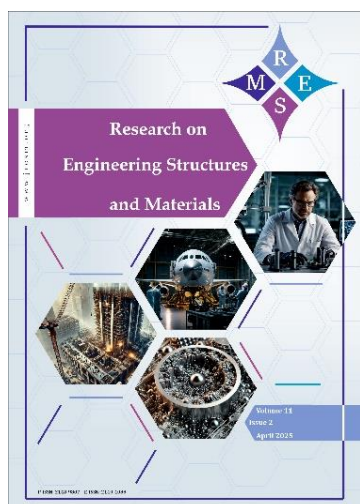




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## Effect of ADG-NH<sub>2</sub> functionalized graphene on fracture and impact resistance of carbon fiber reinforced composites

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Article Info	Abstract
<p><b>Article History:</b></p> <p>Received 24 Dec 2024</p> <p>Accepted 13 Mar 2025</p> <p><b>Keywords:</b></p> <p>Composite;</p> <p>CFRP;</p> <p>Amine functionalized graphene;</p> <p>Tensile test;</p> <p>Fracture toughness;</p> <p>SEM</p>	<p>In this study tensile, single edge notch beam [SENB] and impact tests were done as per ASTM standards to evaluate the influence of the ADG-NH<sub>2</sub> functionalized Nano additives on the mode I fracture and impact resistance characteristics. This study highlights the enhancement in the Tensile strength, Mode I fracture resistance and impact strength properties of Carbon Fiber Reinforced Polymer [CFRP] composites loaded with Lower concentration of Graphene reinforcement. Five symmetrical, modified composite laminates [ADG-NH<sub>2</sub>/Epoxy/CFRP] were fabricated using a hand layup process supported by vacuum bagging. These laminates incorporated various weight content [0.25, 0.5, 0.75, and 1%] of ADG-NH<sub>2</sub>/Epoxy along with a neat epoxy for comparison. Notable improvements were observed in the composite properties with 0.5 wt% ADG-NH<sub>2</sub> graphene Nano-additive reinforcement in comparison with base epoxy CFRP composite laminates, including increase in elastic modulus by approximately 18%, rise in ultimate tensile strength by around 21%, enhanced elongation at break by roughly 19% and Improved toughness, showing a 28% gain. An enhancement of ~29 % and ~ 60% were observed for the fracture resistance [<math>K_{IC}</math> &amp; <math>G_{IC}</math> respectively] of base epoxy resin [value of ~ 0.67 MPa m<sup>1/2</sup> and ~157 J/m<sup>2</sup> respectively] with 0.5 wt% ADG-NH<sub>2</sub> loading obtaining a value of ~ 0.86 MPa m<sup>1/2</sup> and ~251 J/m<sup>2</sup> respectively. Similarly, enhancement of ~ 41% was observed for the impact strength of base epoxy resin [value of ~77 J/m<sup>2</sup>] with 0.5 wt% ADG-NH<sub>2</sub> loading obtaining a value of ~108 J/m<sup>2</sup>. Morphological properties of fracture surfaces were characterized by SEM micrographs analysis.</p>

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### 1. Introduction

Many engineering and industrial applications have broadened the requirement of CFRP composites due to its superior thermal properties i.e. high insulation, stability, high heat resistance, low shrinkage along with enhanced mechanical properties like good dimensional stability, high tensile strength, and modulus etc.[1–5]. Nano additives like 2-D graphene sheet made of honeycomb structure arrangement SP<sup>2</sup> carbon atoms were used in CFRP structures to enhance mechanical, electrical, and thermal properties[6,7]. Earlier research indicated that graphene reinforced nano composites possess superior strength, stiffness and cost effective in comparison with clay, silica and carbon nano composites [8,9]. Additives like graphene nano particles enhances tensile properties and fracture stiffness of polymer composite but at the same time drastically changes the material properties i.e. weight to surface area ratio with the use of very small amount [10,11]. When a high loading content of graphene nano particles [> 1wt%] was added to the composite, fracture toughness was significantly reduced [12]. Graphene and graphene derivatives produced via chemical vapor deposition or mechanical exfoliation with several surface functionalities generally

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enhances the fiber matrix bonding and shows excellent properties compared to single layer graphene composites [13,14]. Surface functionalization with nano additives reduces agglomeration and further ameliorates the particle/ matrix interface [15,16]. Covalent functionalization shows more variation in material properties and excellent bonding between the functional groups and particle surfaces. Covalent bonding between the matrix and additive enables an enhanced electric charge, phonons transfer as well as mechanical loads transfer across the particle/polymer interface [17–20]. To form a strong amide bond with epoxies, amine functionalities take part in the polymerization process which is cured with amine-based hardeners and used in the majority of structural FRPs [21,22]. This functionalization found further applications such as lightning protection [23], aircraft deicing [24], sensory materials [25–29], electromagnetic interference shielding [30], thermal interface materials [31,32] or ballistic applications [33].

Various researchers also studied the effect of fracture resistance and impact strength through fictionalization of CNTs and other nano materials like silica etc. with epoxy based nano composites. In this study, fracture stiffness of the nanocomposite with graphene exhibited an approximate 53% improvement in comparison with base epoxy. The influence of SWCNTs and MWCNTs on mechanical properties, along with the advantages of surface treatment of CNTs, was examined [34,35]. It was observed that the fracture toughness of base epoxy increased by approximately 45% when reinforced with non-functionalized SWCNTs. However, increasing the SWCNT weight percentage beyond 0.3% lead to in a reduction of fracture toughness due to re-agglomeration. Whereas the strengthening mechanisms of nano-silica-reinforced epoxy polymers at a 13 wt% concentration, fabricated using a sol-gel technique [36], demonstrated a substantial increase in fracture stiffness—from 100 J/m<sup>2</sup> to 460 J/m<sup>2</sup>.

The CFRP composites reinforced with ADG-NH<sub>2</sub> exhibit excellent potential in diverse applications due to its superior mechanical and thermal properties. However, it is crucial to thoroughly assess their performance under prolonged environmental exposure, such as humidity, temperature variations, and UV radiation. These factors significantly influence the composites' practical lifespan and overall reliability, which are essential for real-world applications. The thermal characterization studies such as thermal aging, heat resistance ensuring their thermal stability over time and interaction with various chemicals, including solvents, acids and bases ensuring their durability during chemical aggressive environments to be accessed thoroughly. Hence for the Long-term stability and durability of CFRP composites with ADG-NH<sub>2</sub> reinforcement the following future works such as accelerated aging test, Long- term mechanical testing including fatigue testing and dynamic mechanical analysis to be carried out which will provide better understanding of their practical lifespan and ensure reliable and efficient performance in real-world conditions.

From the previously literature review, it has been deduced that the carbon fiber shows poor wettability to the most of the polymeric matrix material. And also, it is well established that the inclusion of non-functionalized nano particles like graphene nano platelets , graphene oxide and functionalized nano particles like amino-functionalized Graphene oxide, hydroxyl graphene and carboxyl reinforced graphene] are extensively being used to augment the bulk mechanical properties. Very few literatures have used amine functionalized graphene as nano filler. Therefore, amine functionalized graphene has been used which helps in better distribution and improves the bonding between carbon fiber and aerospace grade epoxy matrix used in this study. This literature offers the novel manufacturing processing route to introduce ADG-NH<sub>2</sub> into the matrix, thereby focusing on better dispersion of the nano filler with the matrix. The improvement in interfacial adhesion in laminate was verified through tensile test results. This study also includes the out of plane loading performance of CFRP laminate under SENB and impact test and also corroborate it with the help of improvement in fracture resistance and impact strength properties.

This study presents the development of amine-functionalized graphene-epoxy CFRP composites, exploring various ADG-NH<sub>2</sub> loadings [ $<1$  wt%]. It investigates the impact of amine functionalization on key mechanical properties, including fracture stiffness, impact resistance, tensile strength, and morphological characteristics. The paper also details the processing methodology for incorporating ADG-NH<sub>2</sub> into the epoxy matrix, demonstrating that its homogeneous dispersion significantly enhances the composites' overall performance.

## **2. Experimental Procedures**

### **2.1 Materials**

The amine functionalized graphene [product No.: ADG-NH<sub>2</sub>] was received from M/s AdNano Technologies Private Limited, Shivamogga, Karnataka, India. Homogeneous dispersion [purity ~99%] of the amine functionalized graphene [containing 5-10 layers of graphene] with addition of [~ 2 to 5%] NH<sub>2</sub> on graphene in order to attain the desired exfoliation and dispersion increases the thermal and mechanical properties. Covalently functionalized graphene particles have thickness range of 5-10 nm and lateral dimension ranging between 5-10  $\mu$ m with bulk density and surface area 0.1 g/cm<sup>3</sup> and 60-200 m<sup>2</sup>/g respectively. [Refer Fig 1]. Bidirectional carbon fiber woven fabrics [T700], Epoxy [LY 5052] and hardener [CH 5052] were used for this study. The high strength non crimp carbon fiber fabrics [Product No.: T700] were purchased from M/s Carbonext India Private Limited. Nashik, Maharashtra, India having thickness 0.25 mm or 250 gsm.. Epoxy resin [ARALDITE LY5052] and hardener [ARADUR 5052 CH] received from M/s Singhal Chemical corporation, Meerut, UP, India were used for this study. The resin was mixed with hardener at a ratio of 100:38. Initially a baseline T700 carbon fiber fabric CFRP composite with neat epoxy were manufactured. The mould release agent was received from Mohini Organics Pvt. Ltd., Malad [West], Mumbai, Maharashtra, India and an adhesive tape, release film & peel ply were purchased from Aristo Flexi pack, Daman and Diu, India. For this composite Laminate system, the nominal resin and fiber volume was of the ratio 34: 66.

### **2.2 Fabrication of Amine Functionalized Graphene [ADG-NH<sub>2</sub>]/ Epoxy**

As shown in Schematic representation [Refer Fig. 1], during this study all the composite laminates were prepared under similar environmental conditions. The desired Amine functionalized graphene [ADG-NH<sub>2</sub>] solution loading content was added to solvent medium [methanol] and dispersed using an ultrasonic dispersion machine [Hielscher Ultrasonic homogenizer [Product No. UP400 ST] with 22 mm probe], for 1 hour to ensure the homogeneous dispersion of ADG-NH<sub>2</sub> by breaking the Vander walls attractive force of attraction between the nano particles. This process completely removes of ADG-NH<sub>2</sub> aggregates, enabling effective dispersion. For dispersion of ADG-NH<sub>2</sub>, large volume of methanol solvent was used. The base epoxy was then incorporated to the ADG-NH<sub>2</sub>/methanol dispersion and the solution was continuously stirred. Methanol was evaporated from the ADG-NH<sub>2</sub>-epoxy solution by using a rotorvap machine which was operated at 45° C [10 bar]. The resulting mixture was then allowed to settle down inside the oven at 45° C under the vacuum at 10 bar and methanol was completely evaporated. A mixing machine with high speed of rotation [ROSS Laboratory High shear mixer [Model 100LH], NY USA] operating at about 20 min to mix the ADG-NH<sub>2</sub>/epoxy. The mixtures were then allowed to settle down on the beaker stand and agglomerates were completely removed. The neat epoxy was treated similarly as the processing stage of ADG-NH<sub>2</sub> different Wt% filler loadings. Then the Hardener Aradur 5052 CH was mixed to the ADG-NH<sub>2</sub>/ epoxy solution at a ratio of 100:38, which was mixed again using the mixing machine with high speed of rotation at 3000 RPM for 20 minutes. Then degassing of the suspension was carried out in a vacuum chamber [pressure ~10 bar] at 45° C for approximately 20 min while manual mixing through mechanical stirrer was carried out during the entire process. Mixture was then transferred into an open beaker at room temperature [RT] which was used for preparation of CFRP laminate.

### **2.3 Fabrication of Composite Laminates**

Bidirectional carbon fiber was cut into 4 pieces of 300 mm x 300 mm with 0° / 90° orientation and 8 pieces of 45° /-45° orientation to prepare CFRP Laminate. For fabrication of the laminate a 15 mm thick plane aluminum plate was considered and its top face is cleaned thoroughly with MEK on which the Laminate wet Lay-up process to be carried out. A release film [300 x 300 mm<sup>2</sup>] with 15  $\mu$ m thickness was laid and on it a peel ply [30 micron] was applied. Prepared adhesive resins solution [[ADG-NH<sub>2</sub>+ epoxy [LY 5052] + Hardener [5052 CH]] was applied on it using brush mould. Then the first carbon fiber bi-directional woven sheet [in 0° / 90° orientation] kept on the adhesive resin's solution. Then the second carbon fiber layer [at 45° /-45° orientation] was kept over the first carbon fiber layer after applying the adhesive resin solution on the first fabric uniformly with the



help of a brush. To have uniform thickness of laminates and avoid the epoxy starvation between the two carbon layers, the extra number of resins were squeezed with the help of a roller on the carbon fiber woven sheets.

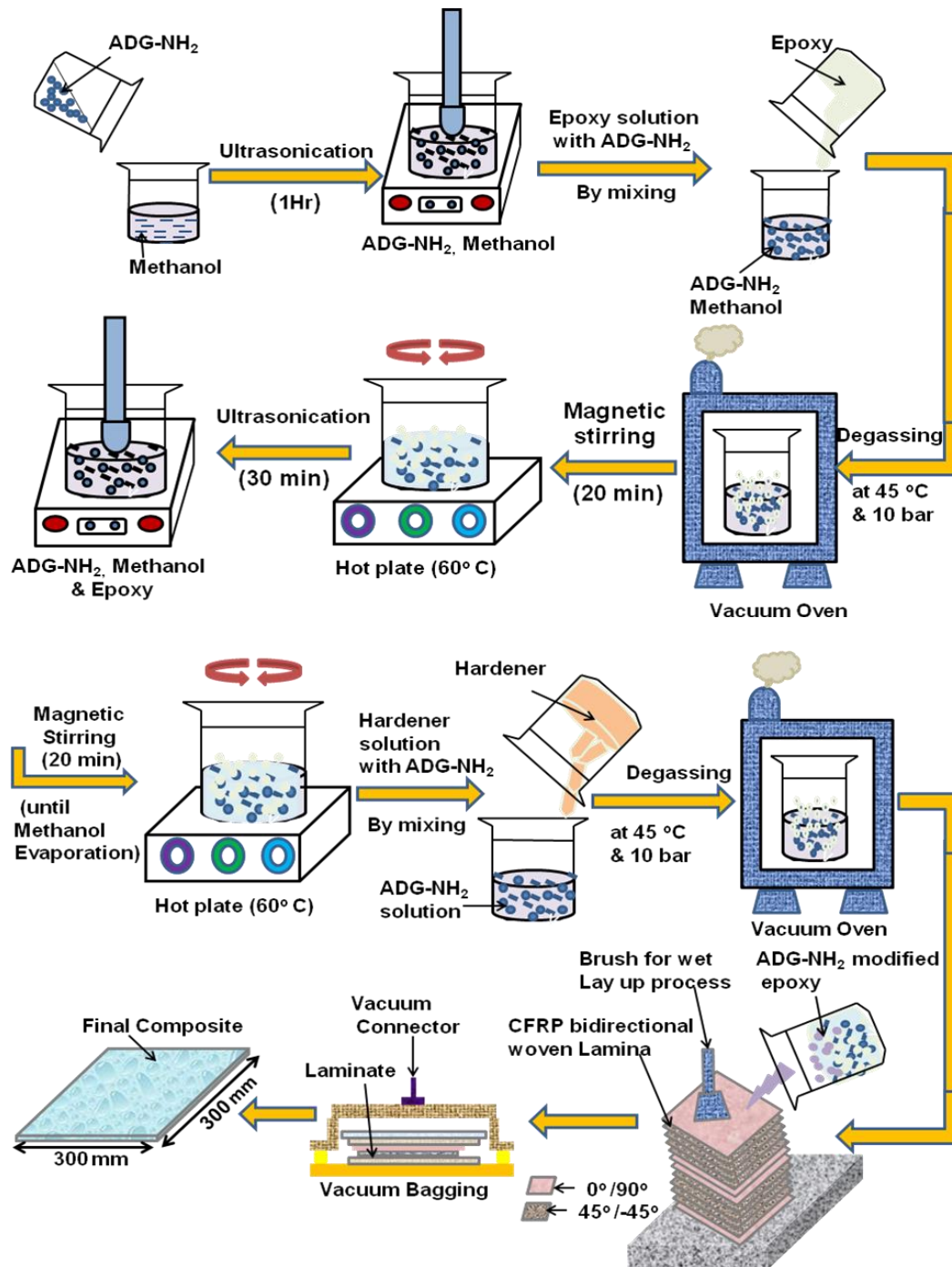


Fig. 1. Schematic diagram of processing of ADG-NH<sub>2</sub> and fabrication of laminated components

The third layer was kept [at 45°/-45° orientation] on the second layer and same procedure was followed. The 4th, 5th and 6th layers were kept at 45°/-45°, -45°/45° and 90°/0° deg respectively in the same manner and the same procedure was adopted towards the preparation of twelve layers of symmetric cross plied quasi-isotropic CFRP Laminate as shown in Figure 1. After laying twelfth layer, peel ply has to be kept and on it aluminum plate has to be kept on the top. The Carbon fiber/epoxy laminate staking sequence along with fiber orientation and thickness is shown in Figure 1. During the manufacturing of the CFRPs, Vacuum Bagging Technique was used for curing of the whole stack of laminate. In this Vacuum Bagging arrangement, first a mild steel plate was taken and cleaned thoroughly with MEK which was used to form the mould base. After treating this

plate with a mould release agent, for making the mould frame Tacky tape was used, with inlet and outlet tubes. The thickness of the laminates fabricated in this entire process was between 3 to 3.5 mm which meets standard testing requirements. During this process in order to address air entrapment and void formation the laminate was cured at full Vacuum [10 bar]. The laminate was kept at this condition for 24 hours curing at Room Temperature. Post curing, Laminate was again cured for 1 hr at 60° C and then again at higher temperature to 120° C for 3 hours. Similar procedure was adopted towards fabrication of other CFRP laminates with different ADG-NH<sub>2</sub>/epoxy Wt% [0.25, 0.5, 0.75, & 1]. The average thickness of various fabricated ADG-NH<sub>2</sub>/epoxy/CFRP composite Laminates were 3.02, 3.12, 3.23, 3.34, 3.45 mm respectively for pristine, 0.25, 0.5, 0.75 and 1 weight% ADG-NH<sub>2</sub>/epoxy. It was noticed that due to increasing addition of ADG-NH<sub>2</sub> loading content the average thickness of fabricated CFRP composite laminate increases minimally. For mechanical and morphological characterization composites were cut into test specimens by means of a high-speed diamond cutter as per the testing standard requirements. The same fabrication technique and identical conditions were adopted for all neat epoxy resin base laminate to compare the performance of graphene content addition.

## 2.4 Methodology of Characterization Study

### 2.4.1 Morphology Study of ADG-NH<sub>2</sub> and ADG-NH<sub>2</sub> Enhanced CFRP Composites

To evaluate the characteristics of amine functionalized graphene [ADG-NH<sub>2</sub>] nano characterization methodologies such as FT-IR and SEM were carried out. FT-IR spectra of the ADG-NH<sub>2</sub> was carried out using a Hover lab FT-IR spectrophotometer [model No HV-5500] with a 2 cm<sup>-1</sup> resolution over 64 scans. The surface was checked by using a SEM [ZEISS Microscopy, Germany, [Model EVO 15] with 20 kV acceleration voltage of and 2.5 mm working distance. For fractographic characterization, fracture surfaces were gold coated and images were studied using a SEM.

### 2.4.2 Single Edge Notch Bending Test

SENB testing was conducted to investigate the fracture properties of the modified CFRP composite as shown in Fig. 2. An UTM [kalpak instruments and controls, Pune, India, [Model No 201201] having max load carrying capacity of 50 KN and with the help of a three-point bending fixture as per ASTM D 5045 [37]. During sample preparation a V notch initial-crack of 2 mm length was generated. The laminate was subjected to normal [bending] and crack initiates along the V notch through the pre-crack. A ratio of length to depth of 4:1 and depth to thickness ratio of 2:1 was considered. The test was carried out at a speed of 10 mm/min at room temperature [25°C]. Five identical specimens were tested for each ADG-NH<sub>2</sub> /Epoxy/ CFRP loading wt% and results were reported. The average dimensions of the laminate samples were length x width x thickness 60x12x3 mm<sup>3</sup>, respectively with a span length of 48 mm. The diameter of the loading roller and the support roller was 6 mm and 3 mm respectively.

By using equations 1 and 2 the critical Stress Intensity Factor [ $K_{IC}$ ] and Mode I fracture toughness [ $G_{IC}$ ] were evaluated as follows.  $K_{IC}$  and  $G_{IC}$  values were evaluated by conducting single edge notch bending test.

$$K_{IC} = \left\{ \frac{PL}{bt^{3/2}} \right\} Y; 0 < Y < 1$$

$$Y = \frac{3(a/t)^{1/2} \left[ 1.99 - \left( \frac{a}{t} \right) \left( 1 - \frac{a}{t} \right) \left[ 2.15 - 3.93 \left( \frac{a}{t} \right) + 2.7 \left( \frac{a}{t} \right)^2 \right] \right]}{2 \left( 1 + 2 \frac{a}{t} \right) \left( 1 - \frac{a}{t} \right)^{3/2}}$$

$$G_{IC} = \frac{(1 - \nu^2) K_{IC}^2}{E}$$

Where;  $K_{IC}$  : Critical Stress Intensity Factor [MPa m<sup>[1/2]</sup>],  $G_{IC}$  : Critical Stress Energy Release Rate [mode I] [J/m<sup>2</sup>], P : Critical load for crack propagation [N], L : Length of the span [mm], Y : Geometrical factor, B : Width of specimen [mm], t : Thickness of specimen [mm], a : Pre crack length

[mm],  $\nu$  : Poisson's ratio for the epoxy resin [taken to be 0.3], and  $E$ : Tensile modulus obtained from fracture testing.

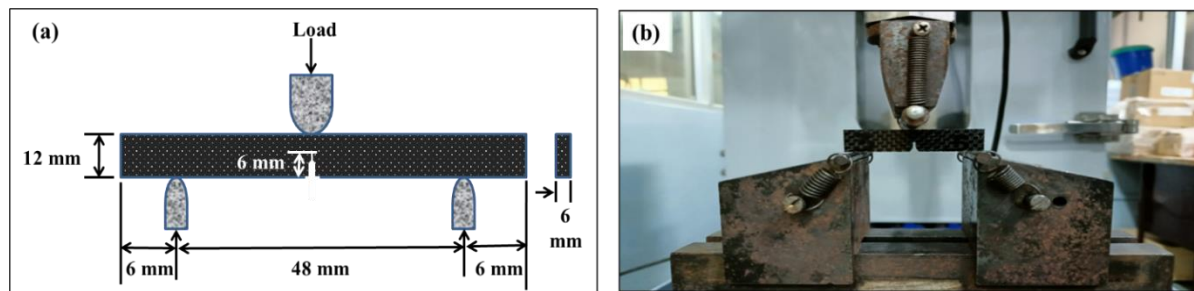


Fig. 2. (a) SENB test ADG-NH<sub>2</sub>/epoxy/CFRP composite dimension as per ASTM D5045 and (b)SEBN Test Setup arrangement

#### 2.4.3 Izod Impact Test

As per the ASTM-D256 [38] standard requirement, Izod Impact test specimens were prepared. The Laminate specimens were cut into beam dimensions of thickness x width x length 3 x 12.7x64 mm<sup>3</sup> respectively. According to the testing protocol, the specimen was kept in vertical position and fractured. The initial point of contact was maintained at a specific distance from both the clamp and the midpoint of the notch, ensuring alignment with the notch.

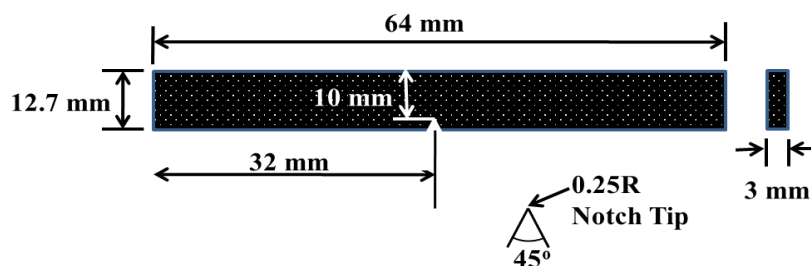


Fig. 3. Specimen dimension of ADG-NH<sub>2</sub>/epoxy/CFRP composite impact test

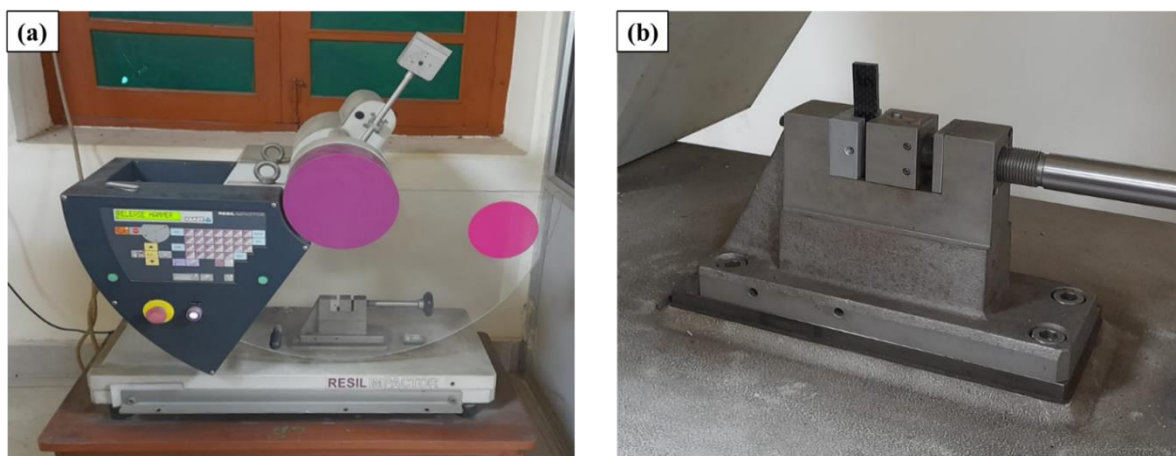


Fig.4. (a) Instron CEAST Resil Impactor (b) Izod Impact test fixture arrangement with ADG-NH<sub>2</sub>/epoxy/CFRP composite specimen

The notch has been done in a milling machine as per the requirement of the standard as per sketch Fig.3. The impact test was conducted using the RESIL IMPACTOR-50J machine from CEAST Instruments, Torino, Italy. The setup included an Izod impact fixture and a hammer with an energy capacity of 15J, expandable up to 25J [as shown in Fig. 4]. The test was done at an impact speed ranging between 2.9 m/s and 3.7 m/s to evaluate the material's impact resistance under controlled

conditions. Tests were conducted for five identical specimens with ADG-NH<sub>2</sub> /Epoxy/ CFRP loading wt% and then average, standard deviations were considered.

#### 2.4.4 Tensile Test

As per ASTM D3039 standards [39] the tensile properties of ADG-NH<sub>2</sub>/Epoxy/CFRP laminates were analysed. Specimens of (length) × (width) × (thickness) i.e 250 X25.4 X 3 mm were prepared as per standard requirements [Refer Fig. 5]. End-tabs were bonded to the rectangular specimens to evaluate the in-plane characteristics of the laminates. Rectangular shaped specimens were chosen over dog-bone-shaped specimens, which tend to split in regions where the width transitions. To mitigate stress concentration introduced by the tension test frame grips, tabs with tapered ends were attached to both sides of the specimens. UTM with 100 kN capacity and constant crosshead speed of 2 mm/min was used to carry out the tensile loading to the specimens. Testing was performed on five identical specimens for each ADG NH<sub>2</sub>/Epoxy/CFRP loading wt%, All tests were carefully monitored and conducted at room temperature.

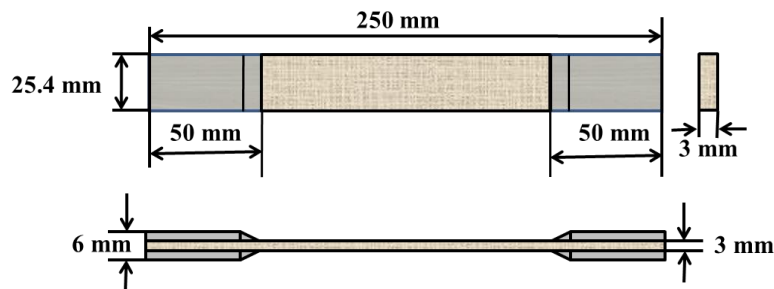


Fig. 5. Schematic diagram of Tensile Test Specimen of ADG-NH<sub>2</sub>/epoxy/CFRP composite as per ASTM D3039

### 3. Results and Discussion

#### 3.1 Morphology study of ADG-NH<sub>2</sub>

As received ADG-NH<sub>2</sub> morphology study was carried out via FT-IR analysis to detect functional groups information as shown in Fig.6 [a].

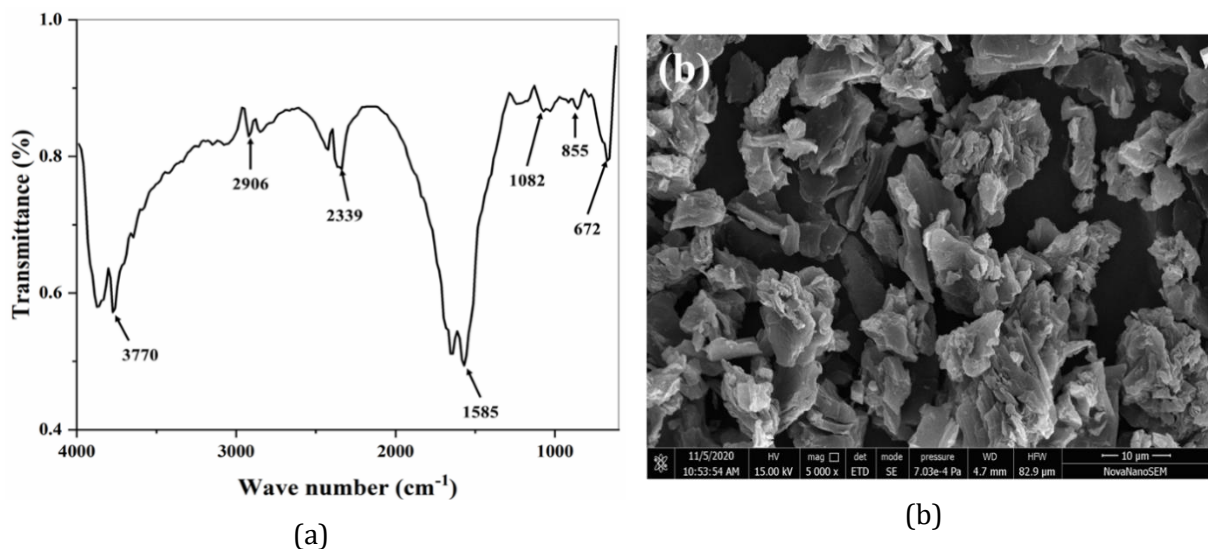


Fig. 6. (a) FT-IR Spectroscopy and (b) SEM images of amine functionalized graphene [ADG-NH<sub>2</sub>]

Micrograph analysis through SEM for the as received ADG-NH<sub>2</sub> with the higher magnification image was carried out [Ref Fig.6 [b]]. The lateral dimensions of functionalized graphene particles were found to be of the order of 5 to 10 μm.



### 3.2 Fracture toughness and Impact strength properties

CFRP composites possess excellent in-plane mechanical properties which will not be significantly affected by incorporating nano additives into the composite system. Influence of ADG-NH<sub>2</sub> on fracture toughness properties was evaluated by means of SENB test method.  $K_{IC}$  and  $G_{IC}$  values increase with an increment in ADG-NH<sub>2</sub> loading up to 0.5 wt%.

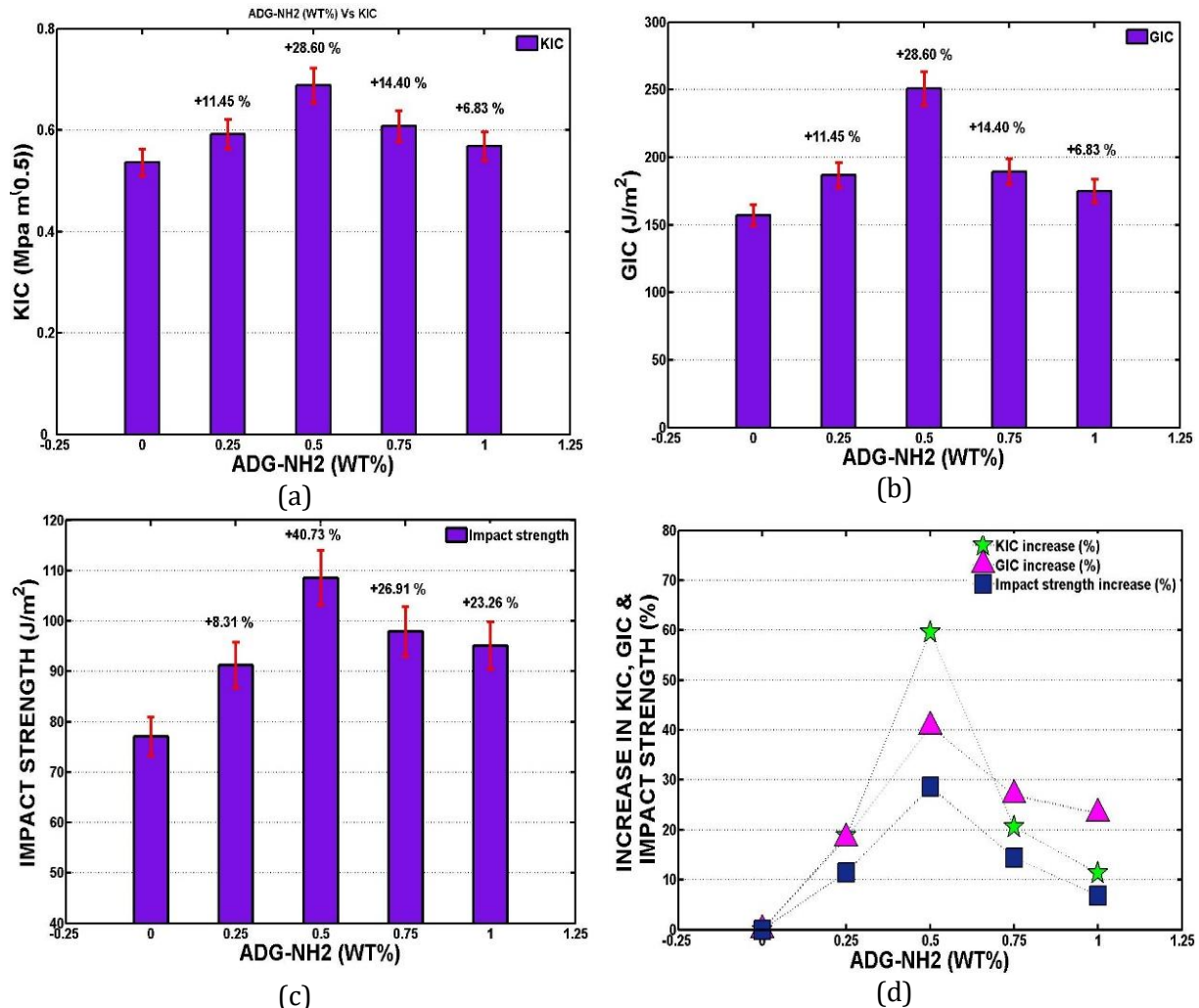


Fig. 7. Variation of (a) Critical stress intensity factor [ $K_{IC}$ ] (b) Critical strain energy release rate [ $G_{IC}$ ] (c) Impact strength [J/m<sup>2</sup>] and (d) % increase in  $K_{IC}$ ,  $G_{IC}$ , Impact strength w.r.t ADG-NH<sub>2</sub> addition in CFRP composite during SENB test

Table 1. Variation of  $K_{IC}$ ,  $G_{IC}$  values observed during the SENB test and Impact strength for different ADG-NH<sub>2</sub> loading relative to the neat epoxy

ADG-NH <sub>2</sub> [wt %]	$K_{IC}$ [MPa m <sup>1/2</sup> ]	Increase [%]	$G_{IC}$ [Jm <sup>-2</sup> ]	Increase [%]	Impact strength [Jm-2]	Increase [%]
Neat	0.67± 0.07	0	157.17 ± 7.86	0	77.15± 3.86	0
0.25	0.74± 0.07	11.45	186.67 ± 9.33	18.77	91.27 ± 4.56	18.31
0.5	0.86± 0.09	28.60	250.71 ± 12.54	59.52	108.57 ± 5.43	40.73
0.75	0.76± 0.08	14.40	189.47 ± 9.47	20.56	97.91 ± 4.90	26.91
1	0.71± 0.07	6.83	174.90± 8.74	11.28	95.09 ± 4.75	23.26

Due to the enhanced chemical bonding between the matrix and ADG-NH<sub>2</sub> and better homogeneous dispersion under controlled environment, the fracture stiffness properties of modified laminates were increased significantly that is evident from Fig.7 and Table 1. The impact resistance properties of the ADG-NH<sub>2</sub> / epoxy / CFRP composites are listed in Table 1. An impact strength

value of 77.15 J/m<sup>2</sup> was observed for neat epoxy CFRP compared to the 108.57 J/m<sup>2</sup> for 0.5 wt % of ADG-NH<sub>2</sub> / epoxy / CFRP, an increase of nearly 40% to the neat epoxy. A notable increase in the resilience to impact for CFRP composite laminate is because the improved bonding strength of the ADG-NH<sub>2</sub>/epoxy resins with carbon fibers. However the impact strength was reduced for ADG-NH<sub>2</sub> of 1 wt % which was 95.09 J/m<sup>2</sup>. As the ADG-NH<sub>2</sub> ratio rises above 0.5 weight percent, there is noticeable reduction in the impact resistive property of CFRP composites. The reduction in impact resistance is because of agglomeration that accumulates in the epoxy resin, which results in non-uniform distribution of inclusion leads to degraded adhesion between fiber and matrix [40,41].

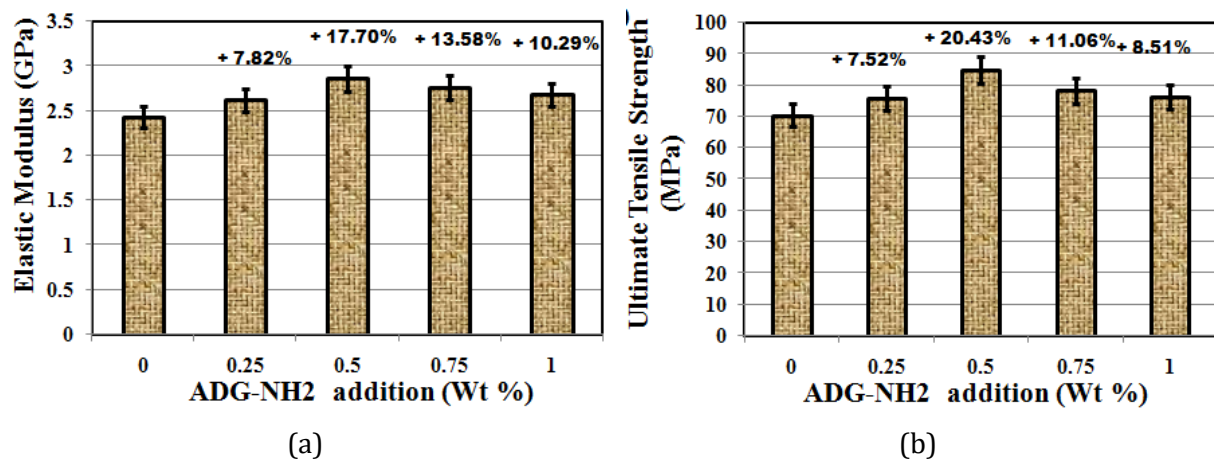
In Table 1, The mode I fracture toughness values [ $K_{IC}$  &  $G_{IC}$ ] of the ADG-NH<sub>2</sub>/epoxy/CFRP composites were improved by the inclusion of amine functionalised graphene particles compared to neat epoxy CFRP. An enhancement in the fracture resistance values of the ADG-NH<sub>2</sub> / epoxy / CFRP were observed up to a ADG-NH<sub>2</sub> ratio of 0.5 wt % and reduces subsequently beyond 0.5 wt% but increases with respect to base structure. Compared to the neat epoxy CFRP laminate an increase of 28.6% observed in  $K_{IC}$  value for 0.5 wt % ADG-NH<sub>2</sub>. This enhancement was observed possibly due to the better dispersion and less agglomeration of the functionalised graphene with carbon fiber composite. This homogenous dispersion will leads to better cross linking through chemical bonding between carbon fiber and epoxy . Hence, resulted to increase in Mode I fracture resistance values.

The reduction in Mode I fracture resistance and the limited enhancement in impact strength at higher ADG-NH<sub>2</sub> loading concentrations (0.75% and 1%) indicate ineffective cross-linking and reduced dispersion of the functionalized graphene. This phenomenon has been noted by various researchers, where excessive loading concentrations weaken the mechanical properties because of re-agglomeration and reduced effectiveness of the reinforcement [34, 42, 43].

ADG-NH<sub>2</sub> functionalized nano-additives improve Mode I fracture and impact resistance through mechanisms such as crack bridging, energy absorption, interfacial bonding, and crack deflection. The interaction of additives and matrix is enhanced by surface functionalization, uniform dispersion, optimized curing parameters, and advanced characterization techniques. By understanding these mechanisms and interactions, we can further optimize the performance of CFRP composites for various applications

### 3.3 Tensile Properties of Neat and ADG-NH<sub>2</sub>/CFRP

The tensile properties, such as elastic modulus (E), ultimate tensile strength (UTS), elongation at break (EL), and toughness (T), which were calculated for both base epoxy and ADG-NH<sub>2</sub>-modified CFRP. A series of ADG-NH<sub>2</sub> loading concentrations 0.25, 0.5, 0.75, and 1 wt% were analysed to determine their effect.



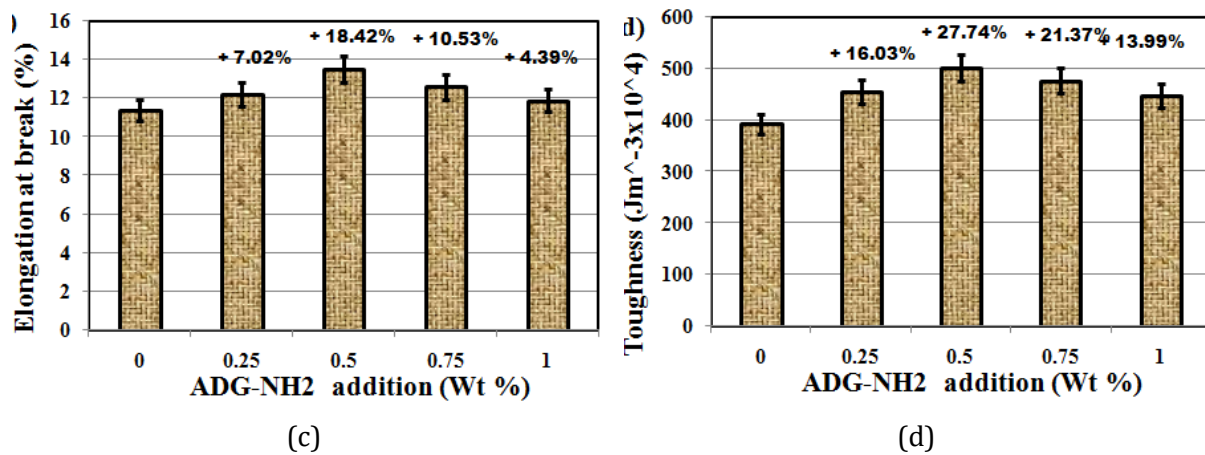


Fig. 8. [a] Elastic modulus, [b] Ultimate tensile strength, [c] Elongation at break [%], [d] Toughness w.r.t ADG-NH<sub>2</sub> addition in CFRP composite during tensile test

Figure 8 presents the results, indicating the mechanical properties with respect to filler concentration. The graphs represent the averages of five test samples, with error bars for each concentration. The study revealed slight improvements in tensile performance with the inclusion of amine-functionalized graphene in CFRP laminates. Detailed comparative data, along with the corresponding increments relative to the base epoxy matrix, are summarized in Table 2.

Table 2. Variation of E, UTS, % EL, and T w.r.t ADG-NH<sub>2</sub> addition in CFRP composite under tensile test

ADG-NH <sub>2</sub> [wt%]	Elastic modulus [GPa]	Increase [%]	Ultimate Tensile strength [MPa]	Increase [%]	Elongation at break [%]	Increase [%]	Toughness [J/m <sup>3</sup> ] x10 <sup>4</sup>	Increase [%]
0	2.43	0.00	70.5	0.00	11.4	0.00	393	0.00
0.25	2.62	7.82	75.8	7.52	12.2	7.02	456	16.03
0.5	2.86	17.70	84.9	20.43	13.5	18.42	502	27.74
0.75	2.76	13.58	78.3	11.06	12.6	10.53	477	21.37
1	2.68	10.29	76.5	8.51	11.9	4.39	448	13.99

As shown in Figure 8[a] there was a distinct increase in the values of elastic modulus ADG-NH<sub>2</sub> / Epoxy weight% loading content of 0.25, 0.5, 0.75 and 1wt % to a value of 7.82 % , 17.70% , 13.58% and 10.29 % respectively with reference to neat epoxy at room temperature. As reported in Table 2, the properties of the ADG-NH<sub>2</sub> / Epoxy loaded laminate was increased upto 0.5 wt% w.r.t neat epoxy laminate. It shows the decreasing tendency of Elastic Modulus(E), Ultimate tensile strength(UTS), Elongation at break(EL) and Toughness(T) beyond 0.5 weight% , However there is a substantial increase in the values with respect to pristine epoxy due to addition of functionalised graphene particles in the CFRP. The integration of functionalized graphene into the epoxy system significantly improves the toughening mechanisms and mechanical properties of CFRP composite laminates, along with a notable increase in stiffness compared to the neat epoxy CFRP composites. Graphene, as a filler, offers an exceptionally large surface area, especially at lower ADG-NH<sub>2</sub> concentrations. The homogeneous dispersion of ADG-NH<sub>2</sub> enables further efficient stress transfer, leading to enhanced tensile strength up to 0.5 wt% compared to base epoxy CFRP laminates. Specifically, the UTS values improved by 20.43% for 0.5 wt%, while EL and T increased by 18.42% and 27.74%, respectively, at the same loading level. However, at higher loading concentrations (0.75 wt% and 1 wt%), a decrease in toughness and elongation at break was observed, likely due to non-homogeneous dispersion in the CFRP matrix. The key highlights is the importance of optimal dispersion for maximizing the performance benefits of ADG-NH<sub>2</sub> in these composites.



### 3.4 Fractography Analysis of ADG-NH<sub>2</sub>/CFRP

The fractography analysis certainly reveals the strengthning mechanism and provides the sufficient corroboration for the fracture toughness improvement with modified ADG-NH<sub>2</sub> in CFRP composite laminate. However, the results clearly show that, when amine-functionalized graphene is added to CFRP composites, the fracture characteristics of the material are improved in comparison to neat carbon fiber reinforced polymer laminates [44,45]. SEM micrograph analysis was carried out to evaluate the failure mechanism and toughening mechanism of the ADG-NH<sub>2</sub> nano material for both neat epoxy and ADG-NH<sub>2</sub>/ CFRP with different wt% filler content [0.25, 0.5, 0.75 and 1] [Refer Fig. 9].

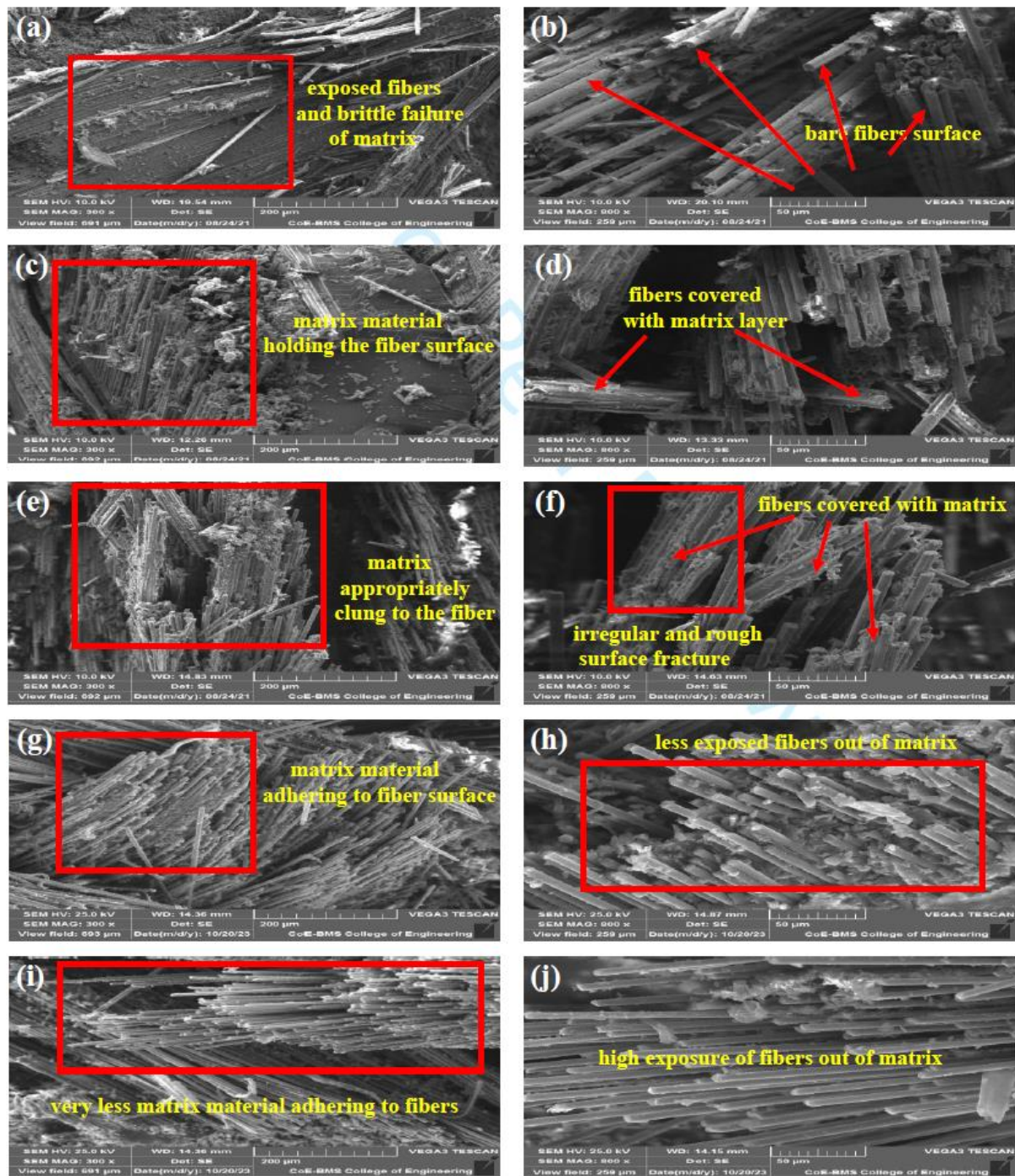


Fig. 9. SEM micrographs [a], [b] neat epoxy CFRP; [c],[d] 0.25 wt % of ADG-NH<sub>2</sub>/CFRP; [e],[f] 0.5 wt % of ADG-NH<sub>2</sub>/CFRP [g],[h] 0.75 wt % of ADG-NH<sub>2</sub>/CFRP; and [i],[j] 1 wt % of ADG-NH<sub>2</sub>/CFRP



Neat and ADG-NH<sub>2</sub>/CFRP composites epoxy with varying filler levels [0.25, 0.5, 0.75, and 1 weight percent] were examined using SEM examination. Amine-functionalized graphene increases fracture toughness, because of its mechanical strength, surface area, wrinkled structure, and good interfacial adhesion with epoxy. The amine groups improve adhesion with the epoxy matrix also aiding uniform distribution of graphene. At higher concentrations [0.75 and 1 wt%], graphene aggregation causes stress concentration and reduced adhesion, negatively impacting performance. SEM images confirm uniform dispersion in lower concentrations but show exposed fibers and poor bonding at 1 wt%, explaining the decline in mechanical performance at higher filler contents.

SEM pictures show weak mechanical properties due to brittle fracture behavior, exposed carbon fibers, and poor bonding in neat epoxy matrix. In contrast, amine-functionalized graphene strengthens adhesion as demonstrated by the firmly bonded carbon fibers in matrix material as shown in the fractured samples in figure 9 [a], [b], [c], [d], [e] and [f] fractured samples. The sample with 0.5 weight percent graphene exhibits the best interfacial strength and rough fracture surfaces, indicating plastic deformation and crack deflection. Increased surface area and energy absorption during fracture propagation are indicated by dimples, which enhance mechanical characteristics.

Performance is adversely affected by graphene aggregation at higher concentrations [0.75 and 1 weight percent], which results in stress concentration and decreased adhesion in figure 9 [g], [h], [i] and [j]. The decrease in mechanical performance at increasing filler amounts can be explained by SEM pictures, which show exposed fibers and weak bonding at 1 weight percent but demonstrate homogeneous dispersion at lower concentrations.

#### **4. Conclusions**

In this study, the effect of amine functionalized graphene [ADG-NH<sub>2</sub>] reinforced CFRP composites has been extensively evaluated for fracture toughness, tensile and impact resistance. The mechanical properties of various CFRP specimens with different wt% were also taken under consideration. The experimental findings have been thoroughly scrutinized, leading to the following key conclusions

- An enhancement in mechanical properties is the consequence of introduction of functionalized ADG-NH<sub>2</sub> to the CFRP laminate up to 1 wt% compared to neat epoxy based CFRP composites.
- For every 0.5 weight percent of ADG-NH<sub>2</sub> reinforced CFRP, the K<sub>IC</sub> and G<sub>IC</sub> values increased up to approximately 29% and 60%, respectively, but decreased at higher concentrations [0.75 wt% & 1 wt%]. Nonetheless, there is gain in the G<sub>IC</sub> and K<sub>IC</sub> values with respect to the base CFRP laminate.
- Compared to neat epoxy CFRP composite laminates, it was determined that 0.5% weight percentage of ADG-NH<sub>2</sub> graphene CFRP laminates increased impact strength by about 41%.
- The tensile characteristics of the 0.5% weight percentage of ADG-NH<sub>2</sub> graphene CFRP laminates showed significant improvements, with an ~18% increase in elastic modulus, ~21% enhancement in UTS, ~19% rise in EL, and ~28% boost in T compared to neat epoxy CFRP composites. These enhancements are attributed towards the superior interaction and bonding at the interface of fiber/matrix. In contrast, the neat epoxy CFRP composites exhibited weaker performance.
- Fractographic analysis demonstrates enhanced adhesion mechanisms and uniform dispersion of amine functionalization of graphene [ADG-NH<sub>2</sub>] aiding an improvement in mechanical and thermal properties of CFRP laminate.
- Due to better mechanical properties, thermal stability and durability, ADG-NH<sub>2</sub> reinforced CFRP composites have various prominent industrial applications such as aerospace, automotive, construction and Sports Equipment etc. However, addressing scalability challenges such as cost-effective production, uniform dispersion, interfacial bonding, environmental impact, and regulatory compliance is crucial for their successful implementation.

## 5. Limitations and Scope for Future Work

During the current research study various assumptions made e.g., uniform additive dispersion, specific impact conditions, consistent hand layup process, room temperature curing etc. Hence, this research study can be extended further for testing under varied environmental test conditions such as temperature, humidity, or UV exposure, optimizing dispersion techniques, or exploring other additive concentrations, quantitative data for analyzing fracture surfaces [e.g., particle distribution, void content, or interfacial adhesion strength], and detailed statistical analyses for the experimental results should be explored in the future to gain deeper insights into the mechanical performance improvements of modified CFRP composites.

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