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# CHARACTERIZATION OF E-GLASS PHENOLIC COMPOSITE AS ABLATIVE LINER FOR FLEX NOZZLES AND JET DEFLECTOR

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## ABSTRACT

The E-glass fabric with thickness of 0.78 to 0.81 mm is impregnated with phenolic resin in the vertical impregnation plant. The laminate is made and the complete characterization is done in terms of physical, mechanical and thermal properties. The physical properties like density, fiber and resin content, barcol hardness. The thermal properties like Oxy-acetylene torch test to estimate erosion rate. The mechanical properties like tensile strength and modulus, Compression strength and modulus, flexural strength & modulus, In-plane shear strength and modulus, ILSS etc. The rocket exhaust gas is of high temperature, the structural steel members of nozzle should be protected from hot gases by highly erosion resistant thermal liners. For this purpose, ablative composite liners made up of Phenolic resin matrix combined with reinforcement material like E-glass used. The jet deflector is used to deflect the exhaust plume of solid rocket motors for vertically launch missiles. This paper briefs about the material characterization of this material for all the necessary physical, mechanical and thermal tests conducted on the laminate to qualify this material for use as ablative liner for flex nozzles and jet deflector.

**Key Words:** *Ablative Liner, Prepreg and Phenolic Resin*

## 1. Introduction

Characterization is essentially the process whereby materials, components, sub-systems and systems are defined in terms of their distinctive attributes, qualities, properties and functions. Composite materials behave in a complicated fashion due to macroscopic anisotropic effects and other coupling effects. Hence, the experimental characterization of composite materials is more complicated than for conventional, homogenous, isotropic materials. Material characterization is done in terms of measurable parameters that the designer may relate to design parameters. Physical properties of laminate such as resin content and fiber volume fraction is determined by acid digestion method according to ASTM D3171 standard [1]. For minimum characterization of a bidirectional composite, four independent elastic constants, namely the elastic moduli in longitudinal and transverse directions, the in plane shear modulus, the major Poisson ratio and five independent strengths namely tensile and compressive strength in the longitudinal and transverse directions and the in plane shear strength are to be determined.

## 2. Manufacturing Process of E-Glass Fabric / Phenolic Resin Laminates

Manufacturing of laminates involves manufacturing of prepreg, layup of prepreg and curing of laminates in Autoclave.

**Table 1: Test Results of E-Glass Fabric**

| S.No | Property | Tested Value |
|---|---|---|
| 1 | Aerial Density , g/m2 | 850 |
| 2 | Thickness, mm | 0.80 |
| 3 | Fabric Count: Warp (Ends/ Inch) | 44 |
| | Fabric Count: Weft (Picks/ Inch) | 34 |
| 4 | Weave | 8 H Satin |
| 5 | Tex: Warp (g/km) | 2x136 |
| | Tex: Weft ( g/km) | 2x136 |
| 6 | Fabric Breaking Strength Warp Direction (Kg/ Inch) | 188.6 |
| | Fabric Breaking Strength Weft Direction (Kg/ Inch) | 152.46 |
| 7 | ILSS (MPa) | 25 |
| 8 | Width of Fabric | 1035 mm |

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### 2.1 E-Glass fabric / phenolic resin parameters

The Specifications & Test results of E-glass fabric in Table No.1, Phenolic resin in Table No.2 and E-Glass Phenolic Prepregs are given in Table No.3

**Table: 2 Properties of Phenolic / Resin**

| S. No | Property | Tested Value |
|---|---|---|
| 1 | Specific Gravity, at 30°C | 1.14 |
| 2 | Viscosity at 30°C , CPS | 140 |
| 3 | Point of trouble , CC ( at specific gravity of resin 0.859) | 11.7 |
| 4 | Solid Resin Content, (% by weight) | 60.19 |
| 5 | Volatile Content (% by weight) | 38.7 |
| 6 | Free Phenol Content (% by weight) | 3.48 |
| 7 | Free Formaldehyde Content (% by weight) | 0.98 |

**Table: 3 Summary of Physical Properties of E-Glass Fabric/ Phenolic resin Prepeg Composite**

| S.No. | Parameter | Specified Value |
|---|---|---|
| 1 | Solid resin content, % | 25-30 |
| 2 | Fiber content | 70-75 |
| 3 | Volatile content, % | 4-9 |

### 2.2 Preparation of E-Glass fabric/ phenolic resin laminates

#### 2.2.1 Lay-up process, vacuum bagging & curing

Eight numbers of 300X300 mm E-Glass Fabric/phenolic prepreg pieces were stacked on open mould. After completion of layup, the laminate is bagged with vacuum bagging materials. After ensuring that there is no leakage in the bagging, the laminate is cured in autoclave as per the cure cycle.

#### 2.2.2 Specimen preparation

Specimens in longitudinal (Warp direction of the fabric) and transverse (Weft direction of the fabric) were prepared from laminate with the help of a diamond edge-cutting tool and tabs were bonded to the specimen using room temperature curable adhesive (AW106/HY953U).

## 3. Test Methods

### 3.1 Physical properties

Physical properties include the determination of Density and Fiber volume fraction.

#### 3.1.1 Density

The procedure for measuring the density of a composite material is same as that used for any other solid according to ASTM D792 standard [2].

The procedure consists of the following steps:

i. Weigh specimen in air to the nearest 0.1 mg
ii. Attach the specimen to analytical balance with a thin wire and weigh while the specimen and portion of the wire are immersed in distilled water.
iii. Weigh air alone, partially immersed up to the same point as in the previous step.

The density of the material at 23°C is determined as follows:

$$\rho = \left[\frac{a}{a+w-b}\right] * 0.9975 \quad (1)$$

Where, ρ-Density

a- weight of specimen in air

b- Apparent weight of fully immersed Specimen and partially immersed wire

w- Apparent weight of partially immersed wire

0.9975- density of distilled water at 23°C (in gm/cc)

#### 3.1.2 Fiber volume fraction

A variety of methods exist for determination of fiber volume fraction, an important property of a composite. When it can be confirmed that the composite material has zero or negligible (less than 1%) porosity, the fiber volume ratio can be obtained from the densities of composite and the constituents.

The acid digestion method according to ASTM D3171-76 (1982) and D3553-76 (1989) standards, is used with composites having a matrix that is soluble in some acid that does not attack the fiber. A sample of the composite material is dried and weighed. Then it is immersed in an acid solution to dissolve the matrix. The acid used should dissolve the matrix without attacking the fibers. The residue is filtered, washed, dried, weighed and the fiber volume ratio is determined by,

Weight fraction of fiber= $(w_3-w_1)/(w_2-w_1)$ (2)

Where

$w_1$ - weight of silica crucible

$w_2$ - weight of silica crucible with sample

$w_3$ - weight of silica crucible with residue after resin burn off

Volume Fraction of Fiber $V_f = (W_f/\rho_f) \times \rho_c$ (3)

Where

$W_f$ - weight fraction of fiber

$\rho_f$ - Density of fiber

$\rho_c$ - Density of composite

Test Results of Density, Barcol hardness, Fiber & Resin content of composite are given in the Table 4

**Table: 4 Summaries of Physical Properties of E-Glass Fabric/ Phenolic Resin Composite**

| S. No. | Type of Test | Observed Value |
|---|---|---|
| 1 | Density , g/cc | 1.86 |
| 2 | Resin content, % by wt. | 25.80 |
| 3. | Fiber content, by volume | 54.30 |
| 4 | Barcol Hardness | 60 |

### 3 .2 Mechanical properties

#### 3.2.1 Tensile test

The tension test on longitudinal specimens was conducted on warp and weft direction specimens to determine tensile strength (XL), Modulus (ET) and major Poisson's ratio (μLT). In this method, the specimens with end tabs have been used. Test is conducted by using servo- mechanical testing machine (INSTRON 4505). Strain measurement is done by using Extensometer and also with strain gauges.

Specimen preparation and testing is carried out according to ASTM D3039 standard [3]. Tensile strength was determined from the ultimate load and tensile modulus is calculated from the stress-strain curve. Tensile Strength and modulus were tested in warp and weft directions. Poisson ratio is measured in both directions using strain gauges.

#### 3.2.2 Shear test

The properties that are determined through the tests are the shear strengths and shear modulus. In these tests the specimen is subjected to loads that produce a pure shear state of stress and the resulting strains are measured.

The test in which shear distortion takes place entirely in the plane of the composite material laminate are termed in-plane shear tests. The in plane shear strength (τ) and in plane shear modulus (G) are determined by this test. In this characterization programme of E-glass-phenolic composites, the in-plane shear properties are determined by the uniaxial tension test on ± 45° specimens according to ASTM D3518 standard [4].

The stacking sequence chosen for the preparation of the laminate is +45°,-45. The importance of a laminate with the stacking sequence is that the laminate will be orthotropic with respect to in-plane forces and strains, the bending, stretching, coupling effects and the in plane and bending anisotropic effects are avoided.

Strain gauges are used on specimens to measure strains along the loading direction and perpendicular to loading direction. The in plane shear modulus is calculated from the stress strain curve.

Shear stress is computed by using the formula:

$$\text{Shear stress } (\tau_{LT}) = \tfrac{1}{2} X_L \quad (4)$$

Where $X_L$ is applied stress

Shear strain is computed with the help of the following reaction:

$$\text{Shear strain } \varepsilon_{LT} = \varepsilon_L - \varepsilon_T \quad (5)$$

$\varepsilon_L$ = strain in longitudinal direction

$\varepsilon_T$ = strain in transverse direction

#### 3.2.3 Flexural strength and modulus

The flexural properties are evaluated using a three-point test fixture for warp direction specimens and weft direction specimens. The specimen is prepared according to ASTM D790M standard [5]. For a high modulus material such as this the three points is generally preferred as the most direct and simplest technique to use. Specimens are prepared according to ASTM D790M standard [ ]. Flat specimens, machined with same care and precision as previously described for tensile testing, are selected for measurements. The material direction under investigation must be oriented along the length dimension of the specimen. The test pieces require a span/depth (l/d) ratio high enough to minimize the influence of inter laminar shear deformation and to achieve failure in bending rather than shear.

The ultimate flexural strength $\sigma_1$, i.e. the stress in the outer fiber at failure, and the flexure modulus ($E_f$) are calculated as follows,

$$\sigma_1 = 3pl/2bd^2 \quad (6)$$

Where, p: load, l: length of the specimen, b: width, d: thickness

$$E_f = l3m/4bd^3 \quad (7)$$

Where, m: slope of the tangent of deflection curve

#### 3.2.4 Inter laminar shear strength (ILSS)

The Inter laminar shear strength is evaluated using a three-point test fixture. The specimen is prepared according to ASTM D2344 standard [6]. Flat specimens, machined with same care and precision as previously described for tensile testing, are selected for measurements. The material direction under investigation must be oriented along the length dimension of the specimen. The test pieces require a span/depth (l/d) ratio low enough to minimize the influence of bending deformation and to achieve failure shear rather than bending.

 

The Inter laminar shear strength (ILSS) is calculated as follows,

$$ILSS = \frac{3p}{4bd} \quad (8)$$

Where, p: load, b: width of specimen, d: thickness of specimen

The mechanical properties test results are given in Table 6 and test plots are given in Fig. 1 & 2

**Table: 5 Thermal Properties of E-Glass Fabric/ Phenolic Composite**

| Temp (°C) | Specific heat (J/Kg/°C) heating rate = 10°C/min (By MDSC) | CTE ((α) (I/°C) x E-06 Warp direction ( Dilatometer) |
|---|---|---|
| 100 | 1976 | 9.109 |
| 200 | 1654 | 7.998 |
| 300 | 1681 | 7.449 |
| 400 | 1521 | 6.997 |
| 500 | ------ | 3.4371 |
| 600 | ------ | -0.971 |

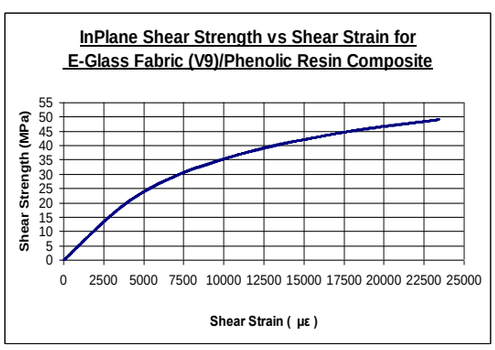


**Fig. 1 Tensile Strength Vs Strain of Composite Tested in Weft Direction**

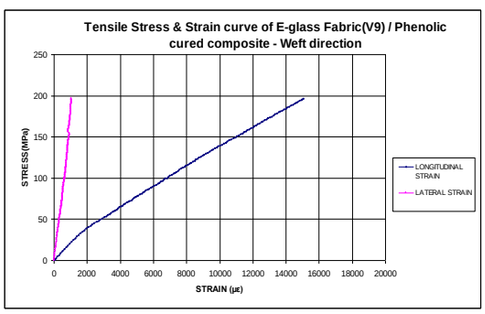


**Fig. 2 In plane Shear Strength Vs Strain Curve of Composite**

### 3.3 Thermal properties

#### 3.3.1 Specific heat or heat absorption of laminate

Specific heat /heat absorption of specimen was investigated by Modulated Differential Scanning calorimeter (MDSC). MDSC scan was recorded from Room temperature to 400°C in Nitrogen atmosphere (flow rate 30 ML/ minute). A heating rate of 10 °C/minute and sample size of 20±2 mg used in the experiment.

#### 3.3.2 Thermal conductivity

Thermal conductivity of specimen is carried out by Thermal conduct meter at 120°C using Fourier's conduction method. A disc sample (ϕ25 mm and thickness 3 mm) is used. Thermal Conductivity at 120° C= 6.2166 x$10^{-4}$ Cal/cm/sec/°C

#### 3.3.3 Coefficient of linear thermal expansion

Dilatometer used for this work is equipped with an alumina sample holder and push rod. The CTE measurements are carried out with an alumina standard and calibrated S-type thermocouple in nitrogen atmosphere. The schematic of dilatometer is shown below. The experiment is carried out from 100°C to 600°C. The length of the samples was 25 mm. The thermal properties results are shown in the Table 5.

**Table: 6 Mechanical Properties of E-Glass Fabric/ Phenolic Resin Composite**

| S.No | Type of Test | No. | Ave Value | % of C V |
|---|---|---|---|---|
| 1 | Tensile Strength Warp MPa | 10 | 277.42 | 9.60 |
| 2 | Tensile Modulus Warp GPa | 9 | 22.87 | 7.38 |
| 3 | Tensile Strength Weft MPa | 11 | 211.72 | 3.52 |
| 4 | Tensile Modulus: Weft GPa | 8 | 14.70 | 5.41 |
| 5 | Poisson Ratio Warp | 3 | 0.0887 | 7.88 |
| 6 | Poisson Ratio Weft | 3 | 0.102 | 3.2 |
| 7 | In –Plane Shear Strength (±45°) MPa | 11 | 59.57 | 8.57 |
| 8 | In –Plane Shear Modulus (±45°) GPa | 3 | 5.32 | 1.97 |
| 9 | Flexural Strength Warp MPa | 10 | 276.54 | 12.68 |
| 10 | Flexural Modulus Warp GPa | 10 | 16.57 | 3.8 |
| 11 | Flexural Strength Weft MPa | 10 | 301.78 | 13.23 |
| 12 | Flexural Modulus: Weft GPa | 10 | 17.53 | 6.78 |
| 13 | ILSS : Warp MPa | 25 | 33.45 | 3.58 |
| 14 | ILSS : Weft MPa | 25 | 21.89 | 6.39 |

### 3.4 Ablative performance by oxy-acetylene torch test

Oxy-acetylene torch test will be conducted on test specimen in order to qualify the ablative liner. Three samples were tested. The details on the preparation of the test specimen, test set-up, test procedure and acceptance criteria are laid down below. The test method is planned according to ASTM E285-80 (1996) standard [7]. The test involves in directing hot gases produced by the combustion of oxygen and acetylene gases along the normal to the test specimen using a welding torch until burn-through is achieved. Since the combustion gas more closely resemble the environment generated in rocket motors this test method is more appropriate for evaluating the ablative characteristics of thermal insulation materials. The erosion rate of the material and the Insulation index numbers are determined as explained in the following Paragraph.

### 3.4.1 Test specimen

Test specimen will be prepared in the form of flat plate of size 125 X 125mm approx (to sit firmly in specimen holder) and uniform thickness of 6±0.2mm using the prepreg belonging to the same batch as that used for manufacturing the ablative liners. Prepreg pieces of appropriate size will be cut and stacked to prepare laminate. Four replicates of the specimen shall be prepared and cured along with ablative liner in the autoclave.

### 3.4.2 Test procedure

Test shall be carried out on at least 3 replicates of specimen.Measure the dimensions and density of the specimen. Thermocouple lead shall be fixed near the centre of the back face of the specimen. Initially hold the specimen in the specimen holder and align the torch such that the distance between the front surface of the specimen and the torch tip is 20.0±1.0 mm and the torch tip is perpendicular to the front surface of the specimen. Remove the specimen from the specimen holder.Adjust the pressure regulators to the following conditions. Ignite the torch and stabilize the flame

a) Acetylene inlet pressure to the flow meter-1.0±0.1 bar

b) Oxygen inlet pressure to the flow meter 4.5±0.3
c) Acetylene flow rate -10.0 Lpm
d) Oxygen flow rate -12.0 Lpm
Record the pressure at the outlet of the flow meters.Swiftly slide the specimen in to the specimen holder and simultaneously start the timer.
Record the following

a) Time for back face temperature change. 80°C, 180°C and 380°C
b) Time for burn-through

### 3.4.3. Erosion rate

Calculate the erosion rate for each replicate by dividing the original thickness of the specimen by the time to burn-through as follows

$$E = \frac{d}{t} \quad (9)$$

Where E = erosion rate, mm/sec
d = thickness of the specimen, mm, and
t = burn-through time, sec

### 3.4.4 Average erosion rate

Calculate the average erosion rate as follows,

$$E\ avg = \frac{\Sigma E}{N} \quad (10)$$

E avg = average erosion rate mm/sec
Σ E = Sum of the individual values of erosion rates
N = number of replicates.

The erosion rate is 0.0861mm/sec. Back wall temperature Vs time is shown in Fig. No. 03
Oxy–Acetylene Torch Test set up is shown in the Fig. 4.

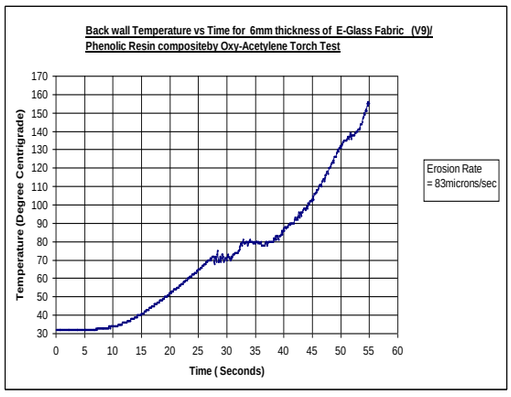


**Fig. 3 Back wall Temperature Vs Time of Composite by Oxy –Acetylene Torch Test**

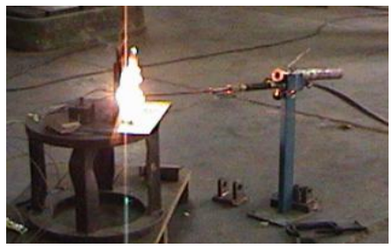

**Fig. 4 Oxy-Acetylene Torch Test Set Up**

 

## 4. Test Results and Statistical Analysis

All the tests were carried out using Universal testing machine (Instron UTM 4505) Composite materials are sensitive for their variability in mechanical properties. The variability is even more prevalent in carbon phenolic composites due to their inherent flaws and in homogeneities. It is important, therefore to present the spread of results, which is in itself a property of the material. Mechanical properties are given as the arithmetic mean (X) of a sample of n specimens, where n must be greater than 10 and preferably around 30; then

$$x = \sum (Xn/n) \quad (11)$$

The standard deviation $\sigma_{n-1}$ is then calculated using the following formula:

$$SD = (\sum (xi^2 - nx^2) / (n-1)) ½ \quad (12)$$

The spread of results are then expressed in terms of 'Coefficient of Variation', which is defined as percentage

$$CV = (SD/x)*100\% \quad (13)$$

Mechanical properties obtained are summarized and given in the Table No 06

## 5. Conclusions

E-Glass fabric/ Phenolic resin composite was characterized for all physical, Mechanical and Thermal properties for generation of design inputs for design and analysis of ablative composite liners for flex nozzle and jet deflector.

For each mechanical property number of replicate specimens was tested. Mean values and standard deviation is calculated. Coefficient of variation was found between 5-10%. Based on the confidence level, the designers can utilize the generated data for design and analysis of ablative composite liners for flex nozzle and jet deflector.

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