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Nano Filler Composite Material Study For Aero Space Application

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Abstract: *Nano composites are found in a number of places and the use of nanoparticle-rich materials predates the understanding of the nature of these materials. In recent times nano composites have provided the aerospace industry with a variety of material solutions. The aim of this experiment is to investigation study on PAN based composite laminates with Nano filler with %age of weight made by hand layup technique. The oven cured laminate subjected to destructive testing as per ASTM standard to evaluate the thermal properties of said laminates. The experimental studies investigate resin content, ILSS, DSC, TGA and ablation studies with help of Oxy-acetylene test bed. The test results are revealed that PAN based laminates with adding Nano filler exhibits the better thermal properties compared without adding nano filler laminate. This experimental study results are very much useful to make a composite materials for better thermal applications in aerospace industry.*
Keywords: *PAN Carbon fabric, Nano filler, ASTM standard, OTB, DSC, TGA.*

I. Introduction

Advanced composite materials are ideally suited for aerospace applications Composite materials are combination of two or more dissimilar materials, intimately bonded to form integrated structure. Composites are distinct interface between constituents and superior properties than individual components. [1]. Composites are so attractive due to its economic gains through light weight construction, weight reduction, improvements in fatigue resistance, corrosion prevention, potential fabrication cost advantages for parts with complex shapes. The composite material are used into wide range of components to supply a diverse and fragmented commercial base that includes customers in aerospace, aircraft, defense, marine industry Phenolic composite products are commonly used in high temperature environment for relatively long period of time and light weight composition [2,3].

The research field focused on the improvement of the thermo mechanical properties of the matrix in order to enhance the efficiency of the composite structure applications, one of the best method used in order to improve the characteristics of the matrix as well as the composite is the incorporation of nano particles. Adding nano particles to a polymer matrix can enhance its performance. This approach is particularly effective in yielding high performance composites, when the nano particles are well dispersed and the properties of the nano scale filler are substantially different or better than those of the matrix. [4,5].

The re-entry vehicle structure like missile airframe and aircraft outer envelope is withstood for high thermo mechanical properties. The major objective of this experimental study is to develop the Poly Acrylo Nitrile (PAN) based laminate with different types of fillers including nano filler and to study the physical, mechanical, thermal properties. The thermal properties can be revealed with help of the oxy-acetylene test bed [6,7]. 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 cured laminates are subjected to destructive testing as per ASTM standard.

II. Selection Of Materials

2.1 PAN carbon fabric: The main reinforcement is PAN Carbon fabric; it is amorphous material for ablative purpose and plays the dominant role in development of composite materials due to their high specific strength, stiffness and low weight applications. The function of reinforcement is to carry the load along the length of the fiber provides strength and stiffness to one direction.

2.2 Phenolic Resin: The phenolic resins are poly condensation products of phenols and formaldehyde. The commercial grade of ABRON-PR100 phenolic resin is a conventional matrix and synthetic polymer to meet low smoke and toxicity in composite industry [2,4,5]. The main function of phenolic resins to transfer stress between reinforcing fibers. The advantages are to retain the position of reinforcement and protect them from mechanical and environmental damages.

2.3 Nano filler: Nano powder contains nano meter size constituents to improve the stiffness of the composites, but there are not good for improving the tensile strength. The main advantage of Nano particles are to increasing temperature tolerance, and physical performance [4,7].Nano particles in the form of powder procured from Archit advanced materials, Dark Force. Nano scale dispersion of nano particles in the composite can introduce new physical properties such as accelerated biodegradability or fire resistance that are absent in the unfilled matrices, effectively changing the nature of the original matrix. The use of nano particles in these polymer matrices, thus creating a nano composite can yield an optimal multi-functional material for aerospace needs and other applications. Hence, the above materials are selected for this experimental study.

III. Experimental Procedure

3.1 Preparation of Laminate:

The PAN carbon fabric reinforcement and phenolic resin as matrix material are to be used to prepare the laminate by using hand layup technique. The hand layup process is one of the most popular types of open molding process. The process involves the following operations.

a) The mould is coated by a release anti-adhesive agent, preventing sticking the mould part to the mould surface.
b) The prime surface layer of the part is formed by applying the gel coating.
c) A layer of fine fiber reinforcing tissue is applied
d) A layer of liquid matrix resin and reinforcing fibers in the form of woven fabric or chopped strands are applied till the required thickness of the sheet is obtained.
e) The resin mixture is either applied by a brush or a roll.
f) The part is cured in autoclave.
g) The sheet which is dry is removed from the mould is called as laminate

Four types of test laminates are prepared without filler and with nano filler added as 5%, 10% and 15% to the Phenolic composite. The cured laminate is cut into required size 250x250x4mm). Post cured laminates are visually verified and ensured that free from cracks, voids and porosities and subjected to testing as per ASTM standard.

3.2 Designation of Laminates:

Type 1 Laminate: Carbon-Phenolic Laminate without filler designated as L_1
Type 2 Laminate: Carbon-Phenolic Laminate with 5% nano filler designated as L_2
Type 3 Laminate: Carbon-Phenolic Laminate with 10% nano filler designated as L_3
Type 4 Laminate: Carbon-Phenolic Laminate with 15% nano filler designated as L_4

3.3 Testing Procedure:

The above designated four types of laminates were tested as per ASTM standard. 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]. 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.

3.4 Oxy-acetylene Test Bed (OTB):

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 refer Fig (1). 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 (5). 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 figures Fig (2) & (3).

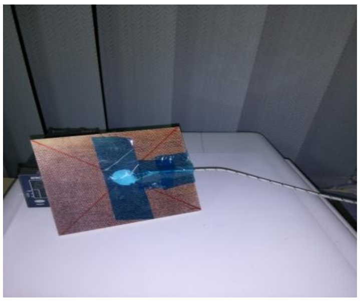


Fig.(1)Thermocouple bonding Fig.(2) Oxy-acetylene Equipment Fig.(3) OTB Laminate Testing

3.5 Differential Scanning Calorimetry (DSC): Thermal analysis of composite laminate where as properties of materials are studied as they change with temperature. DSC (Differential Scanning Calorimetry) is the heat flow to and from a sample and a reference material is measured as a function of temperature as the sample is heated at constant temperature. The measured signal is the energy absorbed by or released by a sample in mill watts. DSC allows you to detect endothermic and exothermic effects, measure peak areas determine temperatures that characterize a peak or other effects, and measure specific heat capacity. The figure (4) represents the DSC apparatus set up in the laboratory.

Fig.(4) Differential Scanning Calorimetry

3.6 Thermo Gravimetric Analysis (TGA): Thermo Gravimetric Analysis is measures the weight and hence the mass of a sample as a function of temperature. Previously the acronym TG was used for this technique. Now a day, TGA is preferred in order to avoid confusion with $T_{g,}$ the glass transition temperature TGA allows you to detect changes in the mass of a sample (gain or loss), evaluate stepwise changes in mass and determine temperatures that characterize a step in the mass loss or mass gain curve.

Fig. (5) Thermo Gravimetry Analyser

IV. Test Results And Discussions

The comparative study has been carried out between Physical properties, Mechanical properties and Thermal properties. Physical properties like Density, Resin

Table (i) <u>LAMINATES WITH NANO FILLER</u>

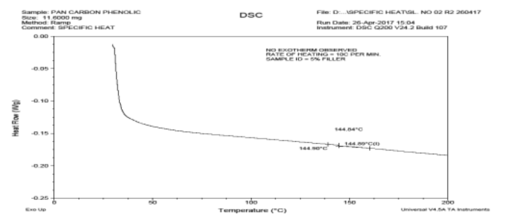
content and fiber content were measured. Mechanical properties like Inter Laminate Shear Stress (ILSS), Flexural strength and Tensile strength were measured. Thermal properties like back wall temperature at the end of test (60 sec), Burn through time and ablation rate can be revealed with help of Oxy-acetylene test method. Refer the Table (i) experimental test results for nano filler laminates.

The test results are indicates that there is close relation between the improved characteristics of the additive of nano filler and without filler composite laminate. The properties of the laminates are greatly influenced by nano scale of composite phase and the degree of mixing between them. The tensile strength of the nano filler addivated composite laminates is lower than the strength of the non-addivated composite laminate.

It was understood that the 10% nano filler laminate exhibits the better physical and thermal properties than other laminates due to its grain size, however, the nano filler laminate are showing the lower strength compared with without adding of filler laminate. The density exhibits the 10% of nano filler laminate L_2 have 1.1436 g/cm^3; it denotes light weight laminate among the other laminates due to nano particles dispersion throughout the matrix. As expected, the tensile strength, ILSS for the samples with nano additivated matrix were inferior to those with non-additivated matrix laminates. But, nano filler laminates presented improved the thermal properties compared to that non additive filler laminate.

The thermo mechanical properties of the composite depend on the efficient dispersion of the nano additive into the polymeric matrix and on the efficient impregnation of the reinforcement fiber plies. So, increasing nano filler amount added in the matrix leads to an increased difficulty to impregnate the reinforcement fiber to obtain reliable composite laminate. 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/m^2 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 ablation rate is very low and that is because the degradation process has not started at eh depth of the sample. 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 (L_2) shows the 0.019mm/sec of ablation rate among the other laminates and have the better thermal properties.

Sl. No	Test Sample	Physical Properties			Mechanical Properties			Thermal Properties		
		Density (g/cm^3)	Resin Content (%)	Fiber Volume Fraction (%)	Flexural Strength (M.Pa)	Tensile Strength (M.Pa)	ILSS (M.Pa)	Back wall Temp (°C)	Burn through time (sec)	Ablation Rate (mm/sec)
1	L_1	1.1454	22.05	49.37	320.16	323.49	26.81	172	144	0.034
2	L_2	1.1786	22.44	52.74	313.64	317.28	12.42	89	201	0.022
3	L_3	1.1436	22.86	53.28	312.18	312.43	18.24	76	232	0.019
4	L_4	1.1865	22.89	52.89	316.80	316.32	16.86	82	222	0.021



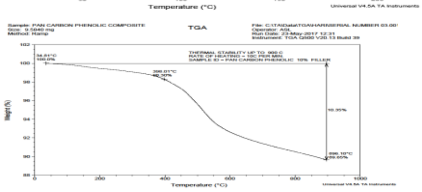


Fig.(6) DSC graph 10% nanofiller laminate

Fig.(7) TGA curve 10% nano filler laminate

In DSC described the measure of heat absorbed or liberated during heating of the laminate, the temperature 144.89°C gives the corresponding heat flow 0.2292 W/g. Refer the figure (6) DSC graph for 10% Nanofiller laminate. TGA measured the change in weight during heating of the laminate, T_g is the glass transition temperature at which polymers converts from glassy to rubbery state it reveals that the sample is fully cured the temperature gone up to 900°C gives the corresponding mass weight loss of 27 %. The test sample is fully cured and there is no exothermic reaction with polymer matrix. The thermal parameters determined by employing TGA results and used to calculate the back wall temperature of the composite laminate. Refer the figure (7) TGA graph for 10% nano filler laminate. Experimental data obtained by OTB the back surface temperature is increased up to 820.6K during 60 sec. As can be seen, the experimental data good agreement with the predicted values of the said laminates.

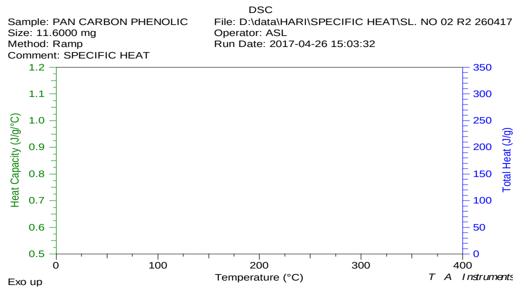
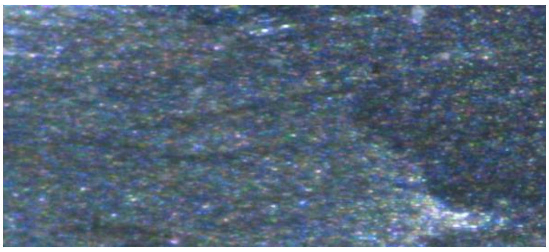


Fig. (8) Specific heat curve



Fig(9)HD stereo microscopic figure

The sample undergoes no decomposition with loss of lattice products over the temperature range shown but solid phase transformation. The temperature 100°C duration of 12 min gives the total heat capacity of 47.57 J/g for non additive of the filler composite laminate where as for additive of 10% nano filler gives the temperature 100°C duration of 12 min gives the total heat capacity of 65.83 J/g. Refer the fugure (8) for Specific heat curve. The post test laminate visualized through High definition stereo microscopic 10% nano filler laminate as shown in figure (9) as above. The test results will give the confirmation that kinetic parameters and degree of thermal degradation reaction that have been determined by employing the OTB, DSC, TGA results.

V. Conclusion

(i) The additive of nano filler particles is suitable for low weight applications due to lower weight density than other non additive nano filler laminate by virtue of its particle sizes.

(ii) The increase in nano filler additive responding the matrix leads to increase difficulty to impregnate the reinforcement fiber due to improper bonding between the matrix and filler.

(iii) The nano filler additive laminate gives the uniformity of the nano particles in the composite surface as compared with non additive filler laminate some dry patches are observed with help of stereo microscope it's may be due to good compatibility between filler and laminate.

(iv) The mechanical strength, ILSS and not improved due to its distribution of nano particles due to bigger grain size which lead to internal fracture of laminates.

(v) As per the DSC curve the nano filler additive laminate withstand higher thermal properties, the specific heat increasing and absorbing energy as ablative material because loaded with nano filler particles.

(vi) The higher the temperature range there is completely cure and it is indicate that there is no exothermic as per TGA curve. However, the ablation rate is improved and enhanced the thermal conductivity. The specific heat curve denotes the nano scale dispersion of particles accelerated biodegradability or higher temperature resistance is effectively changing the nature of the original matrix. Hence, the nano filler composites are best composite laminate for high temperature (thermal) applications, can yield an optimal multi functional material for aerospace needs and other applications.

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