Experimental Study on Pan Based Composites used in Aerospace Application

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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 adding with different type of fillers include. Nano filler by hand layup process cured under temperature and pressure in the autoclave. The test samples are prepared by ASTM standard and subjected to testing. Comparisons of Physical, Mechanical and Thermal properties of the laminates are 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 nano filler exhibits the better thermal protection than other filler laminates. However, Nano filler laminate have the lower strength than the other filler laminates.

Keywords

PAN Carbon Fabric; Phenolic Resin; Hand Lay-Up Technique; Oxy-Acetylene Flame

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].

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 mixed with filler to improve the properties of laminate and withstand the higher transition temperature [4].

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The major objective of this research is to develop the PAN based laminates with different types of fillers including nano filler and to study the physical, mechanical, thermal properties [2, 4]. The thermal properties can be revealed with help of the oxy-acetylene test. The process involves PAN fabric impregnated with Phenolic resin with different proportion of filler material and the laminates are prepared by hand layup technique. The laminates are cured in autoclave under temperature, pressure and vacuum. The quality of laminate found satisfactory then test samples are prepared as per ASTM standard subjected to physical, mechanical & thermal properties.

2. Selection of Materials

The following materials are selected for this experimental study. In the present work, the mechanical and thermal characteristic properties of Phenolic based composites with zirconium, silica & nano fillers and carbon as reinforcement will be studied.

### Table 1: Details of Raw Materials

<table>
<thead>
<tr>
<th>SL. No</th>
<th>Raw material</th>
<th>Grade</th>
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<tbody>
<tr>
<td>A</td>
<td>Reinforcement</td>
<td>PAN Carbon fiber T-300</td>
</tr>
<tr>
<td>B</td>
<td>Phenolic Resin</td>
<td>ABRON-PR 100 (WS)</td>
</tr>
<tr>
<td>C</td>
<td>Filler material-I</td>
<td>Zirconium Powder</td>
</tr>
<tr>
<td>D</td>
<td>Filler material-II</td>
<td>Silica Powder</td>
</tr>
<tr>
<td>E</td>
<td>Filler material-III</td>
<td>Nano Powder</td>
</tr>
</tbody>
</table>

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. PAN carbon fabric 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 used commercially available of ABRON-PR100 (WS) Phenolic resin to meet the requirement for low smoke and toxicity [2, 4]. Hence it is selected.

2.3 Filler Material-I

Zirconium Powder is a highly covalent refractory ceramic material with a hexagonal crystal structure. Zirconium is having the average particle size of 1.5 microns. The zirconium powder procured from Allied Agencies, Hyderabad. Zirconium powder 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 [2]. Hence it is selected.

2.4 Filler Material-II

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 [4]. Hence it is selected.

2.5 Filler Material-III

Nano composites are composite with nano meter size constituents. Nano particles are used to improve the stiffness of the composites, but there are not good for improving the tensile strength. Nano particles in the form of powder procured from Archit advanced materials, Dark Force. Nano particles are proper orientation of the fibers, directional properties can be obtained. Complex shapes can be easily moulded without material wastage. The main advantage of Nano particles are to increasing temperature tolerance, and physical performance [4]. Hence it is selected.

3. Experimental Procedure

In this experimental study, the following four types of laminates were considered, there are Laminate 1 is Carbon-Phenolic without filler, Laminate 2 is Carbon-Phenolic with 5% filler addition, Laminate 3 is Carbon-Phenolic with 10% filler addition and Laminate 4 is Carbon-Phenolic with 15% filler addition. Refer table 1, as we said above the corresponding fillers are Zirconium, Silicon carbide and Nano filler in the form of powders are used in this experiment to study the Physical, Mechanical and Thermal properties.

3.1 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 was PAN carbon fabric cut into required size (250x250x4mm) 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. Four types of test laminates are prepared by hand layup process by using the above said raw materials without filler and with filler added as 5%, 10% and 15% to the Phenolic composite. The laminates are cured in autoclave by a cure cycle of Temperature Vs Time under vacuum and pressure. Post curing cycle laminates are visually verified and ensured that free from cracks, voids and porosities.

3.2 Designation of Laminates

Type 1 Laminate: Carbon-Phenolic Laminate without filler designated as L1

Type 2 Laminate: Carbon-Phenolic Laminate with 5% filler designated as L2

Type 3 Laminate: Carbon-Phenolic Laminate with 10% filler designated as L3

Type 4 Laminate: Carbon-Phenolic Laminate with 15% filler designated as L4

3.3 Preparation of Test Specimen

Test samples are prepared 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 as a part of evaluate the physical properties.

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 [2]. 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 [3]. 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.

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

\[
\sigma_f = \frac{3PL}{2bh^2} \quad (1)
\]

Where, \(\sigma_f\) - 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”

\[
ILSS = \frac{3F}{4bt} \quad (2)
\]

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

Figure 1: Flexural Strength Specimen
3.4 Testing of Specimen on Oxy-Acetylene Test Bed

According to ASTM E285-80 [4], 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) thermocouple. 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 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 60 seconds and the back wall temperature recorded for the said laminates refer the Figure 4 and 5.

4. Test Results and Discussions

The comparative study has been carried out between Physical properties, Mechanical properties and Thermal properties. Physical properties like Density, Resin content and fiber content were measured. Mechanical properties like Inter Laminate Shear Stress (ILSS), Flexural strength and Tensile strength were measured. Thermal properties like back wall temperature at the end of test (60 sec), Burn through time and ablation rate can be revealed with help of Oxy-acetylene test method. Refer the Table 2 for zirconium filler laminates and Table 3 for Silica filler laminates and Table 4 for nano filler laminates. The experimental test results for the said test laminates are as follows:
In the present research work Phenolic composite filled with zirconium, silica and nano fillers with varying volume fraction by weight (%) were prepared. Fabrication of reinforcement laminates samples are prepared by hand layup process using mould at room temperature. Several important considerations can be drawn based up on the test results obtained from the experimental test.

The study of results revealed that the Phenolic composite with filler have the better physical as well as mechanical and thermal properties as compared without filler added to Phenolic composite laminates (Refer Table (1, 2 and 3). The density exhibits the 5% of nano filler laminate L2 have 1.2146 g/cm³; it denotes light weight laminate among the other laminates due to nano particles. It is also noticed that zirconium filler laminates are have the higher physical properties than other two types of silica and nano filler laminates.

The test results exhibits the 10% of zirconium filler added to the Phenolic composites are enhance the ILSS, Flexural strength, Tensile strength properties and physical properties like Density and Resin content as compared with other samples. Zirconium filler laminates are high strength than silica and nano filler laminates. However, the silica filler laminates are better thermal

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<th>Table 4: Laminates with Nano Filler</th>
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properties against zirconium filler laminates; but overall, the nano filler laminates exhibits and withstand the high thermal loads against zirconium, silica filler laminates.

It is noticed that the reinforcement composite laminate filled by (10% volume) of zirconium filler exhibited maximum tensile strength (421.91 MPa) compared with other filled composite laminates this may be due to good particle dispersion and strong polymer/filler interface adhesion for effective stress transfer. Increase in adhesion of filler materials leads to decrease in tensile strength this may be due to more filler distribution and filler metal dominated in the materials. The flexural strength results indicated that composite laminates filled with by (10% volume) of zirconium filler exhibited maximum flexural strength (425.64 MPa) compared with other filled composite laminates this may be due to good compatibility between filler and matrix. However, test results shows that increase in addition of 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 zirconium filler exhibited Inter Laminar Shear Strength (25.99 MPa) compared with other laminates. This is due to good bonding between layers to layer.

Thermo mechanical ablation means the process of mechanical crumbling of particles from surface of composite by a hot high-speed flow. The flame has high velocity at about 3000°C and 15 MW/m² heat flux. The Transition or back wall temperature of the samples are measured, by focusing the thermal beam set up located perpendicular to the sample surface. The OTB post test laminates for zirconium, silica and nano fillers are shown in Figure 6, 7 and 8 respectively.

The investigation revealed that Carbon Phenolic laminate with Nano filler exhibits the better transition temperature i.e. 74°C obtained during the test at end of 60sec as compared with Zr and Si filler laminates. It is also noticed that the burn time through hole maximum obtained 227 seconds in 10% of nano filler laminate is withstand higher burning rate compared with other laminates. The graphical representation of the three types of laminates as shown in Figure 8a for Transition Temperature curve and Figure 8b for burn time through hole respectively.

**Figure 6:** Post test Zr.Laminate

**Figure 7:** Post Test Si.Laminate

**Figure 8:** Post Test Nano Laminate

**Figure 8a:** Transition Temperature Curve
The ablation rate was calculated by dividing the specimen thickness before and after the test by a burn through time for each specimen. The ablation rate test results are revealed that Carbon Phenolic Laminate with Nano filler (L2) shows the 0.017mm/sec of ablation rate among the other two types of laminates and have the better thermal properties. The graphical representation of the ablation rate study of the Carbon Phenolic laminates with and without fillers as shown in Figure 8c.

Figure 8b: Burn Time Through Hole in Laminates

Figure 8c: Ablation Rate Test Results

5. Conclusion

(i) Carbon-Phenolic (C-Ph) fabricated with nano filler particles are suitable for low weight applications due to lower weight density than other laminates by virtue of its particle sizes.

(ii) Further increase in filler content has detrimental effect due to improper bonding between the matrix and filler interface and increased embrittlement of the composites.

(iii) Presence of Zirconium filler in Carbon-Phenolic (C-Ph) composites has exhibits the better physical and mechanical properties than silica and nano filler laminate due to good compatibility between filler and laminate. The Zirconium filler can improve the room temperature flexural and shear strength of C–Ph composites. However enhancement in these properties lost their prominence in the charred matrix.

(iv) Presence of Silica filler in C-Ph composites has exhibits the lower physical and mechanical properties than zirconium filler composites due to bigger grain size which led to internal fracture of laminates. However, the thermal properties are higher than the zirconium filler laminates due to enhanced conversion of solid char into silica carbide and carbon monoxide leading to under protection of carbon fibers facing the oxy-acetylene flame.

(v) Presence of Nano filler in C-Ph composites can show high burn through time, leading to reduced back face temperature when exposed to high temperature environments. The physical and mechanical properties are shows the lower than zirconium and silica filler due to nano particle size dispersion. However, the ablation rate is improved and enhanced the thermal conductivity. Hence, the nano filler composites are best composite laminate for high temperature (thermal) applications.

References


