	20	62	54	51
4.	30	78	66	62
5.	40	88	78	73
6.	50	102	89	85
7.	60	124	98	91

Table 4: Back wall temperature Results

The erosion rate for tested laminates results are tabulated in the table 5.

Sl. No	Test sample	Thickness before test (mm)	Time taken for Temperature achieved in sec	Erosion rate (mm/sec)
1	L_1	3.00	124	0.0241
2	L_2	3.00	98	0.0306
3	L_3	3.00	91	0.0329

Table 5: Erosion rate results

V. DISCUSSION

❖ In the present research work phenolic composite filled with silica filler with varying volume fraction by weight laminates are prepared by hand layup process. Several important considerations can be investigated based up on the test results obtained from the experimental test.

- The test results revealed that the phenolic composite with 5% silica filler added laminate have the better physical properties like Density, Resin content and Fiber content compared with other laminates.
- The flexural strength results indicated that composite laminates filled with by (5% volume) of silica filler exhibited maximum flexural strength (104.89 MPa) compared with other filled composite laminate this may be due to good compatibility between filler and matrix. However, test results shows that increase in addition of filler reduces flexural strength this may be due to that the further increase in filler content as increased brittleness thus failed to withstand bonding load of higher magnitude.
- The ILSS test results indicates that composites filled by (5% volume) of silica filler exhibited Inter Laminar Shear Strength (11.02 MPa). This is due to good adhesion between layers.
- The experimental test result shows the composite laminate L3 with 10% silica filler exhibits the better transition temperature and withstand higher temperature. However, the erosion rate also achieved 0.0329 in Laminate L3 compared with other laminates due to good bonding of silica particles between the layer to layer.

VI. CONCLUSION

The Results of study showed that the carbon / phenolic composites with silica filler exhibited better properties than the carbon / phenolic composites without silica filler. However, 5% silica filler laminates are exhibits the better physical and mechanical properties than others. Further increase in filler content has detrimental effect due to improper bonding between the matrix and filler interface and increased embrittlement of the composites. The increase in percentage of silica filler leads to with stand higher transition temperature and good ablation rate compared with other laminates.

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