Turkish Online Journal of Qualitative Inquiry (TOJQI)
Volume 12, Issue 6, July 2021: 5290-5298

MECHANICAL CHARACTERIZATION OF NANOSILICA DISPERSED EPOXY ADHESIVE FOR ALUMINIUM/GLASS FIBRE HYBRID LAMINATE

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ABSTRACT
Fibre Metal Nano composite Laminates (FMNL) are hybrid composites, fabricated by sandwiching metal (aluminium alloy) with fibres (E-glass). In this work, aluminium alloy and GFRP (Glass Fibre Reinforced Polymers) are used as adherends and epoxy (Araldite LY 556) is employed as an adhesive. Such an FML can be termed as Glass Laminate Aluminium Reinforced Epoxy (GLARE), in which thin aluminium alloy sheets are bonded on either sides of Glass Fibre Reinforced Polymer (GFRP). Airplane fuselage and wing structures makes use of GLARE due to their superior mechanical properties. The most important objective of this work is to analyse the effect of dispersed Nano silica fillers in the epoxy resin. The dispersion of nano fillers in the epoxy resin matrix is over a varying weight percentage of 0 to 7 to form different hybrid laminates. The formed laminates are tested for mechanical properties such as tensile, interlaminar shear, flexural and impact strength. Hybrid laminate with superior mechanical property and enhanced adhesive capacity compared to the other laminate samples are identified.
Keywords: Nano-silica; hand lay-up; Al/GFRP laminate; interlaminar strength; Adhesive bonding.

1. INTRODUCTION

Composite materials have characteristics that varies from individual components. In general, two or more individual components having different physical and chemical properties are the constituents of composites[1]. In general, composite materials are produced to significantly improve a particular property of interest. Usually, an increase in the property of interest by 5 times is desired for the new material to be classified as a composite.

Botelho et al. [2] studied that the hybrid fibre metal laminate is a composite material made from a combination of metal and polymer composite laminates. These laminates were prepared to form a class of materials with enhanced mechanical properties as compared to that of metal or fibres. These new hybrid laminates can be used even in high-temperature application owing to their high thermal stability due to the incorporation of metal in the composite material. Vlot et al. [3] investigated that Fibre Metal Laminate material consisting of bonded thin metal sheets and composite layers. The combination of advantageous properties like fatigue and impact tolerance of laminated structure provides the fibre metal laminates (FML) as a primary material in aerospace industry.

Sinmazçelik et al. [4] reviewed the classification of FML as ARALL, GLARE and CARALL and explained the importance of their key constituents: metals and fibre reinforced laminate. A detailed study on classical bonding techniques has also been reported. A significant property of fibre metal bonded laminates was that they were better at fatigue resistance than the normal mechanical bonding laminates. Katnam et Al. [5] has studied that this type of bound laminates have improved characteristics on handling stress. For instance, they have improved stress distribution on fastening and riveting, high fatigue resistance and are found to be compatible with all advanced materials. L.F.M. da Silva and R.D.S.G. Campilho [6] have also reported similar findings. They have stated that the use of such fibre bound laminates has reduced or eliminated the use of mechanical fasteners in laminates.

Asundi and Choi [7] have replaced the conventional composite laminate by hybrid laminate and have found that they have higher specific strength and stiffness. This makes hybrid laminate a suitable light weight replacement for the former. This makes them suitable for high fatigue performances. In FML using of aluminium as a metal component is termed as the GLARE laminate. Sadighi et al. [8] proposed that GLARE is classified based on the

fibre orientation. These grades are all based on various lay-ups of fibre epoxy prepreg layers composed of unidirectional glass fibre embedded in the adhesive. It is possible to stack prepreg layers with different fibre orientations in between two aluminium layers, resulting in different standard GLARE grades as unidirectional varients (GLARE1&2) and biaxial varients (GLARE3&5).
Krishnakumar [9] has observed that GLARE offers better mechanical properties, maintainability, higher resistance towards impact and environmental degradation, in comparison to composites. The unique arrangement of alternate layers of aluminium and composite prepreg, fibre metal laminates are durable and damage tolerant than aluminium or pure composites. Bhat and Narayanan [10] studied that GLARE, having aluminium alloy sheets and epoxy resin with unidirectional E-glass fibre-based composite prepreg has better fatigue properties and is successfully fabricated. Park and Choi [11] introduces a brief overview of the Mechanical behaviours of GLAREs. It is found to possess a large number of favourable characteristics, such as low density, static strength, high impact, and flame resistance. These properties have made them suitable for aircraft applications.

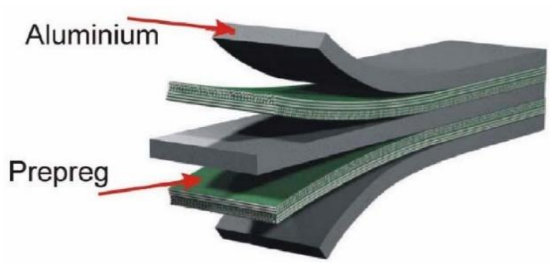

Fig 1. Layup Configuration of Glass laminate aluminium reinforced epoxy resin.[12]

Addition of nanofillers or nano fibres to the FML as adherend or as a part of adhesive is found to have gained significant interest in the recent times. Nanoparticles are added in the adhesive to enhance their bonding capacity, as addition of nanofillers would result in availability of increased bonding area. This, in turn would result in improved mechanical properties of the laminates. Saraç et al. [13] make use of nano-Al_2O_3, nano-TiO_2 and nano-SiO_2 as nanoparticle reinforced epoxy adhesive and stainless steel plates as adherend. It was reported that there was significant improvement in the failure load of nanofiller reinforced adhesive joint as compared to that of conventional resin joints.
Akpinar et al. [14] investigated the effects of nanocomposite adhesives obtained by adding Graphene, Carbon Nanotubes and Fullerene to the rigid, flexible and toughened adhesives at three different ratios on tensile failure load in single lap-joint geometry. In this study, the Nanosilica (nanofillers) was dispersed in epoxy resin and to study its effects on the failure loads and curing time. This nano fillers dispersed resin was used to fabricate an aluminium – GFRP hybrid laminate. The nanofillers compositions are varied from 0 to 7 wt. % in the dispersed epoxy matrix to form different hybrid laminates and their properties are tested to achieve an optimal mix.

EXPERIMENTAL WORK

2.1. Materials

Commercial Aluminium alloy 1100 sheet of thickness 0.8 mm are used as metal adherend, which is procured from Bharat aluminium Dealer Secunderabad, India. The woven roving E-glass fibre mat of 610gsm is used as the main reinforcement (fibre adherend). Nano-silica is used as a secondary reinforcement and a filler on mixing with epoxy (Araldite LY 556) resin. The prepared mixture of resin and filler is employed as an adhesive material. Hardener (Aradur HY 951) is chosen to aid curing.

2.2. Preparation of nano-silica reinforced epoxy adhesive

Nano-silica powder of size 17 nano metre is directly mixed with epoxy resin to obtain a reinforced epoxy resin adhesive. This mixture is rendered homogeneous on stirring at 350rpm for two hours. Various weight percentages of nanofiller (nanosilica), namely 0, 3, 5 and 7 wt% are dispersed in the epoxy matrix to prepare nanosilica reinforced epoxy adhesives. Additive BYK 410 is used to remove the air bubbles and stabilize the system. The stabilized adhesive system is kept at room temperature. At time of fabrication hardener HY 951 of 10 wt% is mixed with the resin and used.

2.3. Surface treatment of aluminium alloy adherend:

The chemical composition of Aluminium alloy is verified using Energy Dispersive X-ray Spectroscopy (EDS) before being used for laminate. EDS of Bruker make is used at a resolution of 20.0 kV.
The result shows that weight percentage of aluminium is observed to be maximum of 85.89 Wt%, confirming that the obtained alloy is of Aluminium alloy series 1 are shown in Table 1.

Table 1. Chemical Composition of AA1100.

Element	Atomic Number	Series	Wt%	At%
Al	13	K	85.89	74.77
C	6		11.43	22.35

O	8		1.46	2.15
Fe	26		0.68	0.29
Si	14		0.54	0.45

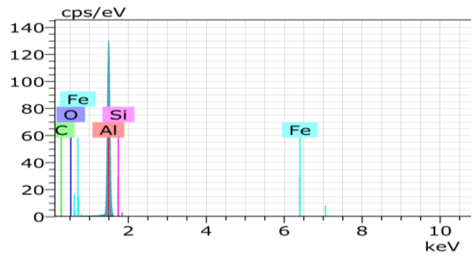

Fig-2. Composition analysis of AA1100 by EDS

To obtain the best adhesive bonding, the aluminium alloy sheet is subjected to mechanical treatment. The faying surface of the aluminium sheet is polished using 80-grade emery paper, to remove contaminants like oxide layer, dirt and grease, followed by cleaning employing acetone as an organic solvent. The mechanically treated and cleaned surfaces of aluminium alloy adherend are characterized by FESEM.

The FESEM images of the pre-treated Al alloy adherend, at different magnification have indicated a significant improvement in the adherend surface area. Figure 3 shows the surface of raw material procured. It can be observed that the surface is uneven with occasional smooth and ridge patches. This uneven surface might result in uneven bonding among adherend and adhesive. Figure 4 indicates that mechanical polishing has generated parallel scratches with hill and valley pattern on the surface of Aluminium alloy sheet.

As the adhesive capacity of a material is highly dependent on bonding surface area, the pre-treatment of aluminium sheet is found to be successful.

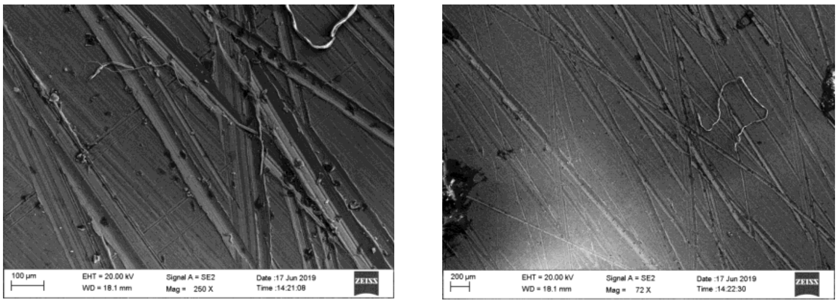

Fig. 3. Procured Aluminium alloy sheet before treatment

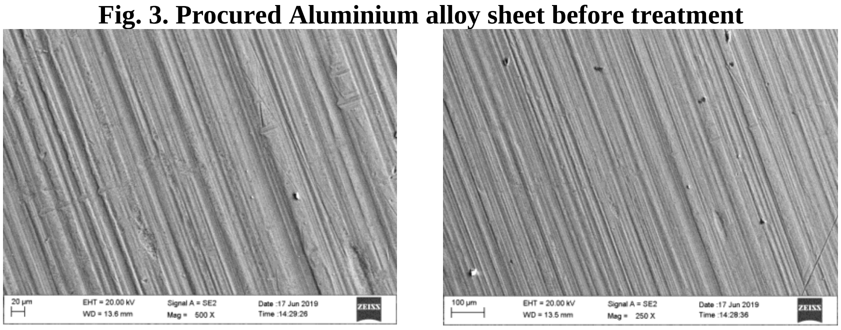

Fig. 4. Mechanically treated Aluminium alloy sheet

2.4 Preparation of Al/GFRP Laminate

The laminate specimen is prepared with a dimension of 300mmx300mmx0.8mm, keeping the thickness of the sheet as a constant factor. GFRP is prepared as an instantaneous laying process instead of prepreg. The entire laminate is prepared by hand lay-up technique, by stacking three layers of the woven roving mat (WRM) in between aluminium sheet. Nanofillers dispersed epoxy resin is used as an adhesive in between the layers of WRM and Aluminium sheets.

The plain surface of the aluminium sheet and the inner surface of the mold cavity are coated with wax. The aluminium sheet is placed inside the mold such that the plain surface and inner surface of the mold touches each other. The faying suface of the aluminium sheet faces upwards. A coating of nano-silica resin and hardener mixture is applied over the faying aluminium surface. Finally, WRM glass fibres are placed over the above coating[15]. Three layers of WRM fibres wetted with nano-silica resin-hardener mix are then placed over the Aluminium sheet.

Over the last layer of WRM glass fibre, aluminium sheet is placed and the laminate gets a complete structure. The mold cavity is sealed by a wax coated plastic film. To obtain uniform pressure, weights are placed over the laminate. The setup is allowed for curing for 12 hours at room temperature. The above mention step of preparation Al/GFRP laminate are shown in the following figure 5. The final laminate obtained is subjected to water-jet cutting process to get the required specimen dimension for mechanical testing.

Fig. 5. a) Abrasion on aluminium sheet b) Cleaned faying surface of Al c) Applying Epoxy Mix on Al sheet d) Glass fibre placement on Al e) Applying epoxy resin on the Glass fibre f) Removing excess resin using roller.

The overall laminate thickness of aluminium and FRP layers is specified in Table 2.

Table 2. Overall Laminate Thickness

Sl. No.	Aluminium thickness fraction	Aluminium (mm)	FRP (mm)	Total Thickness (mm)
1.	0.5	0.8+0.8	1.4	3

The aluminium thickness fraction is calculated by dividing the thickness of aluminium and the total thickness of the hybrid laminate[16]. Aluminium present in the laminate is about 50% by volume.

2.5 Characterization of Al/GFRP Laminate

2.5.1. Tensile test

Universal tensile testing machine with a capacity of the 30KN machine at a crosshead rate of 2 mm/min is used to conduct tensile test. Specimens are prepared as per ASTM standards with dimensions of 250 mm in length, 25 mm in width and 3mm in thickness. Gauge length of 200mm is provided. The tensile properties like deformation, stress and strain of the fabricated Glass Laminate Aluminium Reinforced Epoxy (GLARE) are determined as per ASTM 3039 test standard specifications. The average tensile properties are determined from 4 specimens. Specimens are mounted on the both the grips of a universal tensile tester and load is applied gradually from zero until the specimen fails. The ultimate strength that the material can withstand is determined from the maximum load carried by the specimen before failure. The results are obtained from the graph generated by the tensile tester.

2.5.2. Flexural test

The test for bending strength is conducted in the Universal tensile tester with a capacity of 30KN. Four specimens of dimensions 160mm length, 16mm width and 3 mm thickness are prepared according to ASTM D790. Flexural load is applied at a rate of 2mm/min. Flexural strength of the hybrid laminate composite is determined using this standard specimen and the average flexural results are determined from four specimens. 3 point bending test is widely adopted in industries since the fixtures used and specimen preparation procedure are simple.

The specimen is placed on the stage having simple supports on which transverse load is applied gradually. This three point bending setup is shown in figure 6.

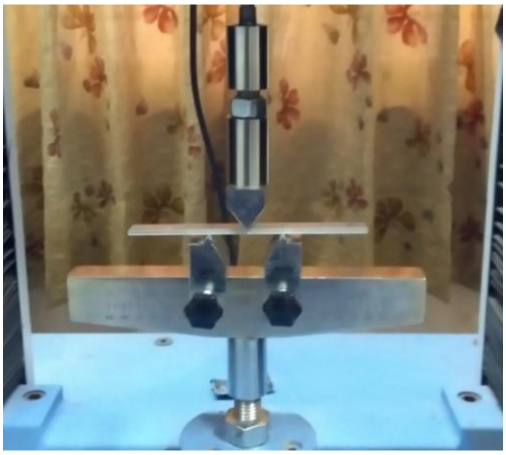

Fig. 6. Flexural test set-up

2.5.3. Lap Shear Test

To find the bonding strength between layers of aluminium sheet and GFRP, lap shear test is conducted in the Universal Tensile Tester with a capacity of 30KN. Specimens are cut with water jet cutting process for 200 mm long, 25mm wide and 3mm thickness, with a span length of 130mm. Shear force is applied at a rate of 2mm/min. Shear test is conducted to find the adhesive strength between adherend and adhesive. Double not shear technique is predominantly used in this procedure. Two notches of size 25mm x 25mm is made on the aluminium layer in order to facilitate the application of shear force. The prepared specimen is clamped in the tensile grip holder along the notch. Gradual tensile load is applied on the specimen along its axis till point of failure.

2.5.4. Charpy Impact Test

To find the toughness of laminate, the Charpy impact test was carried out as per ASTM A370 with standard subsize dimension as 55mm long, 10mm width and a notch in the middle of the specimen. The hammer in the pendulum is clamped at 120^0 with respect to vertical post. At this position the hammer stores energy of 300J which is noted as the initial value. After the pendulum is released to hit the specimen, the value indicated by the pointer is noted as final reading. The average value of the test is concluded from four specimens each for all the four weight percentages to find the impact energy absorbing capacity of the specimen.

The absorbed impact energy and the impact strength of the materials are calculate by using the formula,

Absorbed impact Energy, Ea = Ei - Ef

Impact strength, U = Ea/b*t

Where Ea is the absorbed impact energy, Ei is the Initial Potential energy, Ef is the final potential energy, b and t represents width and thickness of the specimen.

2. RESULTS AND DISCUSSION:

3.1. Tensile Test

The specimens used for tensile test are shown in Fig. 7.

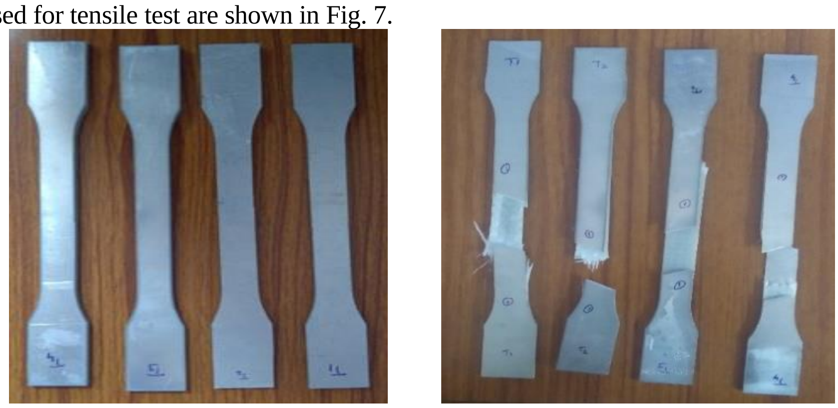

Fig. 7. Specimens for tensile test before and after test

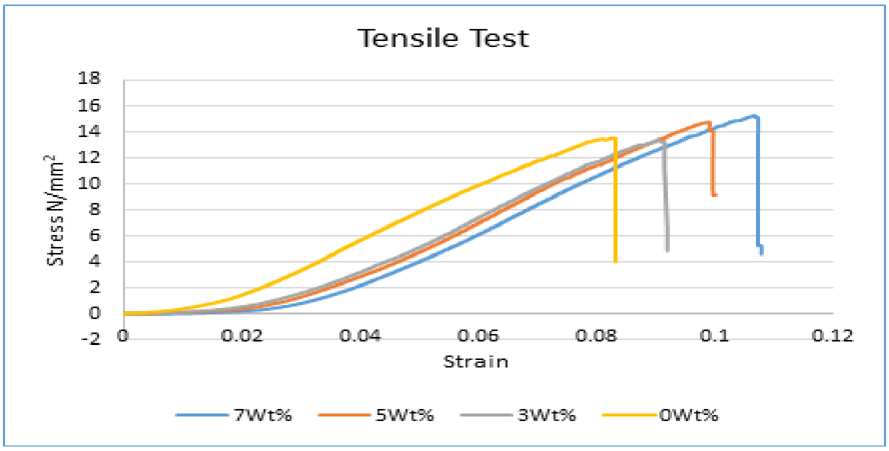

Fig. 8. Stress vs. Strain of Tensile test

The Tensile test result graph is shown in figure 8 which shows the stress vs. strain (displacement) curve of glare sheet with same thickness & different Weight percentage of nano-silica. The specimen is observed to delaminate at the initial stages. This is followed by yielding and failure of the GFRP layer in the hybrid laminate, and finally the aluminium layer fractures. The maximum force that the sample can withstand before failure, tensile strength and yield point of each sample has been enlisted in table 3.

Table 3. Tensile Test results

Specimen	Max. Force (N)	Tensile strength at yield (MPa)	Percentage increase in tensile strength
0 Wt% Nano-silica	9964.54	190.39	-
3 Wt% Nano-silica	9777.46	217.28	14.2
5 Wt% Nano-silica	10859.88	241.33	26.8
7 Wt% Nano-silica	11240.38	249.78	31.2

From the tabulation 3, it is clear that the tensile strength of the specimen is directly proportional to the percentage of nano-silica in the laminate. Accordingly, maximum tensile strength in yield of 249.78 MPa has been observed for the sample with 7 wt% of nano-silica in the resin. This increase in strength can be attributed to the increase in the strength of the polymerizing bond, due to the addition of nanofillers.

3.2. Flexural test

Fig. 9. Flexural test Specimen Before and after test.

Table 4. Flexural Test results

Specimen	Max. Force (N)	Max Flexural strength (MPa)	Percentage increase in flexural strength
0 Wt% Nano-silica	291.46	145.73	-
3 Wt% Nano-silica	346.43	173.21	18.9
5 Wt% Nano-silica	377.94	188.97	29.7
7 Wt% Nano-silica	414.8	207.4	42.3

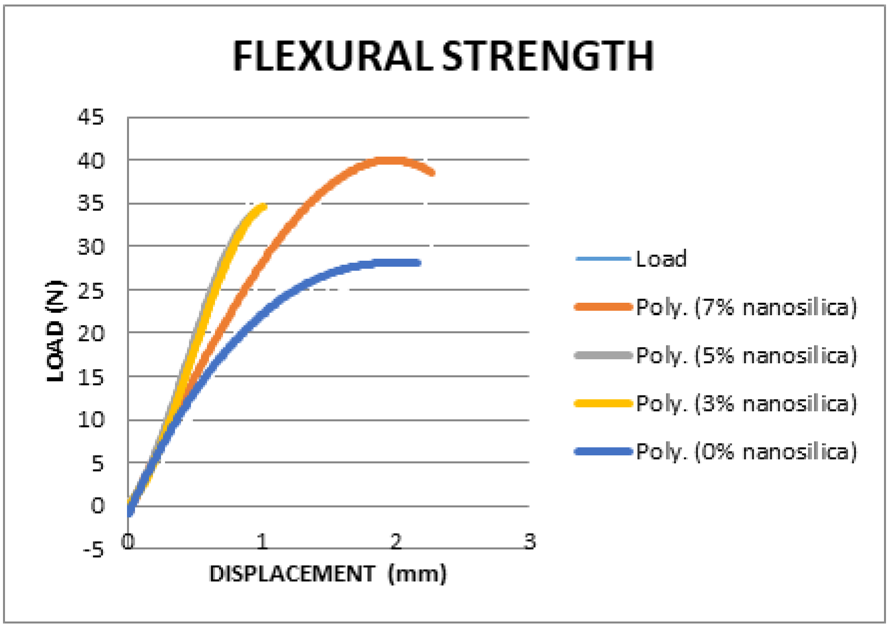

Fig. 10. Load vs Displacement Graph for Flexural test

The specimen before and after testing is shown in figure.9. The result obtained is shown in Table 4, which contain the maximum force that a material can withstand before it yields due to transverse load. The Figure 10 shows the load displacement plot of the specimens 0, 3, 5 and 7 wt % The actual values plotted in the graph are approximated to obtain smooth polynomial curves of degree 3 The main mode of flexural failure is observed to be delamination. No significant changes like fracture or breaking in the aluminium or GFRP sheets are observed during the test. The ductility of aluminium resists the fracture hence allows bending of the entire laminate. Layering of glass fibre and resin having lesser stiffness is used instead of pre-preg having higher stiffness, the entire laminate bends and delaminates but does not breaks.

It can be observed that the samples of 3, 5 and 7 wt % have better flexural resistance than pure GLARE sample, which concludes that the flexural strength increases as the percentage of nanofiller in the resin matrix increases. As a result, the load at which delamination occurs among the four samples is found to increase as the percentage of nano-silica increases. From the polynomial graph constructed above, it can be observed that the laminate with 7wt% is recommendable for applications where bending occurs.

3.3. Lap Shear Test

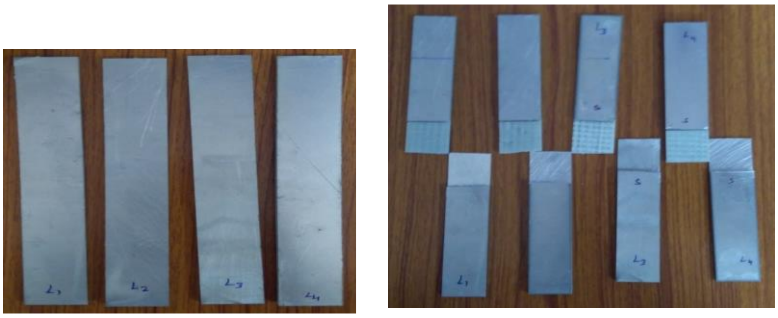

Fig. 11. Lap Shear test Specimen Before and after test.

Table 5. Lap Shear Test results

Specimen	Max. Force (N)	Max Shear stress(MPa)	Max. Elongation (mm)
0 Wt% Nanosilica	223.59	0.37	1.06
3 Wt% Nanosilica	425.61	0.68	1.69
5 Wt% Nanosilica	732.56	1.17	2.17
7 Wt% Nanosilica	1091.48	1.75	3.02

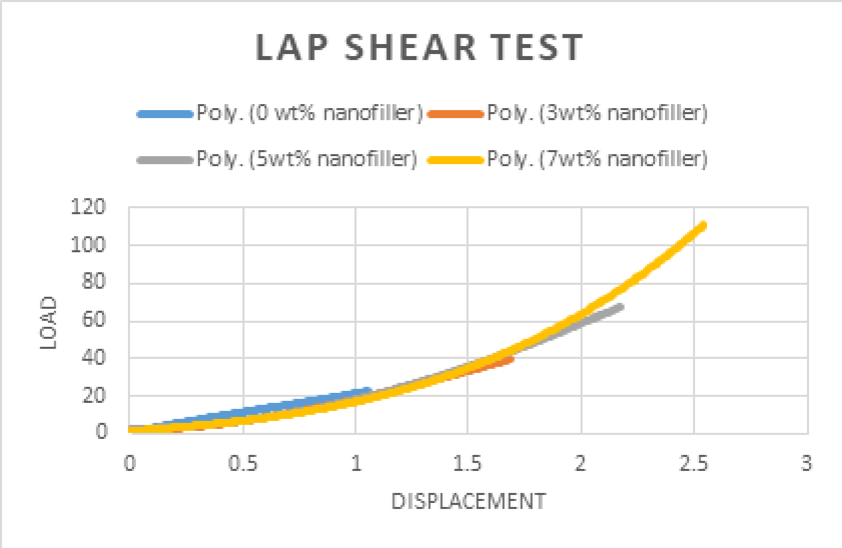

Fig. 12. Load vs Displacement graph for lap shear Specimen.

The samples before and after testing have been shown in the figure 11. On being subjected to lap shear force, the samples are split into two along the cross section. The main mode of failure observed is adhesive failure, in which the bonding between aluminium and GFRP sheets are found to be rendered null.

The result obtained from the test is shown in table 5, which shows the maximum force that the specimen can withstand, the maximum shear stress and the maximum elongation of the specimen. The graph is plotted for the load vs displacement for all the four wt% nanofiller reinforced epoxy resin as shown in figure 12. The results conclusively prove that the addition of silica nanofillers to the epoxy resin has improved the bonding capacity of resin. Inter laminar shear strength or maximum shear strength is observed for laminate with 7 wt % filler. A significant improvement of thrice the lap shear is seen in 7wt % in comparison to no silica filler was observed.

3.4. Charpy Impact Test

Fig. 13. Charpy Impact test specimen before and after impact.

Table 6. Charpy Impact test results.

Element	Average final reading	Absorbed energy (J)
0 Wt% Nanosilica	5	295
3 Wt% Nanosilica	4.7	295.3
5 Wt% Nanosilica	4.58	295.4
7 Wt% Nanosilica	4.2	295.8

The prepared samples before test and damaged samples after being tested are shown in Figure.13. The samples absorb kinetic energy of the hammer till its maximum strength and get collapsed after being hit and bends to a ‘v’ like form.

The relative impact toughness of a material is evaluated and the impact strength is shown in table 6. It has been observed that there is a slight increase in the absorbed energy with the increase in the percentage of nanofiller. The results indicate that the addition of nano-silica does not have a significant improvement in the impact test of the material. This might be due to the minimal interference of resin matrix on the impact test. Few of the impact damages observed are delamination, internal crushing and damage in bonding.

3. CONCLUSIONS

The mechanical properties of the nanofiller dispersed epoxy resin GLARE laminate with different weight percentages of nano-silica are studied and compared. The presence of the nanoparticles in the composite laminate enhanced the mechanical properties. From the results obtained from the mechanical test, the following conclusions are drawn:

- Tensile strength of the GLARE laminate has tremendously increased in 7 wt.% of nanofiller reinforced epoxy specimen.
- Similar improvement in flexural and shear strengths are also observed for 7 wt.% of nanofiller reinforced epoxy specimen.
- The presence of nanofiller is found to have very less effect on the impact strength of the laminates.

Abbreviations

ARALL	Aramid Reinforced Aluminium Laminate
CARALL	Carbon Rein-forced Aluminium Laminate
FML	Fibre Metal Laminate
GFRP	Glass fibre reinforced plastics
GLARE	Glass Laminate Aluminium Reinforced Epoxy

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