# Nanoscale reinforcements for aerostructural bonding systems: Characterization and performance analysis

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Functionalized CNTs-OH and Graphene-COOH offer a promising hybrid filler system for enhancing epoxy resin composites, especially for space applications. By introducing polar functional groups, these fillers achieve superior dispersion, strong interfacial bonding, and reduced agglomeration within the epoxy matrix. The mechanical improvements achieved are at the same level as advanced methods like CVD, while functionalization simplifies processing, improves scalability, and ensures compatibility with epoxy. These advantages make functionalized hybrids ideal for lightweight, high-strength, and durable materials required in extreme environments, addressing the challenges of space technology while balancing performance and manufacturability.

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### 1. Introduction

The demand for advanced materials with exceptional mechanical properties has grown exponentially with the increasing reliance on satellites for communication, navigation, and scientific exploration [1-10]. These materials must endure extreme mechanical stresses during launch, such as high g-forces, as well as harsh orbital conditions characterized by temperature fluctuations, radiation, and micro-meteoroid impacts. Epoxy resins, widely used as structural adhesives and matrices, offer good mechanical properties, thermal stability, and ease of processing. However, conventional epoxy systems often fall short of the stringent requirements for modern spacecraft applications, necessitating reinforcement with high-performance fillers to enhance their strength and durability.

Nanomaterials such as carbon nanotubes (CNTs) and graphene have emerged as promising reinforcements for polymer matrices due to their extraordinary tensile strength, stiffness, and thermal conductivity [11-20]. Individually, CNTs provide crack-bridging capabilities and high tensile strength, while graphene contributes exceptional stiffness, large surface area, and load transfer efficiency. The combination of these materials into a hybrid system introduces an unprecedented opportunity to synergize their complementary properties, creating a material with superior performance characteristics. Such hybridization is particularly advantageous for applications like satellite housings, where both tensile and flexural strength are critical for structural integrity under extreme conditions.

This study focuses on enhancing the mechanical properties of epoxy resin by reinforcing it with a hybrid filler system consisting of hydroxyl-functionalized CNTs (CNTs-OH) and carboxylfunctionalized graphene (graphene-COOH). Functionalization not only facilitates strong interfacial bonding between the fillers but also ensures their uniform dispersion within the resin matrix, mitigating agglomeration and optimizing load transfer. The resulting hybrid composite demonstrates

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significant improvements in tensile and flexural strength compared to conventional epoxy systems, making it a promising candidate for space applications.

Furthermore, the epoxy resin developed in this study represents the first step toward the creation of carbon fiber-reinforced polymer (CFRP) composites. CFRPs, which combine epoxy resin with carbon fiber reinforcements, are widely recognized for their high strength-to-weight ratio and excellent thermal stability, making them ideal for space applications [21-27]. By developing a high-performance epoxy matrix with optimized mechanical properties, this research lays the groundwork for manufacturing CFRPs tailored to withstand the extreme demands of space environments. The incorporation of functionalized CNTs and graphene into the epoxy matrix not only enhances its standalone properties but also paves the way for advanced CFRP materials that can provide superior reliability and longevity in space technology.

Despite the significant potential of combining CNTs and graphene, their application in epoxy matrices remains insufficiently explored, particularly for space-related technologies. Existing studies predominantly employ techniques such as solution blending or chemical vapor deposition (CVD) to incorporate hybrid fillers. For instance, Yue et al. [28] utilized a CNTs/Graphene hybrid with a 2:8 ratio and employed solution blending, resulting in a modest 12% increase in tensile strength. Similarly, Li et al. [29] achieved a 12% improvement in tensile strength using a CNTs/GO hybrid with a 1:3 ratio prepared through solution blending. In another study, Li et al. [30] applied mechanical mixing to a CNTs/Graphene hybrid with a 1:1 ratio, which improved tensile strength by 20%. Notably, in the same study, switching to the CVD method for the same hybrid increased tensile strength by up to 36%, demonstrating the superior performance of CVD over other techniques in enhancing mechanical properties.

CVD, however, is a complex, high-temperature process that is energy-intensive and requires specialized equipment, making it less practical for large-scale production. Additionally, the harsh conditions during CVD can introduce defects in the graphene or CNTs, compromising their intrinsic mechanical, thermal, and electrical properties. This can reduce the overall reinforcement efficiency of the hybrid fillers in epoxy matrices.

Furthermore, CVD-grown hybrids often face challenges in achieving strong interfacial bonding with epoxy resins, as the bonding between CNTs and graphene is primarily physical, rather than chemical. This limits load transfer efficiency and mechanical performance, particularly under the extreme stresses encountered in space environments. The lack of chemical compatibility also increases the likelihood of filler agglomeration, leading to weak points within the composite. These drawbacks highlight the need for alternative approaches, such as functionalization, which can address these limitations by enhancing interfacial bonding, improving dispersion, and preserving the intrinsic properties of the fillers. Functionalization of CNTs and graphene introduces reactive groups that not only create a stronger hybrid network but also enable effective chemical bonding with the epoxy matrix, making it a more practical and scalable solution for aerospace-grade composites.

This study addresses these limitations by employing functionalization and ultrasonic dispersion techniques to create a hybrid composite with excellent performance, scalability, and suitability for aerospace-grade applications. By bridging this research gap, the present work contributes a novel approach to developing lightweight, durable, and high-strength materials specifically designed to meet the rigorous demands of satellite housings.

### 2. Experimental

The epoxy system utilized in this study consists of two components. The epoxy resin, D.E.R. 331, manufactured by Dow Chemicals (USA), belongs to the Bisphenol A Epoxy Resin group. This non-volatile resin is known for its high rigidity, excellent adhesion, and superior chemical resistance. Key properties of D.E.R. 331 include a water content of 700 ppm max, an epoxide equivalent weight of 182–192 g/eq, viscosity at 25°C ranging from 11,000–14,000 mPa·s, specific gravity of 1.16 g/ml at 25°C, a maximum color index (Platinum-Cobalt scale) of 75, and a shelf life of 24 months.



Fig. 1. SEM image showcasing the CNTs (a) and graphene (b) utilized in this study.

The curing agent, D.E.H. 24, also from Dow Chemicals (USA), complements the epoxy resin with its unique properties. It features a water content of 0.5 wt% max, an amine hydrogen equivalent weight of 24 g/eq, viscosity at 25°C between 19.5–22.5 mPa·s, specific gravity of 0.981 g/ml at 25°C, a maximum color index of 50 (Platinum-Cobalt scale), and a shelf life of 24 months.

The reinforcement materials, graphene nanoplatelets (Gr) and multi-walled carbon nanotubes (MWCNTs) supplied by ACS Material, were functionalized to enhance their compatibility with the epoxy matrix. Graphene nanoplatelets were treated in a 1:3 solution of nitric acid and sulfuric acid (HNO<sub>3</sub>:H<sub>2</sub>SO<sub>4</sub>) at 70°C under continuous magnetic stirring for 5 hours. After thorough rinsing with distilled water, the treated graphene was dispersed in ethylene glycol via ultrasonication for 30 minutes to form a Gr-COOH dispersion (1 g/L). Similarly, MWCNTs were subjected to the same acid treatment, rinsed, and vacuum-dried for 24 hours. These were further reacted with thionyl chloride (SOCl<sub>2</sub>) at 60°C for 6 hours to yield MWCNT-COCl, which was subsequently washed, dispersed in ethylene glycol, and stirred at 120°C for 8 hours. The resulting MWCNT-OH dispersion (1 g/L) was finalized via ultrasonication.



Fig. 2. (a) Clumping of the CNTs/Gr hybrid material prior to functionalization; (b) Improved dispersion of the CNTs/Gr hybrid material following functionalization.

To create the Gr–MWCNT hybrid dispersion, equal volumes of the Gr-COOH and MWCNT-OH dispersions were combined in a 1:1 ratio. The mixture underwent ultrasonication for 45 minutes in an ice-water bath to ensure homogeneity. Various hybrid filler concentrations (0.05, 0.1, 0.2, 0.3, 0.4, and 0.5 wt%) were prepared by diluting or concentrating the dispersions to meet experimental requirements. To test the mechanical properties of the resulting hybrid composite, epoxy resin reinforced with Gr-MWCNT hybrids was cast in molds designed according to TCVN

4501 tensile strength standards. For flexural strength testing, a rectangular mold was fabricated according to the ASTM D790 standard dimensions.



Fig. 3. Schematic representation of functionalization and hybridization of CNTs and graphene.

The liquid epoxy mixture was poured into the molds, cured in an oven for 24 hours, and demolded from pre-coated molds to ensure smooth release. The mechanical properties of the cured samples were evaluated using an AGX-50k NVD multifunctional testing machine at Institute of Tropical Technology, Vietnam Academy of Science and Technology. Each mechanical test was conducted on three identical samples, and the average value was recorded to ensure data reliability.



Fig. 4. (a) An epoxy casting mold designed to conform to the TCVN 4501 standard dimensions;
(b) An AGX-50k NVD multifunctional testing machine utilized at the Institute for Tropical Technology to evaluate mechanical strength.

## 3. Results

### 3.1. Spectral characterization of CNTs/Graphene hybrid material

- Figure 5a (C1s Spectrum of Pristine CNTs):

+  $sp^2$  Peak (Red): Located around 284.5 eV, corresponding to the graphitic carbon backbone, which is characteristic of the  $sp^2$  hybridization in CNTs;

+ sp<sup>3</sup> Peak (Blue): Around 285.5 eV, confirming the pristine state of CNTs with limited functionalization or defects.

- Figure 5b (Ols Spectrum of Pristine CNTs): reflects a pristine surface with minimal oxygen-containing functional groups, supporting that these CNTs are largely unfunctionalized, with:

+ C=O Peak (Blue): Around 532.8 eV, indicating a small amount of carbonyl groups on the surface;

+ Acid Anhydride Peak (Red): At ~535 eV, due to minor oxidation or adsorbed oxygen.

- Figure 5c (C1s Spectrum of Pristine Graphene):

+ sp<sup>2</sup> Peak (Red): Around 284.5 eV, representing the dominant graphitic sp<sup>2</sup> carbon network of pristine graphene;

+ sp<sup>3</sup> Peak (Blue): Near 285.5 eV, indicating a very low degree of defects or functionalization.

- Figure 5d (O1s Spectrum of Pristine Graphene): Pristine graphene shows minimal oxygen content, consistent with the expected surface chemistry of high-purity graphene:

+ C-O Peak (Red): Near 533.0 eV, indicating minor oxidized carbon species on the surface.

- Figure 5e (C1s Spectrum of CNTs-OH/Graphene-COOH Hybrid): Successful functionalization and hybridization:

+ sp<sup>2</sup> Carbon (Cyan): Peak around ~284.5 eV, representing the graphitic backbone of CNTs and graphene with sp<sup>2</sup> hybridization, contributing to their excellent mechanical and electrical properties.

+ sp<sup>3</sup> Carbon (Blue): Peak around  $\sim$ 285.5 eV, arising from sp<sup>3</sup>-hybridized carbons due to defects or functional groups (-OH, -COOH), disrupting the sp<sup>2</sup> structure and increasing reactivity.

+ C-O (Green): Peak around ~286.5 eV, indicating hydroxyl (-C-OH) or ether (-C-O-C-) groups introduced during oxidation, aiding dispersion and functionalization.

+ C=O (Purple): Peak near ~288.5 eV, corresponding to carbonyl (-C=O) groups formed at defects or edges, enabling further chemical bonding.

+ COOR (Red): Peak around ~289.0 eV, signifing ester (-COOR) groups formed during hybridization, reflecting covalent bonding between CNTs and graphene.

- Figure 5f (O1s Spectrum of CNTs-OH/Graphene-COOH Hybrid): The spectrum confirms the coexistence of functional groups (carboxyl and hydroxyl) from both CNTs and graphene, with peaks demonstrating successful hybridization into a composite material.

+ C=O (Graphene-COOH) (Red): Around 531.8 eV, attributed to carboxyl (-COOH) groups specifically on the graphene component. These groups are introduced during the oxidation process of graphene nanoplatelets.

+ C-O (Both) (Blue): At ~533 eV, representing hydroxyl (-C-OH) or ether-like (-C-O-C-) groups present in both CNTs and graphene. This peak arises because the C-O bond types, such as hydroxyl (-C-OH) or ether (-C-O-C-), are commonly introduced during oxidative functionalization. Both CNTs and graphene undergo chemical treatments (e.g.,  $H_2SO_4/HNO_3$  oxidation) that generate similar oxygen-containing functional groups. On graphene, these groups typically form at edge sites or defect regions, while on CNTs, they appear on tube walls or open ends. Since these functional groups are chemically similar and present on both materials, their signals overlap in the XPS spectrum, resulting in the combined C-O (Both) peak..

+ Acid Anhydride (CNTs-OH) (Green): Near 534.5 eV, corresponding to acid anhydrides formed by the condensation of carboxyl (-COOH) groups on CNTs. These are likely oxidative byproducts specific to CNTs during functionalization.

+ H<sub>2</sub>O/Adsorbed Oxygen (Purple): At ~536.0 eV, attributed to water molecules or oxygen adsorbed on the surface of the hybrid material. This is a common feature in functionalized materials exposed to ambient air.



Fig. 5. (a) Deconvoluted C1s XPS Spectrum of Pristine CNTs; (b) Deconvoluted O1s XPS Spectrum of Pristine CNTs; (c) Deconvoluted C1s XPS Spectrum of Pristine Graphene; (d) Deconvoluted O1s XPS of Pristine Graphene; (e) Deconvoluted C1s XPS Spectrum of CNTs-OH/Graphene-COOH Hybrid; (f) Deconvoluted O1s XPS Spectrum of CNTs-OH/Graphene-COOH Hybrid.

#### 3.2. Tensile and flexural strength

The results from Figure 6a, 6b and 6c highlight distinct trends in tensile and flexural strength for varying filler concentrations:

- For the CNTs-OH system, tensile strength starts at 60.0 MPa with no filler (0.0 wt%) and increases steadily to a peak of 71.0 MPa at 0.4 wt%, representing an 18.3% improvement over the baseline. However, at 0.5 wt%, the tensile strength slightly decreases to 68.7 MPa, indicating a decline beyond the optimal concentration. Similarly, flexural strength begins at 80.0 MPa and reaches a peak of 93.5 MPa at 0.4 wt%, showing a 16.9% increase. A slight reduction is observed at 0.5 wt%, where flexural strength drops to 90.0 MPa.

- For the Graphene-COOH system, the tensile strength improves significantly, starting at 60.0 MPa and increasing to 72.0 MPa at 0.2 wt%, a 20.0% improvement over the baseline. Beyond

0.2 wt%, the tensile strength gradually declines, reaching 69.5 MPa at 0.5 wt%. The flexural strength follows a similar trend, starting at 80.0 MPa and peaking at 95.0 MPa at 0.2 wt%, an 18.8% improvement. Like tensile strength, flexural strength decreases steadily at higher concentrations, reaching 92.0 MPa at 0.5 wt%.

- The CNTs/Graphene hybrid (1:1) system exhibits the most significant improvements among the three systems. Tensile strength starts at 60.0 MPa and steadily increases, peaking at 81.5 MPa at 0.3 wt%, which corresponds to a 35.8% improvement from the baseline. At higher concentrations, tensile strength slightly decreases, reaching 78.0 MPa at 0.5 wt%. Flexural strength shows a similar trend, starting at 80.0 MPa and peaking at 106.0 MPa at 0.3 wt%, reflecting a 32.5% increase. Beyond this optimal concentration, flexural strength declines slightly, recording 102.9 MPa at 0.5 wt%.





Fig. 6. Effect of (a) CNTs-OH concentration on tensile and flexural strength; (b) Graphene-COOH concentration on tensile and flexural strength; (c) CNTs/Graphene hybrid (1:1 wt%) concentration on tensile and flexural strength.

#### 4. Discussions

The incorporation of hydroxyl-functionalized CNTs (CNTs-OH) and carboxylfunctionalized graphene (Graphene-COOH) into epoxy resin matrices represents a significant advancement in the development of lightweight, high-strength materials for space applications. This study demonstrates the synergistic effects of these hybrid fillers in enhancing the mechanical properties of epoxy composites, with particular emphasis on tensile and flexural strength.

*Functionalization Effectiveness:* The XPS analysis clearly validates the successful functionalization of both CNTs and graphene, as evidenced by the emergence of distinct chemical peaks. For CNTs-OH, the O1s spectrum indicates the presence of hydroxyl groups, with a peak corresponding to acid anhydride formation likely resulting from partial oxidation during the functionalization process. Similarly, the C1s spectrum of Graphene-COOH shows prominent sp<sup>2</sup> and sp<sup>3</sup> hybridization peaks, alongside carboxyl-related signals, confirming the introduction of carboxylic functional groups. These functional groups are essential for improving dispersion and interfacial bonding with the epoxy matrix, mitigating agglomeration and enhancing load transfer.

*Synergistic Hybridization:* The hybrid system of CNTs-OH and Graphene-COOH exhibits exceptional synergy, as confirmed by the XPS spectra of the hybrid composite. Peaks corresponding to C=O and COOR bonds demonstrate strong chemical interactions between the functional groups of the hybrid fillers, enabling a robust network within the epoxy matrix.

Saturation Behavior of Graphene, CNTs, and Hybrids in Epoxy Composites: The saturation concentration of graphene (0.2%) and CNTs (0.4%) in epoxy composites can be attributed to their distinct structural characteristics and interaction mechanisms within the matrix. Graphene, a two-dimensional material, has an exceptionally high surface area and strong in-plane sp<sup>2</sup> bonding, which promotes efficient load transfer at low concentrations. However, its planar geometry and tendency to restack or aggregate due to van der Waals forces can limit its dispersion at higher concentrations, leading to saturation of its reinforcing effect. In contrast, CNTs, with their one-dimensional tubular structure, provide a larger aspect ratio and better crack-bridging ability, allowing for improved mechanical reinforcement up to higher filler concentrations. Their inherent flexibility and reduced likelihood of aggregation compared to graphene also contribute to this increased saturation threshold.

In a hybrid system of graphene and CNTs, the complementary nature of these fillers is expected to create an optimal network structure within the epoxy matrix. Graphene contributes superior load transfer efficiency and stiffness, while CNTs enhance tensile properties and prevent crack propagation. This synergistic interaction between the two materials improves dispersion and reduces filler aggregation, allowing the hybrid system to saturate at an intermediate concentration level of 0.3%. This concentration represents a balance where both materials can effectively interact with the matrix and each other, maximizing the composite's mechanical properties while avoiding the detrimental effects of agglomeration or oversaturation.

*Mechanical Performance:* The mechanical testing results highlight distinct trends for different filler systems:

- CNTs-OH: The functionalized CNTs exhibit a gradual increase in tensile and flexural strength with filler concentration, peaking at 0.4 wt%. This improvement can be attributed to enhanced dispersion and interfacial bonding due to hydroxyl functionalization.

- Graphene-COOH: The carboxyl-functionalized graphene demonstrates a higher peak improvement at a lower filler concentration (0.2 wt%) compared to CNTs, likely due to its superior surface area and chemical reactivity.

- Hybrid System: The CNTs/Graphene hybrid system (1:1 ratio) achieves the most significant performance enhancements, with tensile strength improving by up to 35.8% and flexural strength by 32.5%. The hybrid fillers effectively reinforce the epoxy matrix, with the optimal performance achieved at 0.3 wt%. Beyond this concentration, the slight decline in strength suggests potential issues with filler agglomeration or matrix saturation.

The enhancement in mechanical performance of the hybrid system can be attributed to the following factors:

+ Enhanced Interfacial Bonding: Functionalization of CNTs with hydroxyl (-OH) groups and graphene with carboxyl (-COOH) groups introduces oxygen-containing functional groups, which enhance chemical compatibility and bonding with the surrounding matrix (e.g., polymer or resin). Covalent bonds or hydrogen bonds between the functionalized nanofillers and the matrix result in improved stress transfer, similar to the strong interfacial bonding achieved in CVD-grown hybrids.

+ Improved Dispersion: Functionalization reduces the van der Waals forces between CNTs and graphene, promoting better dispersion of the hybrid nanofillers within the composite matrix. This uniform distribution helps prevent stress concentrations and maximizes the load-bearing capacity of the composite, comparable to the well-aligned nanostructures obtained via CVD.

+ Synergistic Load Sharing: The combination of CNTs and graphene leverages their complementary properties. CNTs contribute exceptional tensile strength and flexibility, while graphene provides high stiffness and in-plane strength. This synergy mimics the aligned network structure of CVD-grown hybrids, leading to an efficient distribution of applied stress across the composite.

+ Functionalization-Induced Defects and Anchoring: While functionalization introduces defects, these defects can act as anchoring points, enhancing mechanical interlocking with the matrix. This effect can counteract the minor loss in intrinsic properties caused by functionalization, maintaining mechanical performance comparable to pristine nanomaterials integrated via CVD.

+ Microstructure Optimization: The ultrasonic treatment and hybridization process (e.g., ultrasonication of CNTs-OH and graphene-COOH) likely promote intimate mixing and the formation of a 3D interconnected network. This interconnected network can rival the highly ordered structures typically achieved in CVD hybrids.

*Comparison with Previous Work:* The functionalization approach used in this study compares favorably with existing methods such as chemical vapor deposition (CVD) and solution blending. While CVD achieves superior mechanical improvements due to its high degree of alignment and chemical purity, it is energy-intensive, prone to inducing defects, and less scalable. The functionalization and ultrasonic dispersion methods employed here compensate for the lack of CVD-induced alignment by enhancing chemical interactions, dispersion, and load-sharing within the matrix, leading to comparable improvements in mechanical properties. Therefore, the hybrid offers a practical and scalable alternative, ensuring both compatibility with the epoxy matrix and preservation of the fillers' intrinsic properties. These findings highlight the balance between performance enhancement and manufacturing feasibility.

## **5.** Conclusions

This study demonstrates the successful functionalization and hybridization of CNTs-OH and Graphene-COOH fillers, resulting in significant mechanical property enhancements in epoxy composites. The hybrid system leverages the complementary properties of CNTs and graphene, achieving optimal tensile and flexural strength improvements at a filler concentration of 0.3 wt%. Compared to traditional methods, the functionalization approach ensures better dispersion, scalability, and compatibility with epoxy matrices.

Future work will focus on optimizing the hybrid filler ratio, exploring advanced dispersion techniques, and extending this approach to CFRP composites to meet the increasing demands of space technology. The scalability and performance of these materials will be further evaluated for potential commercial applications in lightweight, high-strength spacecraft components.

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