

## COMPOSITES OF NANO AL<sub>2</sub>O<sub>3</sub> FIBER-REINFORCED POLYMER SCRIPT AND CHARACTERIZATION

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DOI : <https://www.doi.org/10.56726/IRJMETS45228>

### ABSTRACT

The current work focuses on a hand layup approach for the manufacture and characterization of AL<sub>2</sub>O<sub>3</sub>-fiber-reinforced polymer composites. to understand the mechanical characteristics of an epoxy- and hardener-based E-glass (610-GSM) fiber-reinforced polymer composite. In order to create AL<sub>2</sub>O<sub>3</sub> powder, high-energy ball milling is used. The testing samples are made in accordance with ASTM-D-638, ASTM-D-790, and ASTM-D-785 standards, utilising five layers of E-glass fibre for each laminate and varying volume percentages (0%, 0.5%, 1%, 1.5%, 2%, and 2.5%). These samples go through tensile, flexural (three-point bend), and hardness testing. For all the laminates, the findings are contrasted within the volume percentage range (0%, 0.5%, 1%, 1.5%, 2%, and 2.5%). The many benefits of E-glass fiber-reinforced polymer composites, including their great wear resistance and high strength-to-weight ratio, have led to a wide range of applications. Results of the composite's tensile strength, flexural strength, and hardness were compared.

**Keywords:** Ultimate tensile strength, impact strength, hardness, DSC, DTA, TGA.

### I. INTRODUCTION

The creation and utilisation of composite materials is a very ancient technique. Modern composites are lightweight, high-strength materials with outstanding mechanical, thermal, and electrical qualities. These composite materials are widely employed in the maritime, aerospace, aircraft, automotive, and military industries due to their exceptional qualities. They are used to produce hulls, ships, submarines, and ship components. Due to its exceptional adhesion, chemical resistance, high strength and modulus of elasticity, low shrinkage, and ease of processing [1], epoxy is the most extensively used structural polymer. Various nanocomposites and nanohybridized glass fiber-reinforced composite laminates were fabricated using nanophased epoxy[2]. The glass fibre is formed of extremely fine glass fibres that are low in density, stiff, and elastic. Due to their simplicity of manufacture, low cost, and great strength and stiffness, almost 95 percent of the fibres used in polymer matrix are glass fibres [3]. Polymer composites with two or more different reinforcements are hybrid composites. In composites, natural and synthetic fibres are sometimes combined in hybrid form to maximise the benefits of both fibre types, a process known as hybridization [4]. Polymers are advantageous because of their chemical resistance, low density, improved wettability, and ease of moulding into desired and custom shapes [5]. Polymer matrices filled with micro- and nanofiller composites have increased in popularity due to their superior strength-to-weight and stiffness-to-weight ratios [6]. Oil and gas pipelines, as well as other steel structures, are commonly coated with epoxy-based coatings. In addition to its superior performance in terms of corrosion protection, weather resistance, and humidity resistance [7, 8]. Thermosets are typically formed of a liquid mixture of monomer molecules with multifunctional groups that can form three-dimensional cross-links. [9]. They have been widely used in a variety of industries, including aerospace, maritime, automotive, medical, sports, and machine tools, as well as a variety of other structures. The frictional properties of composites are significantly affected by a combination of filler content, sliding velocity, and load. The rate of deterioration is proportional to both sliding velocity and load. Functionally graded materials are characterised by a continual fluctuation of material properties coming from non-homogeneous microstructures in a chosen direction, resulting in controllable material attributes. Nano-Al<sub>2</sub>O<sub>3</sub> particles boost the bearing strength of a material. Nano-Al<sub>2</sub>O<sub>3</sub> is one of the most established nanofillers among several inorganic or metal-based nanofillers due to its superior mechanical and thermal properties and lower manufacturing cost compared to carbon-based nanofillers. [14]. It is common knowledge that metal matrix composites with the appropriate additives will be used to replace conventional materials since they aim to

increase mechanical qualities and decrease thermal expansion coefficient. Due to its unique mechanical, thermal, and electrical properties, various researchers have attempted to develop complex CNT composite materials with at least one of these characteristics. [16] If the generated heat is not dispersed, the completed product's structural integrity may be jeopardised. Therefore, it is necessary to improve the thermal properties of polymer composite insulators. [17,18] The effect of an aqueous environment and fluctuating temperature on the properties of coupon specimens of glass-fiber epoxy resin prepreg. [19] Due to their rapid setting and high strength, polymer composites are used in a variety of construction, rehabilitation, and repair applications, including bridges, pipelines, and other structures. To overcome the limitations of a single filler in a polymer matrix, research has been conducted on the use of a combination of inorganic fillers in polymer composites. [21,22] Inorganic nanofillers are becoming more acceptable as potential fillers due to their inexpensive cost and facile production technique. [23] Researchers have discovered that nanoscale Al<sub>2</sub>O<sub>3</sub> improves the tensile and shear strengths, fracture toughness, and stiffness of composites. [24] Due to their potential applications in electromagnetics, electronics, catalysis, nonlinear optics, heat conducting materials, etc., nanocomposites are incredibly exciting. [25] The alumina-modified epoxy composite improves the hardness and impact energy in comparison to earlier modifiers. [26] Fiber-reinforced epoxy compounds are essential in a variety of industries due to the increased use of a number of applications, such as gears, rollers, seals, bearings, clutches, wheels, and camshafts, as a result of a number of characteristics, such as enhanced mechanical capabilities. [27].Enhanced mechanical qualities were achieved by altering the reinforcing in this series of composite materials. To reinforce the chopped E-glass fibre infused with Al<sub>2</sub>O<sub>3</sub> nanoparticles, an epoxy matrix has been applied. Mechanical properties have been evaluated by means of tensile, flexural, and hardness testing.

## II. MATERIALS AND METHOD

### 2.1 Raw materials

Epoxy resin (LY 556) has been used as the matrix and hardener (HY951) as the curing agent. Chopped E-glass fiber and Al<sub>2</sub>O<sub>3</sub> nanoparticles have been used as reinforcements.

### 2.2 Fabrication of composite

In the past, GFRP-based composites have been made using the hand layup method. Scissors were used to initially cut five-ply E-glass fibre mats. For 10 minutes, a glass rod is used to manually swirl a mixture of Al<sub>2</sub>O<sub>3</sub> nanoparticles and plain epoxy. The epoxy adhesive mixture is made by adding the hardener at a ratio of 10:1 after the epoxy has been thoroughly mixed.

After positioning the first ply inside the mould box, a coat of the epoxy adhesive mixture is painted on using a brush until the ply is completely saturated. After adding more glue, the second layer is laid on top of the first and left to soak up the liquid. This procedure is done until all five plies have been layered upon one another. To remove any extra glue mixture and keep the composite surface constant in thickness, a mild steel roller is rolled over each layer. In this experiment, the Al<sub>2</sub>O<sub>3</sub> powder was combined in a variety of concentrations (0%, 0.5%, 1%, 1.5%, 2%, and 2.5%). This means that the six possible permutations of the manufactured composites are. Composite laminates were cured for a whole day at room temperature. The laminates were created, and then samples were cut from them (according to ASTM standards)..

### 2.3 Mechanical testing& Thermal analysis

The adhesion between the matrix and the reinforcement is crucial to the strength of a glass fiber-reinforced polymer composite. The composites are characterised through mechanical testing. The hardness, tensile strength, and flexural strength of GFRP composites have all been tested mechanically and thermally.

#### 2.3.1 Tensile testing

According to ASTM D3039, tensile tests on nanocomposites were conducted. On a computerised universal testing apparatus, the tensile tests were performed. 1 mm/min crosshead speeds were chosen. At room temperature, tensile tests were performed. Six samples were examined.

#### 2.3.2 Flexural testing

The three-point flexural test, which was performed using the same universal testing machine in accordance with ASTM standard D-790, was used to study the flexural properties.

**2.3.3 Hardness tests**

Hardness was measured by the Rockwell hardness testing machine. Hardness was measured for each composite sample.

**2.3.4 Differential Scanning Calorimeter (DSC)**

On a DSC calorimeter (Mettler Toledo), differential scanning calorimetric (DSC) measurements of the samples were made in the temperature range from 0 to 350° C when the atmosphere is present. At the halfway point of the procedure, the melting and crystallisation temperatures were determined

**2.3.5 Derivative Thermal Analysis (DTA)**

DTA testing were performed using a Shimadzu TGA-50 /DTA /thermogravimetric analyzer. Under atmospheric circumstances, the samples were heated at a steady rate of 10°C/min from ambient temperature to 1000°C.

**2.3.6 Thermo gravimetric Analysis (TGA)**

TGA testing were performed using a Shimadzu TGA-50 thermogravimetric analyzer. Under atmospheric circumstances, the samples were heated at a steady rate of 10°C/min from ambient temperature to 1000°C.

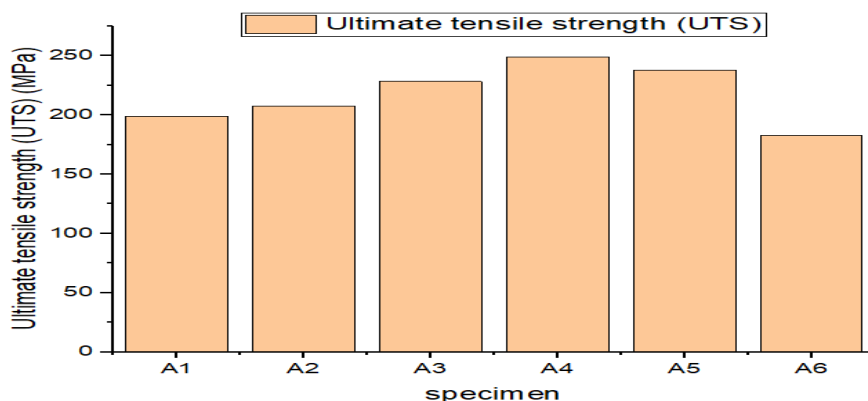
**III. RESULT & DISCUSSIONS**

**3.1 Tensile test results**

The impact of nano-AL<sub>2</sub>O<sub>3</sub> particles on GFRP composites is shown in Figure 1. Due to the strong filler/matrix contact and good particle dispersion, which result in an efficient stress transfer, the addition of nanoparticles considerably increases the tensile strength of composite materials. According to the results, the composite specimen filled with 1.5% nano-AL<sub>2</sub>O<sub>3</sub> particles had the highest ultimate strength (248.99 MPa), and its tensile strength variation was greater than that of other compositions. Beginning with a composite specimen filled with 2% nano-AL<sub>2</sub>O<sub>3</sub> particles, the ultimate tensile strength begins to decline.

**Table. 1** Ultimate tensile strength

| Specimen | Ultimate tensile strength(MPa) |
|----------|--------------------------------|
| A1       | 199                            |
| A2       | 207.55                         |
| A3       | 228.37                         |
| A4       | 248.99                         |
| A5       | 237.91                         |
| A6       | 182.96                         |



**Fig.1:** Variation of Tensile strength w.r.t Nano-Al<sub>2</sub>O<sub>3</sub> Content in composites

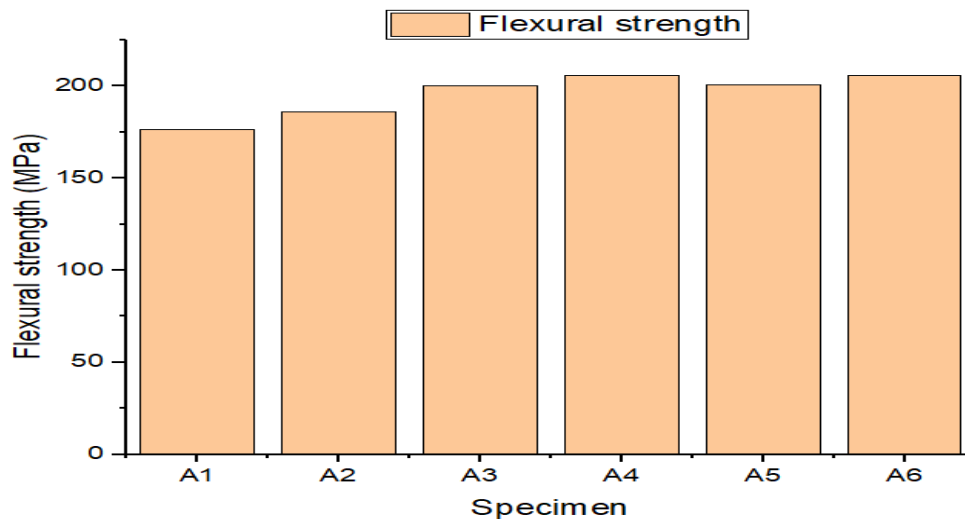
**3.2 Flexural test results**

The flexural strength of composite samples with various amounts of nano-AL<sub>2</sub>O<sub>3</sub> particles is shown in Figure 2. In comparison to other composite specimens, the specimen with 1.5% nano-AL<sub>2</sub>O<sub>3</sub> particles had the highest flexural strength (205.88 MPa), which is again attributable to the uniform distribution of nano-fillers and the

strong bond between epoxy and filler materials, which increases energy absorption and inhibits crack initiation and growth. Additionally, it can be observed that the flexural strength of composite specimens steadily increases as the fraction of nano-fillers rises.

**Table. 2:** Maximum Flexural strength.

| Specimen | Maximum flexural strength(MPa) |
|----------|--------------------------------|
| A1       | 176.5                          |
| A2       | 186.2                          |
| A3       | 200.2                          |
| A4       | 205.88                         |
| A5       | 200.81                         |
| A6       | 205.63                         |



**Fig. 2:** Variation of flexural strength w.r.t Nano-Al<sub>2</sub>O<sub>3</sub> Content in composites

### 3.3 Hardness test results

The micro-hardness of the composite is measured using a digital Leco micro-hardness tester. To leave an impression on the surface, a diamond indenter is pressed into the composite specimen for 10 seconds while being subjected to a stress of 3 N. In Table 3, the typical Vickers hardness is listed. The hardness of composites is seen to be gradually rising, with 25.3 Hv being the maximum value. However, the hardness values show that there isn't much of a hardness difference between compositions.

**Table. 3:** Micro hardness values

| Specimen | Micro Hardness Hv |
|----------|-------------------|
| A1       | 17.2              |
| A2       | 18.5              |
| A3       | 20.1              |
| A4       | 21.6              |
| A5       | 22.4              |
| A6       | 25.3              |



Fig. 3: Variation of Hardness value w.r.t to nano-Al<sub>2</sub>O<sub>3</sub> Content in composites.

### 3.4 DSC test results

The effects of micro particles on the glass transition temperature of the composite are analyzed for A1, A2, A3, A4, A5, and A6 types of compositions. The glass transition temperature with different modifiers is reported in Table 4. There is no significant change in the tg values.

Table. 4: Glass transition temperature

| Specimen | Glass transition temperature(°C) |
|----------|----------------------------------|
| A1       | 60.8                             |
| A2       | 61.6                             |
| A3       | 62.4                             |
| A4       | 63.6                             |
| A5       | 66.0                             |
| A6       | 67.5                             |

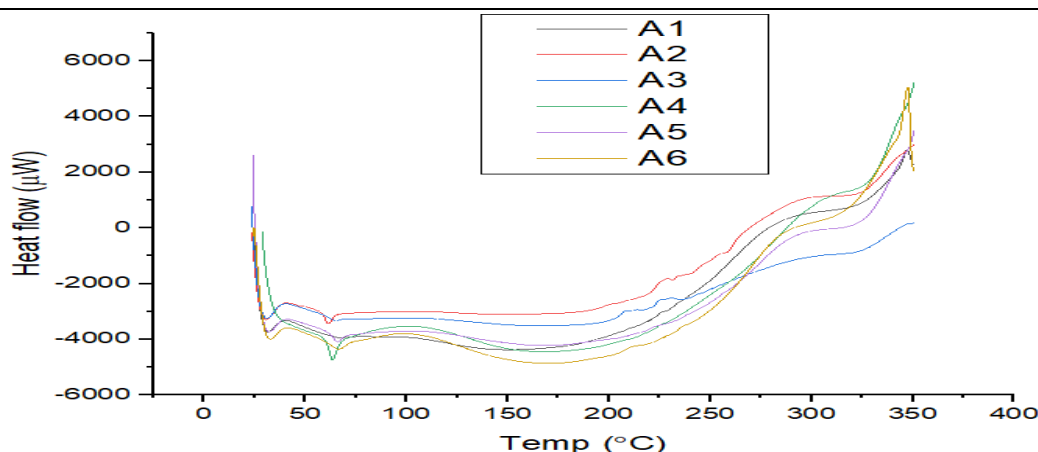


Fig. 4: Temperature vs. Heat flow Graph

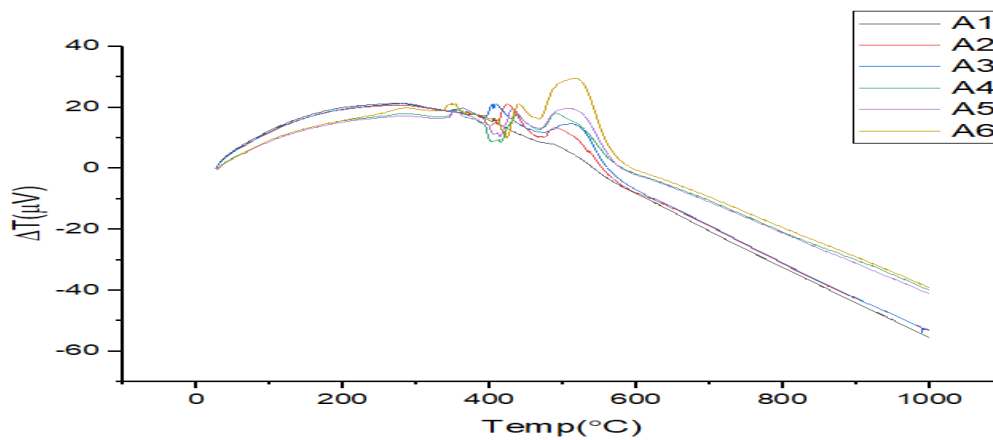
### 3.5 Derivative Thermal Analysis (DTA)

DTA test results

Fig 5.show DTA graphs of nano AL<sub>2</sub>O<sub>3</sub> filler material composite in an oxygen atmosphere. In this Fig 5, the DTA peak signifies the oxidation temperature of AL<sub>2</sub>O<sub>3</sub> nanocomposites. At 474°C large spike appeared. This is due to the sudden loss of mass of nano AL<sub>2</sub>O<sub>3</sub>. In addition, there is a great weight loss between 400° and 500°C which is related to pyrolysis of the polymer materials, and between 600°C and 700°C another weight loss occurs smaller than the other one.

**Table. 5** Peak value temperature

| Specimen | Peak value Temperature (°C) |
|----------|-----------------------------|
| A1       | 269                         |
| A2       | 423                         |
| A3       | 457                         |
| A4       | 464                         |
| A5       | 471                         |
| A6       | 474                         |



**Fig. 5:** Temperature vs. Heat flow Graph

### 3.6 TGA test results

Table 6 shows beginning and final temperatures of nano AL2O3-filled composites. Nanofillers in polymer matrix improve composites' thermal stability. Thermolysis has two phases. First, interlayer moisture evaporates, causing composites to degrade. The composite degraded 10% at this stage. Nano AL2O3 packed composite degrades between 233°C and 257°C. In the second step, thermal deterioration is related to filler loading, bonding strength, and filler-matrix compatibility. A3 has a higher weight loss percentage than A4. Therefore, 1.5% nano AL2O3 composite is thermally stable. Fig.6 shows that ceramic nano AL2O3 filler is more stable and tougher than epoxy matrix. Nano AL2O3 is heat-resistant. Thus, the nano AL2O3 filler can improve the composite's thermal stability. Increasing nano AL2O3 decreased degradation temperatures. Nano AL2O3 improves polymer composites' heat stability.

**Table 6:** Initial and Final degradation temperatures

| Specimen | Initial Degradation temperature(°C) | Final Degradation temperature(°C) |
|----------|-------------------------------------|-----------------------------------|
| A1       | 233                                 | 573                               |
| A2       | 260                                 | 568                               |
| A3       | 265                                 | 582                               |
| A4       | 271                                 | 576                               |
| A5       | 280                                 | 562                               |
| A6       | 257                                 | 575                               |

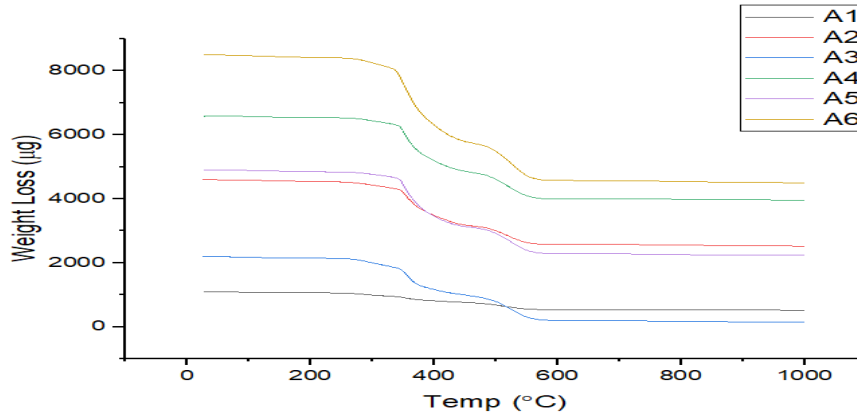


Fig. 6: Weight loss vs temperatures

#### IV. FRACTURE SURFACE ANALYSIS THROUGH SCANNING ELECTRON MICROSCOPY (SEM)

Different micro and nanoscale failures in various sets of composites often have underlying mechanisms that may be determined by the SEM study of the fracture surface. As a result, all of the composite specimens that failed the tensile tests underwent fractographic examinations. The strength and toughness of a specimen are unquestionably increased when nano-Al<sub>2</sub>O<sub>3</sub> is added to epoxy, and it is anticipated that this will change how typical FRP composites fail.

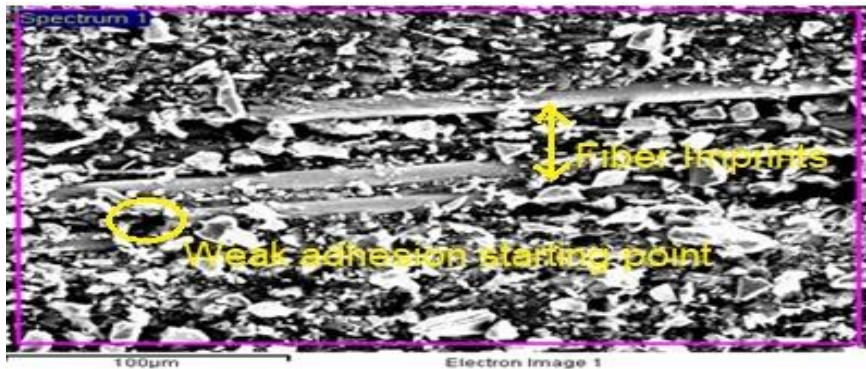


Fig. 7: Fracture study of a 0.5% nano Al<sub>2</sub>O<sub>3</sub> composite.

The delamination surfaces of the 0.5% nano-Al<sub>2</sub>O<sub>3</sub> composites evaluated at room temperature are shown in Fig. 7. The delamination of the fibres from the polymer matrix is seen in the composite's morphology as very smooth fibre impressions (Fig.7). However, composites with nano-Al<sub>2</sub>O<sub>3</sub> incorporated in them change the shape of fibre impressions. As shown in Fig.7, all nano-Al<sub>2</sub>O<sub>3</sub> boosted composites have matrix deformation and fibre impressions on the delaminated surface.

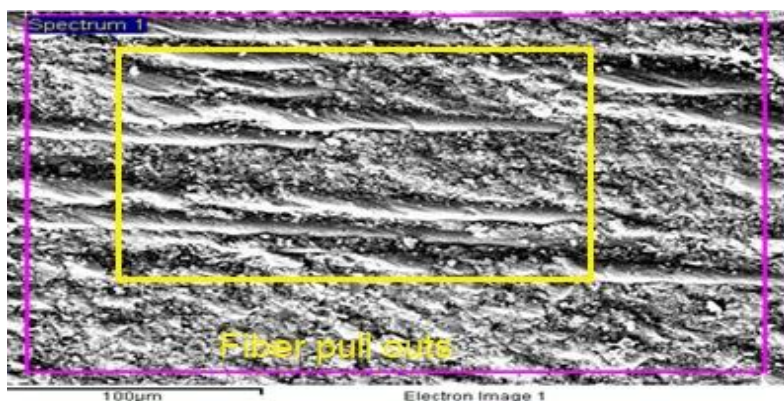
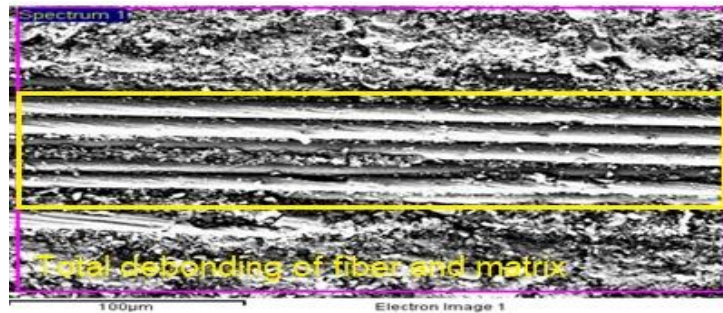


Fig. 8: Analysis of 1.5% nano Al<sub>2</sub>O<sub>3</sub> composite fracture.

The 1.5 weight percent nano-Al<sub>2</sub>O<sub>3</sub> composites evaluated at room temperature exhibit various deformation morphologies, as shown in Fig.8. In 1.5 wt% nano-Al<sub>2</sub>O<sub>3</sub> composites, Fig.8 shows fibre pull out and hardening of the matrix phase, which undoubtedly improves strength and modulus in comparison to control Al<sub>2</sub>O<sub>3</sub> composites, where significant matrix deformation occurred as seen in Fig.7.



**Fig. 9:** Fracture study of a 2.5% Nano Al<sub>2</sub>O<sub>3</sub> composite.

The rough and bare fibre surface in Fig.9, which lacks any indication of a polymeric phase, demonstrates fiber/matrix debonding, which may be related to insufficient interfacial bonding. In contrast, a coating of the epoxy matrix can be seen covering the fibres' surface in the case of the 0.5 weight percent nano-Al<sub>2</sub>O<sub>3</sub> composite, which also shows a strong fiber/matrix interfacial connection. Also visible on the fibre surface in Fig.9 are morphologies like mirror, mist, and hackle.

## V. CONCLUSION

Experiments were conducted on varying six different vol. percentages of nano Al<sub>2</sub>O<sub>3</sub> used as reinforcement and epoxy was used as a matrix to build composite laminates to evaluate the mechanical properties and exhibit the change in mechanical properties and dispersion stability of nano Al<sub>2</sub>O<sub>3</sub>. Increasing the vol.% of nano Al<sub>2</sub>O<sub>3</sub> in glass fibre reinforced polymer laminates boosted their mechanical characteristics. Fiber orientation, nano Al<sub>2</sub>O<sub>3</sub> content, resin content, and fabrication technique affect laminate efficiency. Stability, agglomeration, voids, and dispersion are nano Al<sub>2</sub>O<sub>3</sub> laminate issues. Different characterisation instruments were used to analyse nano Al<sub>2</sub>O<sub>3</sub> epoxy samples' mechanical and microstructural behaviour. Ultimate tensile strength, flexural strength, and microhardness were determined using a universal testing machine, a three-point bending test, and a tensile test. DSC, DTA, and TGA tests do thermal analysis. All failed composite specimens were SEM-analyzed. SEM analysis checks matrix epoxy and filler dispersion.

- Tensile test findings under ASTM D638 standard for laminates with varied vol.% of nano Al<sub>2</sub>O<sub>3</sub> (0vol.%, 0.5vol.%, 1vol.%, 1.5vol.%, 2vol.% and 2.5vol.%) are 199MPa, 207.55MPa, 228.37MPa, 248.99MPa, 237.91MPa and 182.96MPa, respectively.
- The 1.5 vol.% nano Al<sub>2</sub>O<sub>3</sub> epoxy sample had the highest tensile strength of all compositions, 248.99MPa.
- 2% of nano Al<sub>2</sub>O<sub>3</sub> sample tensile strength dropped from 237.91MPa to 182.96MPa. Some places may have nano Al<sub>2</sub>O<sub>3</sub> aggregation.
- Flexural test results under ASTM D790 standard for laminates with varied vol.% of nano Al<sub>2</sub>O<sub>3</sub> (0 vol.%, 0.5 vol.%, 1 vol.%, 1.5vol.% and 2 vol.%) are 176.5MPa, 186.2MPa, 200.2MPa, 205.88MPa, 200.81MPa and 205.63MPa correspondingly.
- The 1.5 vol.% nano Al<sub>2</sub>O<sub>3</sub> epoxy sample had the highest flexural strength of all compositions, 205.88 MPa.
- A 2 vol.% nano Al<sub>2</sub>O<sub>3</sub> sample had a flexural modulus of 200.81MPa, while a 2.5 vol.% sample had 205.63MPa. Some places may have nano Al<sub>2</sub>O<sub>3</sub> aggregation.
- The Vickers Hardness test under ASTM D785 standard for laminates with varied vol.% of nano Al<sub>2</sub>O<sub>3</sub> (0 vol.%, 0.5 vol.%, 1 vol.%, 1.5 vol.%, 2 vol.% and 2.5 vol.%) is 17.2Hv, 18.5Hv, 20.1Hv, 21.6Hv, 22.4Hv and 25.3Hv.

The 2.5 wt% nano Al<sub>2</sub>O<sub>3</sub> epoxy sample had the greatest Vickers hardness value of 25.3Hv.

- The brittleness of nano Al<sub>2</sub>O<sub>3</sub> enhances hardness. Beyond 1.5vol.% nano Al<sub>2</sub>O<sub>3</sub>, flexural and tensile strength decline while hardness increases.

DSC showed that tg values hadn't changed.



- According to the TGA, A3 lost 90.5% more weight than A4, which lost 38.36%.
- DTA demonstrates that when nano  $Al_2O_3$  % grows, so does peak value. In accordance with the ASTM D785 standard, the Vickers hardness test results for laminates containing various volume percents of nano- $Al_2O_3$  are 17.2Hv, 18.5Hv, 20.1Hv, 21.6Hv, 22.4Hv, and 25.3Hv, respectively.

According to the findings of the Vickers hardness tests performed on the nano  $Al_2O_3$ -epoxy samples, the 2.5 wt% sugarcane bagasse sample had the greatest hardness value of all the compositions, measuring 25.3Hv.

Because of the increase in brittleness, the hardness value rises as the volume percent of nano increases. As a result, hardness improves but flexural and tensile strength decreases above 1.5 vol% of nano.

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