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Editorial

Each volume gathers contributions on specific topics:

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This collection contains the proceedings of the 21st European Conference on Composite Materials (ECCM21), held in Nantes, France, July 2-5, 2024. ECCM21 is the 21st in a series of conferences organized every two years by the members of the European Society of Composite Materials (ESCM). As some of the papers in this collection show, this conference reaches far beyond the borders of Europe.

The ECCM21 conference was organized by the Nantes Université and the Ecole Centrale de Nantes, with the support of the Research Institute in Civil and Mechanical Engineering (GeM).

Nantes, the birthplace of the novelist Jules Verne, is at the heart of this edition, as are the imagination and vision that accompany the development of composite materials. They are embodied in the work of numerous participants from the academic world, but also of the many industrialists who are making a major contribution to the development of composite materials. Industry is well represented, reflecting the strong presence of composites in many application areas.

With a total of 1,064 oral and poster presentations and over 1,300 participants, the 4-day event enabled fruitful exchanges on all aspects of composites. The topics that traditionally attracted the most contributions were fracture and damage, multiscale modeling, durability, aging, process modeling and simulation and additive manufacturing.

However, the issues of energy and environmental transition, and more generally the sustainability of composite solutions, logically appear in this issue as important contextual elements guiding the work being carried out. This includes bio-sourced composites, material recycling and reuse of parts, the environmental impact of solutions, etc.

We appreciated the high level of research presented at the conference and the quality of the submissions, some of which are included in this collection. We hope that all those interested in the progress of European composites research in 2024 will find in this publication sources of inspiration and answers to their questions.

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HOW CAN HIERARCHICAL COMPOSITES WITH CARBON NANOTUBE-GRAFTED CARBON FIBRES ENHANCE COMPRESSION PERFORMANCE?

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Keywords: carbon nanotubes, carbon fibres, interphase, interfacial shear strength, epoxy, polypropylene, grafted fibre, fuzzy fibres, hairy fibres, chemical vapour deposition, compression

Abstract

Compressive strength of carbon fibre reinforced polymer (CFRP) composites underperforms relative to tensile strength by up to 50%. This research explores the effects of grafting carbon nanotubes (CNTs) uniformly onto carbon fibres (CF), which increases the apparent interphase volume and interfacial shear strength. CNT-grafted-CF were synthesised via a continuous chemical vapour deposition process, achieving uniform CNT coverage with controlled CNT network corona lengths ranging between 363 to 831 nm. This process increased the specific surface area by more than an order of magnitude to 5.35 $m^2.g^{-1}$ with CNT loadings up to 2.9 wt.% (relative to the CF). Semi-empirical calculations indicated potential improvements with CNT-grafted-CF compared to unsized CF in composite longitudinal stiffness and tensile strength, primarily due to a projected 10% increase in effective fibre volume fraction at a primary fibre content of 0.4 V_f. Compression strength of CFRP could be even greater, with an estimated 27% increase in interphase shear modulus, as predicted by the modified Halpin-Tsai equation.

1. Introduction

In the development of carbon fibre reinforced polymer (CFRP) composites, improvements in compressive strength have lagged behind tensile strength. CFRP compressive strength can be lower than 50% of its tensile strength [1]. Composite compression strength is known to be influenced by matrix shear properties, but the interphase may be controlling in some cases. A reduction in the fibre-matrix interphase regions may significantly reduce the uniaxial compression strength of the overall composite [2]. Grafting carbon nanotubes (CNTs) onto carbon fibres (CFs) increases interphase volume and enhances the interfacial shear strength (IFSS) by improving fibre-matrix interactions and fibre surface wettability [3-5] . An improvement in compressive strength (up to 26%) in epoxy-based composites has been demonstrated when incorporating CNTs onto the CF surface [6-8].

The synthesis of CNT-grafted-CF has been successfully scaled from a batch to a continuous process [9], resulting in significantly enhanced CNT growth from sparse to dense, achieving uniform coverage along a 12K CF tow. The length of CNTs was controlled by varying the pull-through speed (1.92 - 3.36 $m.h^{-1}$) in a bespoke continuous chemical vapour deposition (CVD) reactor. This process produced a range of CNT-grafted-CF configurations with CNT network corona lengths (the thickness of the grafted layer) ranging from 363 to 831 nm. The resultant grafted fibres exhibited a substantial increase in specific surface area, reaching 5.35 $m^2.g^{-1}$, compared to the baseline unsized CF (0.46 $m^2.g^{-1}$). Calculations were performed to determine the effective fibre volume fraction, interphase and bulk matrix moduli, and shear modulus to estimate the anticipated influence of CNT grafting on the mechanical properties of CF composites.

2. Materials and methods

Commercially available unsized polyacrylonitrile (PAN)-based CF (AS4-12K, 7.1 µm diameter, 0.858 $g.m^{-1}$) supplied by Hexcel Composites (Hexcel, GB) was used as a continuous tow. The catalyst precursor was prepared using iron (III) nitrate nonahydrate (≥98% ACS reagent, Merch, DE), nickel (II) acetylacetonate (≥98%, VWR, GB) and ethanol (>99.7% BDH Prolabo, VWR, GB). Acetylene in nitrogen (N_2 98.7 vol% and C_2H_2 1.3 vol%, C certificate, BOC gases, GB), hydrogen in nitrogen (N_2 97.6 vol% and H_2 2.4 vol%, C certificate, BOC gases, GB), nitrogen (99.998 vol% minimum, BOC gases, GB) were used for continuous CNT synthesis. A Gurit Prime 27 epoxy resin with its associated Prime Extra Slow hardener (Marineware Ltd.) with cured density of 1.14 $g.cm^{-3}$ was used as matrix of choice for calculations and future composite rods manufacturing. All chemicals and materials were used as received.

2.1 Continuous synthesis of carbon nanotube grafted carbon fibres

Bi-catalyst deposited CF tow (1 wt.%, Ni:Fe = 1:0.64 mol) was continuously fed through a three-zone CVD reactor and subjected to different gas environments, achieved through an adjustable arrangement of internal quartz tubes, at varying pull-through speeds (1.92 - 3.36 $m.h^{-1}$). Upon entering the furnace, the CF tow was initially exposed to a nitrogen atmosphere (10000 sccm). In the hot zone (550 °C), the tow encountered a mixture of hydrogen in nitrogen (2.4 vol.% hydrogen, 1700 sccm) to facilitate catalyst reduction. Post-reduction, the tow was introduced to acetylene in nitrogen (1.3 vol.% acetylene, 40 sccm), serving as a carbon source for CNT synthesis. The synthesised CNT-grafted-CF passed through another nitrogen region before exiting the reactor and being collected on a spool.

2.2 Characterization of the carbon fiber morphology

As-received fibres and CNT-grafted-CFs morphologies were captured using a Zeiss Sigma 300 high-resolution field emission gun scanning electron microscope (SEM) with an acceleration voltage of 5 kV and an in-lens detector for high spatial resolution at a working distance of ca. 7 mm. SEM specimens were prepared on 25 mm diameter aluminium stubs. The 12K tow was spread on the stub and attached using silver DAG, this allowed a clear global view of all fibres. To limit observer bias and data misinterpretation, a statistical sampling approach was adopted. A minimum of 65 fibres were randomly selected and imaged to achieve a 95% confidence level. Images were captured at magnifications of x10k and x200k for detailed evaluation of CNT network corona length and CNT diameter, which were analysed using ImageJ software (version 1.53a, National Institutes of Health).

2.3 Surface area characterization of fibres

The specific surface area of as-received CF and CNT-grafted-CFs was determined using the Brunauer, Emmett and Teller (BET) method following the ISO 9277 standard [10] using a Quantachrome NOVAtouch gas sorption analyser with N_2 (Anton Paar GmbH, AT). Gas sorption data was processed in the Quantachrome TouchWin software (version 1.22, Anton Paar GmbH, AT). To achieve reliable results, the tests were conducted on four samples of each set which consisted of 12 K fibre tows of approximately 2 m long. The samples were degassed in N_2 for at least 6 h at 100 °C before characterization.

3. Results and discussion

3.1 Controllable growth of uniform CNT on carbon fibres surface

Achieving homogeneous growth of CNTs on CF tow via a continuous CVD process is challenging due to common sparse and uneven growth results, often arising from variations in CVD parameters and catalyst application. Precise control over the CVD parameters and catalyst preparation is essential to ensure uniform CNT growth and prevent induced stress concentrations in composites [11]. Through experimental observations, an optimized CVD parameter space was identified, enabling higher catalyst activity and uniform growth across the entire 12K CF tow based on the statistical sampling analysis of SEM micrographs (Figure 1.C). This process allowed for controllable variation of the CNT network corona length (Figure 3.A) by adjusting the pull-through speed, effectively altering the dwell time in the growth zone. The CNT network corona length was controlled in a range of 363 to 831 nm while maintaining a consistent CNT diameter of 10 nm, resulting in systematic increase in specific surface area (2.30 - 5.35 $m^2.g^{-1}$) . The independently measured increase in specific surface area, grafted CNT mass loading, and CNT average diameter, are mutual self-consistent based on the expected geometric surface area of the CNTs. The CNT loading was estimated using (Eq.1) where $m_{CNT\text{-}g\text{-}CF}$ is the mass of the CNT-grafted-CF and $m_{Bi\text{-}cat}$ is the mass of the bi-catalyst loaded CF. To measure the differences in weight per unit length accurately, a tow sample length of 10 m, was used to determine a CNT loading of 2.9 wt.% for the CNT-grafted-CF 12K tow synthesised at 1.92 $m.h^{-1}$.

$$CNT\ loading\ \% = \frac{m_{CNT-g-CF} - m_{Bi-cat}}{m_{Bi-cat}} \times 100 \quad (1)$$

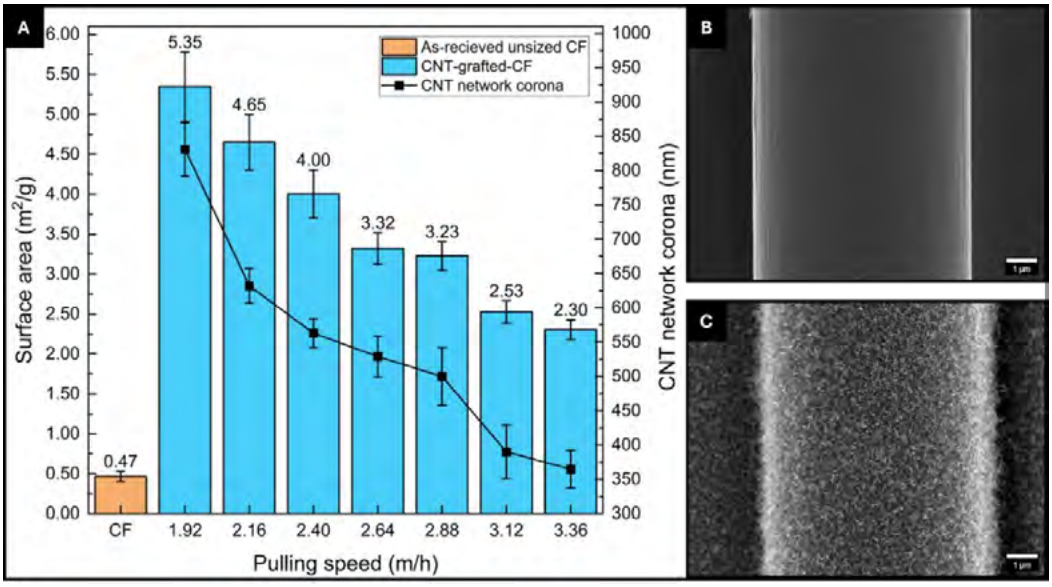

Figure 1. (A) Specific surface area measurements and CNT network corona lengths as a function of varying pull-through speeds. (B) SEM micrograph of as-received unsized carbon fibre. (C) SEM micrograph of CNT-grafted-carbon fibre at a pulling speed of 1.92 $m.h^{-1}$.

3.2 Mechanisms of compressive strength improvement via carbon fibre CNT-Grafting

CNT-grafted-CFs may enhance the composite compressive strength by maximizing the fibre volume fraction without compromising the effective fibre volume fraction. At higher fibre volumes, fibre misorientation is reduced due to closer packing. However, increased fibre-fibre interactions (touching) decrease the interfacial surfaces, thereby reducing the effective fibre volume fraction due to induced stress concentrations. This reduction can be estimated using the fibre interaction probability ratio (Eq. 2), derived from an experimentally verified model, where V_f is the fibre volume fraction and n is fibre interaction probability ratio [12]. For a fibre volume range of 0.4 - 0.7, n is estimated to be 0.75. Introducing CNTs around each fibre in principle can minimise the probability of fibre-fibre interactions due to the high stiffness of the CNT corona; as a result, the effective fibre volume fraction should approach the actual fibre volume fraction (Figure 2). In addition, the CNT grafting is expected to improve the interfacial shear strength (IFSS) [13] and enhance the wettability of CF [14], providing further enhancement of the effective fibre volume fraction. As an example, at V_f ~40%, approximately 10% improvement in longitudinal composite stiffness is expected which would even be greater at higher fibre volume fractions. Similar improvements in strength may be anticipated if load is distributed more effectively.

$$V_{f\,effective} = V_f(1 - n^2V_f^2) \tag{2}$$

In addition, the CNTs create a reinforced interface that directly increases IFSS and the broader properties of an enlarged interphase volume, potentially including all of the matrix volume, depending on primary fibre volume fraction and CNT corona size. This enhancement should facilitate better load transfer and reduce stiffness mismatch, which may simultaneously minimize debonding and delay the onset to shear instabilities (micro-buckling). The elastic modulus of the CNT modified interphase and bulk matrix composite can be estimated using the modified Halpin-Tsai equation (Eq.3-5) [14-16] where $E_{c(i,m)}$, E_m (3.2 GPa), and E_{CNT} (~83 GPa) [18] are the moduli of CNT/interphase, CNT/matrix, bulk matrix and CNTs, respectively. The modification factor η is calculated based on the constituent modulus. The orientation factor α is 1/6 [19] to account for the randomness of the CNT network in 3D. The length (L) of nanotubes is 1900 nm using the Kuhn model (Eq. 6) where c is corona length (831 nm) and k is the Kuhn length (42 nm) with average diameter (d) of 10 nm measured through SEM micrographs. The CNT mass loading relative to interphase volume and matrix volume ($W_{CNT(i,m)}$) can be calculated (Eq. 7) by taking the effective CNT loading on CF (2.9 wt.%) and mass of CF per unit length (m_{CF}), CF volume (v_{CF}), and interphase or matrix volume fraction ($V_{i,m}$) in relation to ρ_m, the matrix density. The volume fraction of CNTs (V_{CNT}) can be calculated (Eq. 5) with ρ_{CNT}, the CNT density of 1.63 g.cm^{-3}. For a CNT-grafted-CF reinforced composite with V_f of 40%, the averaged relative improvement compared to the bulk matrix modulus of an unsized CF composite, is only around 7%; therefore, the improvement in overall fibre composite axial modulus, is negligible, once the rule of mixtures is applied (Eq. 9). However, more locally, the interphase modulus increases by 27% which should support the primary fibres against shear instability by directly improving the shear modulus (G_m); the improvement in shear modulus can be estimated (Eq. 10) using Poisson ratio of matrix , u_m (0.35).

$$E_{c(i,m)} = \frac{1 + \left(\frac{2L}{d}\right)\eta V_{CNT(i,m)}}{1 - \eta V_{CNT(i,m)}} E_m \tag{3}$$

$$\eta = \frac{\frac{1}{6}\left(\frac{E_{CNT}}{E_m}\right) - 1}{\frac{1}{6}\left(\frac{E_{CNT}}{E_m}\right) + \frac{2L}{d}} \tag{4}$$

$$V_{CNT(i,m)} = \frac{\frac{W_{CNT(i,m)}}{\rho_{CNT}}}{\frac{W_{CNT(i,m)}}{\rho_{CNT}} + \frac{1 - W_{CNT(i,m)}}{\rho_m}} \tag{5}$$

$$L = k\left(\frac{c/2}{k}\right)^{\frac{5}{3}} \tag{6}$$

$$W_{CNT(i,m)} = \frac{m_{CF} \times (wt.\%CNT_{CF})}{\left(\frac{v_{CF}(V_{(i,m)})}{V_f}\right)\rho_m} \quad (7) \qquad V_i = V_f \frac{A_i}{A_f} \quad (8)$$

$$E_c = E_f V_f + E_m(1 - V_f) \quad (9) \qquad G_m = \frac{E_m}{2(1 + u_m)} \quad (10)$$

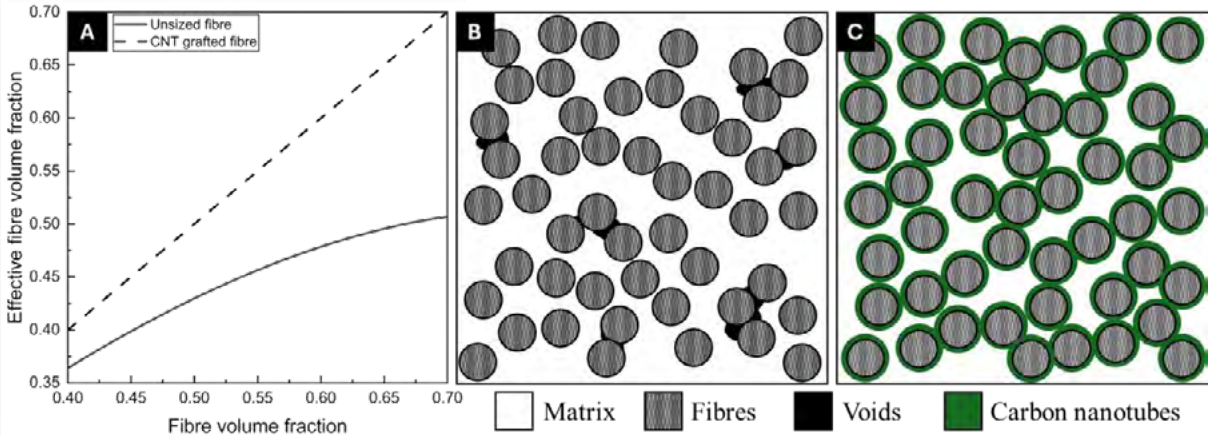

Figure 2. (A) Effective fibre volume fraction vs. fibre volume fraction prediction using equation 2 with n=0.75 for unsized fibres and n=0 for CNT-grafted fibres. (B) Cross-section of the composite model showing fibre-fibre surface contact. (C) Cross-section of the composite model of CNT-grafted fibres, eliminating fibre-fibre surface contact.

4. Conclusions

Through precise control and optimization of CVD parameters, uniform CNT growth across the 12K CF tow was achieved, as evidenced by rigorous SEM analysis. The CNT network corona length was controlled between 363 and 831 nm by adjusting the pulling speed, while maintaining a consistent diameter of 10 nm. This optimization resulted in a maximum measured CNT loading of 2.9 wt.% and corresponding specific surface area of 5.35 $m^2.g^{-1}$, consistent with the observed CNT lengths, diameters, and a uniform coverage.

The incorporation of CNTs onto the CF surface may prevent fibre contacts, improve IFSS, and reinforce fibre-matrix interphase. Improvements in longitudinal stiffness and tension strength mainly related to increase in effective fibre volume fraction, can be project at up to 10% at 40% V_f. Enhancement in compression strength may be even greater due to increase in shear modulus at the interphase, which was estimated to be 27%, predicted by the modified Halpin-Tsai. Ongoing experimental work is focused on tensile and compressive strength testing of manufactured unidirectional CFRP rods. These tests aim to provide a comprehensive understanding of the mechanical behaviour of CNT-grafted-CF reinforced composites. Future work will involve detailed characterisation of the interphase morphology and local matrix properties using techniques such as nanoindentation and atomic force microscopy. These analyses will validate the Helpin-Tsai model estimate.

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