ON FABRICATION AND TESTING OF GLARE

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ABSTRACT

Various aspects related to fabrication and testing of fiber metal laminate (Glare) comprising 2014-T6 aerospace aluminum alloy sheets alternately bonded with, epoxy resin impregnated, E-glass fiber based composite prepregs are discussed in the paper. Procedures adopted in processing of laminate ingredients and in fabrication of the laminate are elucidated. Experimental techniques for measurement of mechanical properties of Glare v*iz.* tensile, flexural and shear strengths and interlaminar fracture toughness are reviewed. Pertinent results are presented. Energy dispersive X-ray spectroscopy of aluminum alloy and optical microscopy and residual stress measurement in aluminum layer of the fabricated laminate are touched upon. Viability of laminate fabrication method is proved by theoretically checking the quality of interfaces between un-identical material layers of the laminate.

Keywords: fiber metal laminate, glare, Inter-laminar fracture toughness, strength properties.

1. INTRODUCTION

Fibre metal laminate (FML) is an advanced hybrid material system that consists of layers of thin and light metallic sheets which are alternately bonded and cured with composite prepregs by heat and pressure, each prepreg built up of several resin impregnated unidirectional fiber layers laid in similar or different orientations. Besides offering gain in specific strength, FML's exhibit properties like excellent fatigue and fracture resistance, reasonably good impact strength and high fire resistance that make them a better substitute, even at increased costs, for monolithic metals and their alloys in aerospace and aircraft applications. Various types of FML's containing different types of fibers in their prepregs have been successfully tried and tested over the years *viz.* Arall with kevlar, Care with carbon and Glare with glass fibre. Among all the FML's, Glare has proved to be reliable and promising because of flexible and superior properties of glass fiber, especially of S-glass fiber, in it.

Hitherto, several research results have been reported on fabrication and mechanical tests of FML's. Notably, Kawai *et al.* [1] presented off-axis inelastic strength behavior of Glare. Khalili *et al.* [2] tested FML samples obtained from various lay ups of glass fiber/epoxy with steel and aluminum sheets and compared their strengths with each other and with monolithic metals and conventional fiber-resin composites (FRP's). Moussavi-Torshizi *et al.* [3] examined tensile properties of Arall and Glare. Tamer Sinmazcelik *et al.* [4] investigated surface treatment procedures of metals, their bonding techniques with composites and tensile properties of various types of FML's. However, there is need to further collect and consolidate the fabrication data and test results of FML's that can act as a benchmark in mechanized mass manufacture of FML's that may be necessitated by their potential of use in many upcoming strong and light weight commercial structures other than in the aerospace sector.

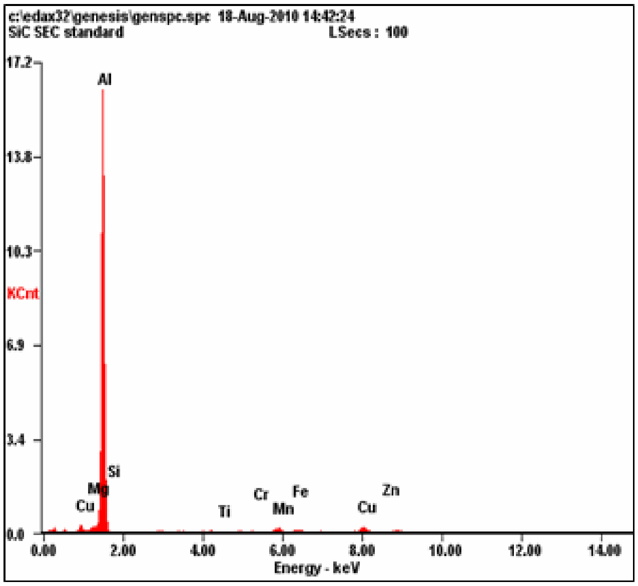
Glare, comprising thin 2014-T6 aerospace aluminum alloy sheets and E-glass fibre based composite prepregs, is considered for experimental investigations in the present work. Sequence of processing of ingredients and fabrication of the laminate is reviewed. Energy dispersive X-ray spectroscopy of parent aluminum alloy and optical microscopy and residual stress measurement in aluminum layer of the fabricated laminate are touched upon. Strength properties of Glare *viz.* tensile, flexural and shear are measured. Interlaminar fracture toughness of the laminate is also obtained.

2. FABRICATION OF GLARE

2.1. Processing of ingredients

2 mm thick, 2014-T6 aerospace aluminum alloy sheets were procured for fabrication of Glare. Chemical composition of the alloy was first checked by Energy dispersive X-ray spectroscopy (EDAX) before further processing of the sheets. The results, available at Figure-1, were as per expectations.

VOL. 9, NO. 9, SEPTEMBER 2014 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences



Element	Wt %	At %
MgK	01.19	01.41
AlK	86.55	92.67
SiK	00.06	00.06
TiK	00.13	00.08
CrK	00.09	00.05
MnK	02.68	01.41
FeK	01.37	00.71
CuK	07.79	03.54
ZnK	00.15	00.07

Figure-1. Composition test data (EDAX).

The sheets were subsequently cold rolled to desired thickness of 0.4 mm. Uni-axial tensile tests on cold rolled samples gave the following results:- Yield strength = 259.61 MPa, Ultimate tensile strength = 302.88 MPa, % elongation = 3 and Hardness (HV5) = 82.6, 81.6, 82.1. Deviation in results from T6 state was attributed to cold work. The sheets were therefore heat treated to achieve T6 state. Following heat treatment cycles, recommended for aluminum alloys, were tried on cold rolled samples:-

Heat treatment cycle No. 1

i) Stress relief annealing (The sample was heated to 315 deg. C followed by slow cooling)
ii) Solution heat treatment (The sample was heated to 495 deg. C and kept at that temp. for 1-2 hrs.)
iii) Air Quenching (The sample was cooled in air)
iv) Precipitation hardening (The sample was heated to 195 deg. C and kept at that temp. for 15 hrs.)
v) Cooling (The furnace was switched off and the sample was cooled to room temperature)

Mechanical properties of heat treated sample were: - Yield strength = 312.50 MPa, Ultimate tensile strength = 384.61 MPa, % elongation = 6.8 and Hardness (HV5) = 100, 103, 102. Since the values were again inferior than that in T6 state, a new heat treatment cycle No. 2 was adopted.

Heat treatment cycle No. 2

i) Stress relief annealing (The sample was heated to 380 deg. C, kept at that temperature for 4 hrs. followed by slow cooling)
ii) Solution heat treatment (The sample was heated to 502 deg. C and kept at that temp. for 30 min.)
iii) iii)Water quenching (The sample was quenched in cold water, time interval between removal of material from furnace and immersion in water was 10 seconds). The sample was gently struck with a mechanical tool for straightening in case of warpage noticed after quenching
iv) Precipitation hardening (The sample was heated to 160 deg. C and kept at that temp. for 18 hrs.)
v) Cooling (The furnace was switched off and the specimen was cooled to room temperature)

Mechanical properties of heat treated sample were: - Yield strength (MPa) = 372.00, 380.24 (Average = 376.12), Ultimate tensile strength (MPa) = 415.00, 419.75 (Average = 417.37), % elongation = 8, 8.4 (Average = 8.2) and Hardness in HV5 = 110, 109, 121 (Average = 113.33). The results matched well with T6 state. Therefore cycle No. 2 was adopted for heat treatment of bulk aluminum alloy for use in the laminates.

Anodizing and grit-blasting are two recommended surface treatment methods for aluminum alloy to increase their roughness for better adhesion and bonding with composite prepregs during fabrication of the laminate. However in the present work that was of limited scale the surfaces of aluminum alloy sheets were manually scratched by abrasive paper of 240 grit. Details of other ingredients used in the laminate are as follows:-

a) Fiber

Type: Unidirectional E-glass cloth, Thickness: 4 mil or 0.1 mm, Weave:Plain and Grammage:110 gsm

b) Resin

Type: Epoxy-CY 205/HY 905, Viscosity: 12000 cP, Index: 5.27

Properties of the laminate constituents are listed in Table-1.

VOL. 9, NO. 9, SEPTEMBER 2014 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

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Table-1. Material properties.

Property	2014-T6 Aerospace aluminum alloy, *al*	E-Glass fibre, *f*	Epoxy resin, *r*
Modulus of elasticity, MPa	72000.0	71000.0	3500.0
Shear modulus, MPa	27060.0	29710.0	1250.0
Poisson's ratio	0.33	0.22 (Major)	0.33
Yield strength, MPa	372.0	---	---
Ultimate tensile strength, MPa	415.0	3450.0	60
Percent elongation	8.0	4.8	4.0
Density, g/cc	2.71	2.45	1.54
Coefficient of thermal expansion, C^{-1}	23×10^{-6}	5.0×10^{-6}	57.5×10^{-6}

2.2. Prepegging and bonding

Prepregging was carried out in a prepreg machine with process temperature varying from 120 deg. C - 130 deg. C. Constituents of a prepreg are shown in Figure-2. A composite $c90$ layer was inserted between two composite $c0$ layers. Composite $c90$ had fibers oriented in x direction (along laminate width and transverse to the applied load) with thin resin layers on both the sides whereas composite $c0$ had fibers oriented in y direction (along laminate length and parallel to the applied load) with thin resin layers on both the sides. Approximate volume fractions of fiber and resin in each composite layer were 0.522 and 0.478 respectively. Prepregs, after preparation, were stored in a controlled environment prior to use in the laminates.

For laminate fabrication, three processed aluminum alloy sheets and two prepregs were cut to the desired size of 200 mm (Length) × 50 mm (Width). Each prepreg was alternately placed between two aluminum sheets. The assembly was loaded into hydraulic press that was preheated to 90 deg. C. Pressure of 10 bar was applied over the assembly. Temperature was raised to 160 deg. C and the assembly was cured for 3 hrs. After curing, the press was cooled to room temperature followed by the release of pressure. The assembly was taken out and visually inspected for defects. Expected thickness of the laminate was 2.346 mm. However, thickness varied between 2.2 mm to 2.9 mm due to uncontrollable manual application of resin over fiber cloth and also due to slight variation in thickness of aluminum alloy sheets induced during cold rolling. Fabricated Glare is displayed in Figure-3.

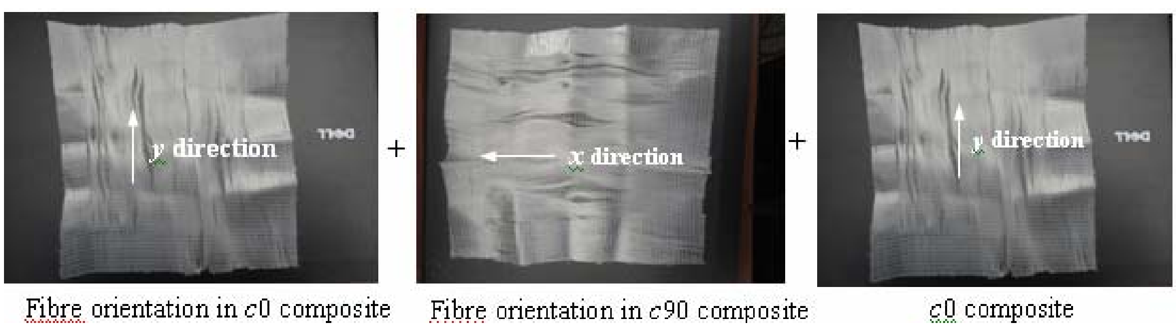

Figure-2. Ingredients of a prepreg.

VOL. 9, NO. 9, SEPTEMBER 2014 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

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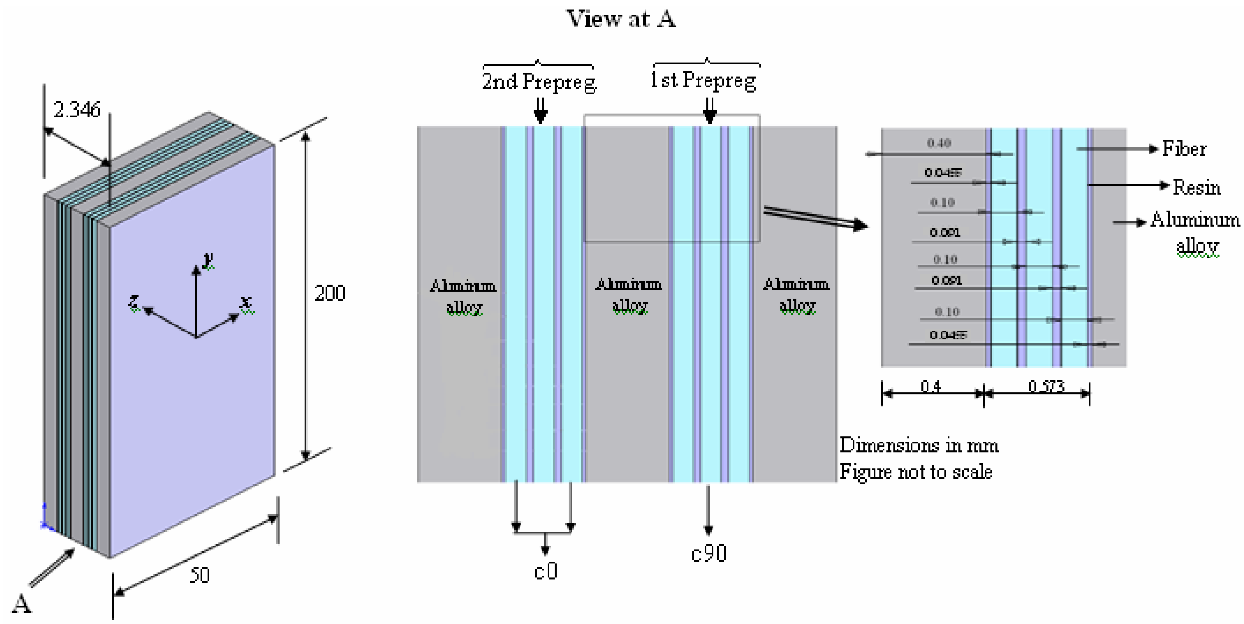

Figure-3. Fabricated glare.

3. TESTING OF GLARE

3.1. Optical microscopy and residual stress measurement

Before proceeding with mechanical tests of Glare, optical microscopy and residual stress measurement tests were undertaken on its external aluminum alloy layer.

In optical microscopy, 1% HF solution was used as an etchant to polish the surface of the aluminum layer. Micro-structure confirmed fine dispersion of $Cu\text{-}Al_2$ and Al-Si particles in the matrix of aluminum solid solution. The image is shown at Figure-4.

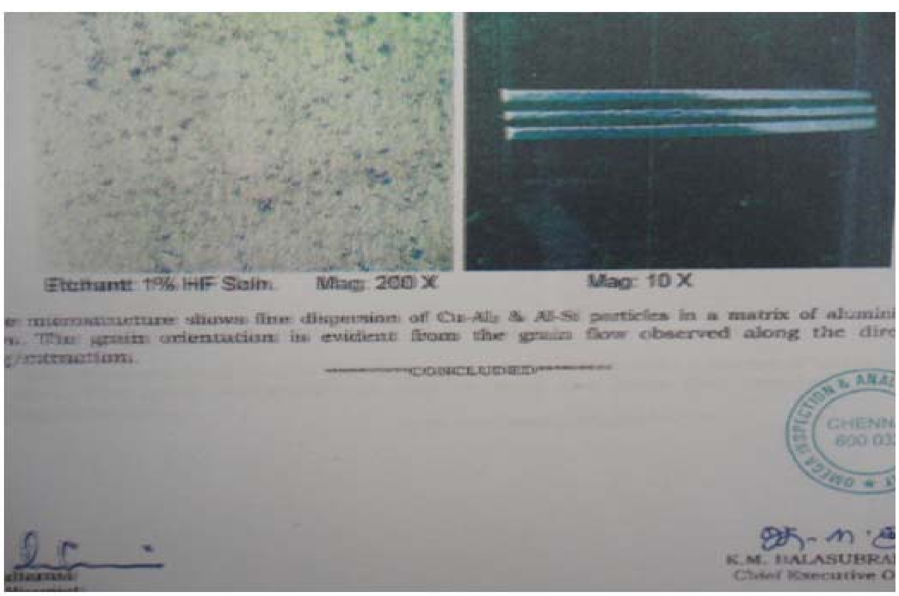

Figure-4. Optical image of aluminum alloy layer and of the laminate (from side).

The residual stress was measured in y direction at five different locations with the help of X-ray diffraction (XRD) technique. Following parameters were used in the measurement system:-

i) Radiation	CrK α
ii) 2θ	139.3 deg.
iii) Spot size	3 mm

VOL. 9, NO. 9, SEPTEMBER 2014 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

iv) Exp. time angles	15s, 3/3 tilts, -45/45 deg. psi
v) Calculation background	Cross correlation, linear
vi) Measurement method	Modified $d(\sin^2 \wp)$

Results were as follows: - (+ve: tensile, -ve: compressive)
Test laminate 1 (At five spots, MPa): +22.2, +36.9, +12.6, -0.5, +9.5
Test laminate 2 (At five spots, MPa): +14.3,-18.9, +24.9, -0.2, +17.3
(Average residual stress was +11.81 MPa, tensile)

3.2. Strength tests

Glare laminates were subjected to tensile, bending and shear loads. Test specimens were machined from the laminates for this purpose as per ASTM codes. Several specimens were used in each test to achieve consistency in test results. The laminates were also tested for inter-laminar fracture toughness. Test details and their outcome are as follows:-

a) Tension test (ASTM D3039): Refer Figure-5. Specimen of size, 200 mm$\times$30 mm $(m)\times$3 mm thk. (n), was loaded under uni-axial tension at test speed of 1-2 mm/min till fibers, following aluminum layers, failed leading to separation or fracture of the specimen. Engineering yield strength, σ_{ys}, and ultimate tensile strength, σ_{uts}, of the specimen were determined from measured yield load, P_{ys}, and ultimate tensile or break load, P_{uts}, respectively in the relation, $\sigma_{ys,uts} = \dfrac{P_{ys,uts}}{m \times n}$. Results of σ_{ys} (MPa), σ_{uts} (MPa) were 269.67, 360.30; 273.05, 365.13; 277.28, 370.62. Average values of σ_{ys} and σ_{uts} were obtained as 273.33 MPa and 365.35 MPa, respectively.

b) Flexural test (ASTM D790): Refer Figure-6. Specimen of size, 160 mm $\times$16 mm (m) $\times$ 3.2 mm thk. (n), was subjected to transverse load at test speed of 0-0.1 mm/min. Load was applied at the centre of the specimen till it achieved 5% deflection or failed earlier. Flexural strength, σ_f, was obtained from critical load, P_c, with the help of the relation, $\sigma_f = \dfrac{3 \times P_c \times X}{m \times n^2}$, where X was the distance between the supports. Values of σ_f (MPa) from various specimens were 728.80, 763.00, and 730.10. Average value was 740.63 MPa.

c) Short beam or inter-laminar shear test (ASTM D2344): Refer Figure-7. Very small specimen of size, 50 mm$\times$8 mm (m) $\times$3.2 mm thk. (n) was subjected to transverse load at test speed of 1 mm/min. Load was applied at the centre of specimen till it failed by the growth of delaminations. Shear strength, σ_{ss}, was found from the critical load, P_c, with the help of the relation, $\sigma_{ss} = \dfrac{P_c}{2 \times m \times n}$. Values of σ_{ss} (MPa) were 37.79, 40.04, 42.74. Average value was 40.19 MPa.

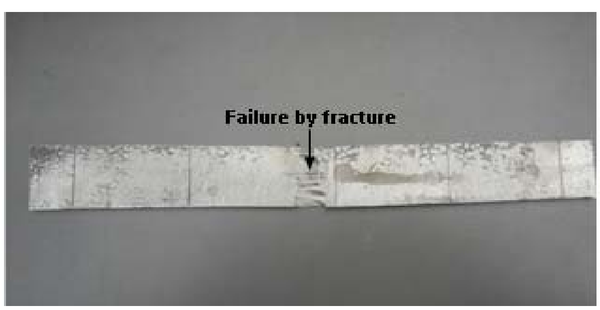

Figure-5. Tensile tested specimen.

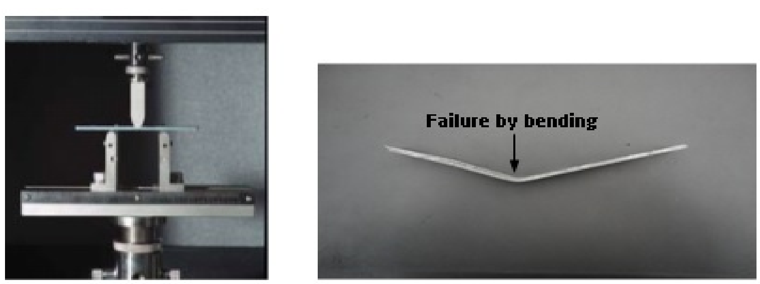

Figure-6. Flexural test arrangement and tested specimen.

VOL. 9, NO. 9, SEPTEMBER 2014 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

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Figure-7. Shear test arrangement and tested specimen.

3.3. Interlaminar toughness test

Refer Figure-8. End notched flexure (ENF) Glare laminates with single delamination on one side, single delamination on both the sides, double delaminations on one side and double delaminations on both the sides were tested for, Mode II, interlaminar fracture toughness, ζ_c. A prepreg was carefully pierced by a cutting tool to create a delamination. The delamination end or tip was found to be blunt due to the shape of the cutting tool edge. Separated material flaps on either side of the delamination were therefore gently gripped and opened in a controlled manner by tensile load for the delamination to grow by a small extent on its own as the Mode I type thereby creating a sharp interfacial crack tip needed for toughness tests.

Each delamination had the length of 50 mm app. (c) $\times$50 mm width (w). $2L$, the length of the laminate, was equal to 200 mm. Transverse load was applied at the centre of the laminate in a 3-point bend fixture till the delaminations became unstable resulting in separation of aluminum alloy layers in the laminate. Recorded load-displacement plots for all the types of delaminations are shown in Figure-9. Critical loads, P_c, were measured from the plots. Compliance, C, during each delamination growth was computed by dividing the value of displacement under load by magnitude of load in linear segments of the curves. ζ_c was approximated from $\zeta_c = \dfrac{9P_c^2 C c^2}{2w\left(2L^3 + 3c^3\right)}$ that is meant for delaminations in conventional fibre-resin composites (FRP's). Application of above equation to Glare was justified by the fact that the effect of larger bending and plastic deformation of aluminum sheets in Glare, that conventional composites don't exhibit, is included in the value of C. Toughness values obtained are as follows:-

Single delamination on one side

i) $C = 0.0138\text{mm/N}, P_c = 810\text{N}, \zeta_c = 850\ \text{J/m}^2$

Single delamination on both the sides

i) Delamination 1, $C = 0.00833\text{mm/N}$, $P_c = 600\text{N}, \zeta_c = 283\ \text{J/m}^2$

ii) Delamination 2, $C = 0.0266\text{mm/N}$, $P_c = 600\text{N}, \zeta_c = 907\ \text{J/m}^2$

Double delaminations on one side

i) Delamination 1, $C = 0.00177\text{mm/N}$, $P_c = 450\text{N}, \zeta_c = 34\ \text{J/m}^2$

ii) Delamination 2, $C = 0.0175\text{mm/N}$, $P_c = 510\text{N}, \zeta_c = 412\ \text{J/m}^2$

Double delaminations on both the sides

i) Delamination 1, $C = 0.00269\text{mm/N}$, $P_c = 260\text{N}, \zeta_c = 17.2\ \text{J/m}^2$

ii) Delamination 2, $C = 0.00833\text{mm/N}$, $P_c = 300\text{N}, \zeta_c = 71\ \text{J/m}^2$

iii) Delaminations 3 and 4, $C = 0.0233\text{mm/N}, P_c = 570\text{N}, \zeta_c = 717\ \text{J/m}^2$

Figure-8. Inter-laminar fracture tested speecimen.

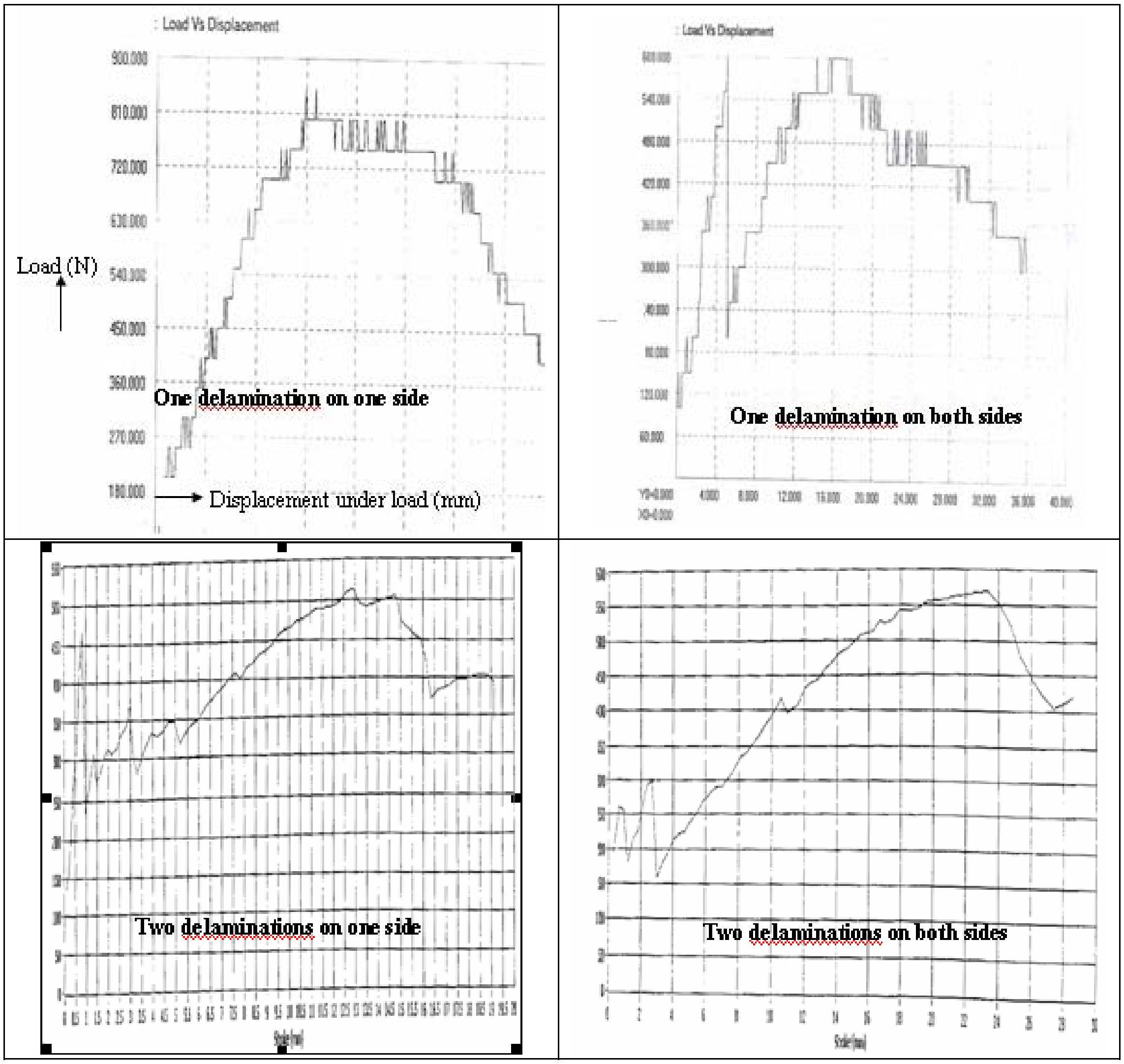

Figure-9. Load displacement plots of interlaminar fracture toughness tested specimens.

4. DISCUSSIONS

Optical microscopy confirmed grain structure of aluminum alloy. Mechanical properties of aluminum alloy, post heat treatment cycle No. 2, matched with the reported values of T6 state given in Table-1 that validated the heat treatment procedure adopted for property restoration of cold worked alloy. On the whole, good consistency in mechanical properties of Glare *viz*. tensile, flexural and shear strengths and inter-laminar fracture toughness supported the accuracy of the results.

Tensile yield strength of Glare was expectedly found to be less that that of plain 2014-T6 aluminum alloy because of stress enhancement in aluminum alloy layers of the laminate upon loading due to two reasons. Firstly, elastic redistribution of applied stress, that takes place in all elasticity mismatched material layers for maintaining same strain in all the layers, caused the stress in aluminum alloy layer to exceed the applied stress. Secondly, tensile residual stresses were inherently present in aluminum alloy layer that developed during curing of the laminate due to un-identical stiffness and coefficients of thermal expansion of the materials as seen in Table-1. Since aluminum alloy layers in the laminate failed first during tensile test of the laminate, residual stress and elastic stress redistribution in aluminum alloy layer of the laminate is estimated as follows:-

Residual strain, $\{\varepsilon\}_{rs}$, in aluminum alloy layer is found from

$$\{\varepsilon\}_{rs} = \left[\begin{Bmatrix} \alpha_x \\ \alpha_y \\ 0 \end{Bmatrix}_{al} - \begin{Bmatrix} \alpha_{tl} \\ \alpha_{ll} \\ 0 \end{Bmatrix} \right] \times \left(T_{cuiring} - T_{ambient} \right)$$

where α_{tl} and α_{ll} are transverse and longitudinal coefficients of thermal expansion of the laminate in x direction and in y direction respectively, T_{curing} and

$T_{ambient}$ being curing and ambient temperatures respectively. The values of α_{tl} and α_{ll} obtained from the fundamental equations of composites and laminates are $19.77\times10^{-6}\mathrm{C}^{-1}$ and $19.4\times10^{-6}\mathrm{C}^{-1}$ respectively. Knowing residual strain, residual stress, $\{\sigma\}_{rs}$, in aluminum alloy layer is determined by Eq. (1)

$$\{\sigma\}_{rs} = \{\mathrm{M}\}_{al}\times\{\varepsilon\}_{rs} \qquad (1)$$

Stiffness matrices of materials, $\{\mathrm{M}\}$, in plane stress condition is found as follows:-

$$\{\mathrm{M}\}_{al} = \begin{bmatrix} 80.79 & 26.66 & 0 \\ 26.66 & 80.79 & 0 \\ 0 & 0 & 27.06 \end{bmatrix}\text{GPa}, \quad \{\mathrm{M}\}_{r} = \begin{bmatrix} 3.92 & 1.29 & 0 \\ 1.29 & 3.92 & 0 \\ 0 & 0 & 1.25 \end{bmatrix}\text{GPa}$$

and $$\{\mathrm{M}\}_{f} = \begin{bmatrix} 74.61 & 16.41 & 0 \\ 16.41 & 74.61 & 0 \\ 0 & 0 & 29.70 \end{bmatrix}\text{GPa}$$

Stiffness matrix of the laminate is determined by classical theory,

$$\{\mathrm{M}\}_{lam} = \{\mathrm{M}\}_{al}\times\frac{t_{al}\times3}{d}+\{\mathrm{M}\}_{r}\times\frac{t_{r}\times12}{d}+\{\mathrm{M}\}_{f}\times\frac{t_{f}\times6}{d},$$

where t_{al}, t_{r} and t_{f}, that represent thickness of an aluminum, a resin and a fiber layer in the laminate, are 0.4 mm, 0.0455 mm and 0.1 mm respectively. Constants 3, 12 and 6 are total number of aluminum, resin and fiber layers respectively in the laminate and, *d*, is the thickness of the laminate that is taken as 2.346 mm.

$$\{\mathrm{M}\}_{lam} = \begin{bmatrix} 61.31 & 18.13 & 0 \\ 18.13 & 61.31 & 0 \\ 0 & 0 & 21.72 \end{bmatrix}\text{GPa}; \quad \{\mathrm{M}\}_{lam}^{-1} = \begin{bmatrix} 0.0178 & -0.0052 & 0 \\ -0.0052 & 0.0178 & 0 \\ 0 & 0 & 0.046 \end{bmatrix}$$

Strain in the laminate under applied stress, $\sigma_{applied}$, is obtained from

$$\{\varepsilon\}_{lam} = \begin{Bmatrix} \varepsilon_x \\ \varepsilon_y \\ \gamma_{xy} \end{Bmatrix}_{lam} = \{\mathrm{M}\}_{lam}^{-1}\begin{Bmatrix} \sigma_x \\ \sigma_y \\ \tau_{xy} \end{Bmatrix}_{applied}$$

.Value of redistributed stress, $\{\sigma\}_{ds}$, in aluminum alloy layer is found from Equation (2).

$$\{\sigma\}_{ds} = \{\mathrm{M}\}_{al}\times\{\varepsilon\}_{lam} \qquad (2)$$

Using T_{curing} as 160 deg. C, $T_{ambient}$ as 30 deg. C and applied stress, $\begin{Bmatrix} \sigma_x \\ \sigma_y \\ \tau_{xy} \end{Bmatrix}_{applied}$, over the laminate during tensile test, equal to average yield strength of the laminate i.e. $\begin{Bmatrix} 0 \\ +273.33 \\ 0 \end{Bmatrix}$ MPa, in Eq. (1) and Eq. (2), $\{\sigma\}_{rs}$ and $\{\sigma\}_{ds}$ are obtained as $\begin{Bmatrix} +46.17 \\ +48.36 \\ 0 \end{Bmatrix}$ MPa and $\begin{Bmatrix} 14.87 \\ +355.3 \\ 0 \end{Bmatrix}$ MPa respectively. Yield strength of aluminum alloy layer in the laminate, along the direction of applied load in *y* direction, is found by superimposing the corresponding values of $\{\sigma\}_{rs}$ and $\{\sigma\}_{ds}$. The value is obtained as +403.66 MPa (tensile) that is close to yield strength of +376.12 MPa (tensile) of plain aluminum alloy provided in Section 2.1 post heat treatment cycle 2. If actual or experimentally obtained residual stress values, provided in Section 3.1, are used, then the value drops from +403.66 MPa to +367.11 MPa which is more close to +376.12 MPa. These findings support the authenticity of magnitude of laminate yield strength. With this, the viability of laminate fabrication procedure is also proved. The important criterion of a good and acceptable laminate is strong and intact interfaces, between un-identical material layers, of the laminate that don't fail under applied stress. The phenomenon of stress redistribution verified above substantiate sound interfaces because stress redistribution can take place only when the interfaces are intact thereby allowing load transfer from one material layer to another without the layers separating from each other before the laminate finally yields and fractures.

5. CONCLUSIONS

Glare, comprising three thin aerospace 2014-T6 aluminum alloy sheets and epoxy resin impregnated unidirectional E-glass fiber based composite prepregs, is successfully fabricated and tested for mechanical properties *viz*. tensile, shear and flexural strength and inter-laminar fracture toughness. Consistency in results supports the accuracy of the values. Viability of laminate fabrication procedure is proved. Quality of interfaces between un-identical material layers of the laminate is tested theoretically and is found to be good.

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