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Edited by:

Prof. Christophe BINETRUY
ECCM21 Conference Chair
Institute of Civil Engineering and Mechanics (GeM)
Centrale Nantes
Nantes Université

Prof. Frédéric JACQUEMIN
ECCM21 Conference Co-Chair
Institute of Civil Engineering and Mechanics (GeM)
Nantes Université

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Editorial

Each volume gathers contributions on specific topics:

Vol 1. Industrial applications
Vol 2. Material science
Vol 3. Material and Structural Behavior – Simulation & Testing
Vol 4. Experimental techniques
Vol 5. Manufacturing
Vol 6. Multifunctional and smart composites
Vol 7. Life cycle performance
Vol 8. Special Sessions

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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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HIERARCHICAL NANO-STRUCTURED FIBRE INTERPHASE TO IMPROVE COMPRESSIVE PROPERTIES OF CARBON FIBRE COMPOSITES

Rupam Gogoi[1], David B. Anthony[2], Stephen J. Eichhorn[3] and Milo S.P. Shaffer[4]

[1]Department of Chemistry, Imperial College London, London, United Kingdom
rgogoi@imperial.ac.uk, linkedin.com/in/rupam-g-40771615

[2] Department of Chemistry, Imperial College London, London, UK
d.anthony08@imperial.ac.uk, https://www.imperial.ac.uk/people/d.anthony08

[3] Bristol Composites Institute, University of Bristol, Bristol, UK
s.j.eichhorn@bristol.ac.uk, https://research-information.bris.ac.uk/en/persons/steve-eichhorn

[4] Department of Chemistry and Department of Materials,
Imperial College London, London, UK
m.shaffer@imperial.ac.uk, https://www.imperial.ac.uk/people/m.shaffer

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Abstract
A nanostructured hierarchical coating inspired by the structure of the natural nacre was developed on individual fibres of an AS4 12k carbon fibre tow. A simple layer-by-layer (LbL) deposition method was adopted to coat layered double hydroxide (LDH) nanoparticles to mimic a brick-and-mortar-like architecture found in natural nacre. LDH particles of suitable size and surface charge were synthesized and characterized using X-ray diffraction, dynamic light scattering, and transmission electron microscopy. The quality of the nanoparticles was initially observed using a monolayer of LDH particles coated on a glass slide. This coating displayed a homogeneous monolayer of LDH particles with no significant particle agglomeration and uncoated spots. Following the same coating protocol, 15 layers of LDH particles were coated using LbL deposition on a surface-treated unsized AS4 12k carbon fibre tow and characterized using scanning electron microscopy. The initial findings indicate the formation of a well-structured LDH multi-layer coating. The main objective of preparing such coated fibres is to inhibit or delay micro buckling under compression when used as reinforcement in a polymer composite. In forthcoming studies, these coated fibre tows will be transformed into unidirectional epoxy-matrix composite rods and subjected to compression testing.

1. Introduction

The compressive strength of carbon fibre-reinforced polymer (CFRP) composites is reported to be almost half their tensile strength [1]. The typical failure mode in CFRP composites is kink band formation which occurs due to the initiation of fibre micro-buckling and is known as the primary cause of low compressive strength [1]. In this study, a natural nacre-inspired hierarchical nano-structured fibre interphase, in the form of a coating on carbon fibre (CF), is created to inhibit or delay the fibre micro-buckling when incorporated into a composite architecture. Figure 1 illustrates a nacre-inspired coating on fibre. Natural nacre is an excellent example of a stiff material with a toughening mechanism that allows crack deflection and propagation within its brick-and-mortar-like internal structure before final failure [2-3]. The hypothesis that nacre-like fibre interphases, containing a high concentration of stiff particles, will provide local shear stiffness to resist the shear instability that leads to kink band formation.

Previous nanoindentation measurements indicate that the nano-nacre composites have a transverse modulus of ~65 GPa, more than ten times that of the traditional thermoset composite matrices [4]. In addition, under compression there will be multiple crack deflections in the layered interphase, which will provide toughening with a strain-hardening response, allowing progressive fibre debonding/slippage and local stress relaxation. This interphase should therefore delay the micro-buckling initiation whilst avoiding debonding. In addition, it may help to isolate compression-induced fibre shear failures. The toughening mechanisms of nacre-inspired fibre interphases have been previously demonstrated for unidirectional (UD) composites under tension [5]. A similar approach is adopted in this study to create the fibre interphase. Exploiting the benefits of nacre-inspired coatings demands the preparation of a well-ordered layered structures with a similar phase proportion (95:5) and particle aspect ratio ~10 to that of natural nacre. To mimic a nacre-like architecture on the surface of CFs having a diameter of ~7 µm, it was necessary to scale down the brick-and-mortar structure by more than one order of magnitude in comparison to the size of the natural-nacre.

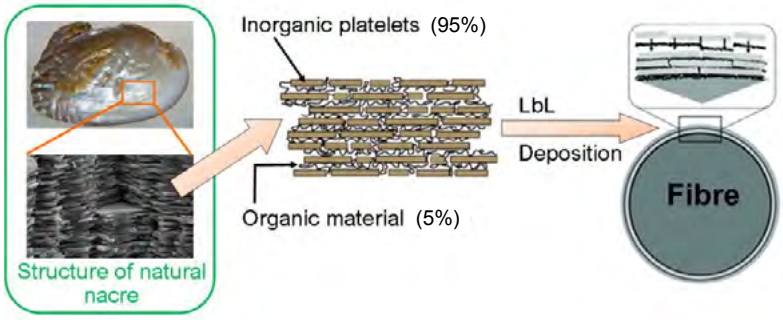

Figure 1. Illustrations depicting nacre-inspired coatings. On the left, the intricate internal arrangement of natural nacre is shown. In the centre, a representation of the nacre comprised of inorganic nanoplatelets linked by polymeric interlayers in 95:5 ratio is displayed. On the right, a single fibre model coated with numerous layers of synthesized LDH nanoplatelets scaled down to have a conformal coating on CF via LbL deposition, mirroring the structure found in natural nacre, is presented. {Adapted from [6]}.

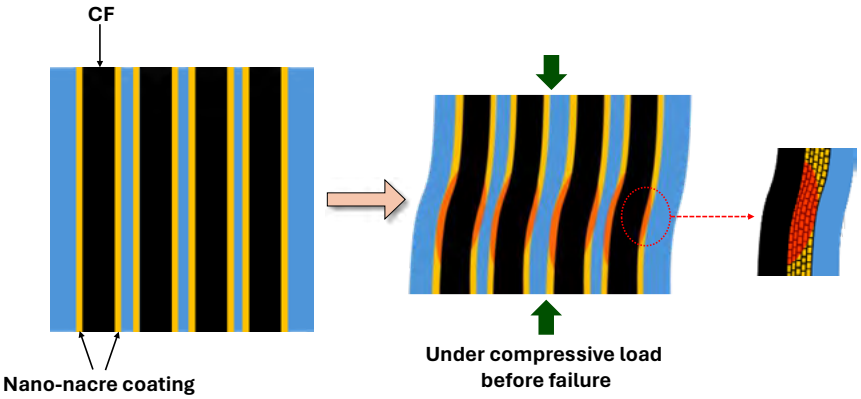

Figure 2. Schematic demonstrating micro buckling under compression in a UD composite reinforced with nano-nacre coated CFs. It is anticipated that the stiff nano-nacre coating will provide resistance to micro buckling at the localized region of interest, marked in red here. In addition, crack deflection and propagation will occur through the nacre-like interphase around the fibre with progressive compressive loading, allowing energy absorption. With all these mechanisms occurring, it is expected to have a delayed ultimate failure under compressive load and thereby improved strain-to-failure for composites with such nacre-like coating on individual fibres.

Layer-by-layer (LbL) coating of nanoparticles based on electrostatic interaction is a simple coating technique that can be used to make the nacre-like architecture. For a close-packed and well-ordered coating on CF using LbL, the chosen nanoparticle must have regular geometry (preferably hexagonal) with a high surface charge density. Here, LDH particles were chosen due to their tuneable size and the stability of the solution after synthesis. Alternate layers of positively charged LDH particles and negatively charged poly(sodium-4-styrenesulfonate) (PSS) were coated on unsized AS4 12k CFs using LbL to create a multi-layered coating/interphase. PSS acts as the interlayer polymer holding the LDH particles within the coating and performs a similar role as the organic phase found in the natural nacre architecture. The next step in this study is to prepare UD composites using the coated fibres and test them under compression. New test methods are being developed within the Next Generation Fibre-Reinforced Composites project [7] to evaluate composites at the tow level, which will be suitable for samples that consist of nanostructured interphase-modified fibres shown here.

2. Materials and methods

2.1. Materials

For the synthesis of LDH particles, magnesium nitrate hexahydrate, $Mg(NO_3)_2.6H_2O$, aluminium nitrate nonahydrate, $Al(NO_3)_3.9H_2O$, and sodium hydroxide, NaOH, was purchased from Sigma Aldrich. Poly(sodium 4-styrenesulfonate) (PSS, M_w 70,000, 30 wt.% in H_2O) and PDDA poly(diallyldimethylammonium chloride) (PDDA, M_w 100,000-200,000, 20 wt.% in H_2O) for the LbL deposition were also procured from Sigma Aldrich. An unsized AS4 12k CF tow, comprising individual fibres of diameter ~7 µm, was supplied by Hexcel Corporation.

2.2. LDH synthesis

LDH particles were synthesised by initially mixing 10 ml of 0.15 M $Mg(NO_3)_2.6H_2O$ and 0.05 M of $Al(NO_3)_3.9H_2O$ in 15 ml of 0.4 M NaOH vigorously for 10 min at room temperature [8]. The mix was centrifuged at 20,000 *g* for 15 min to collect the slurry produced during the co-precipitation. The supernatant was replaced with deionised water and the solution was bath sonicated for 5 min. In this way, the slurry was washed twice and dispersed in deionized water. Subsequently, the slurry was redispersed in 40 ml of deionised water by bath sonication and placed in an autoclave for hydrothermal treatment at 100 °C for 32 h.

2.3. Layer-by-layer deposition method

The LbL method based on the electrostatic charge difference has been used for coating the LDH particles on glass slides and CF. The method, as shown in Figure 3, consists of sequential steps of dipping a substrate (glass slide or CF) in different solutions containing opposite charges.

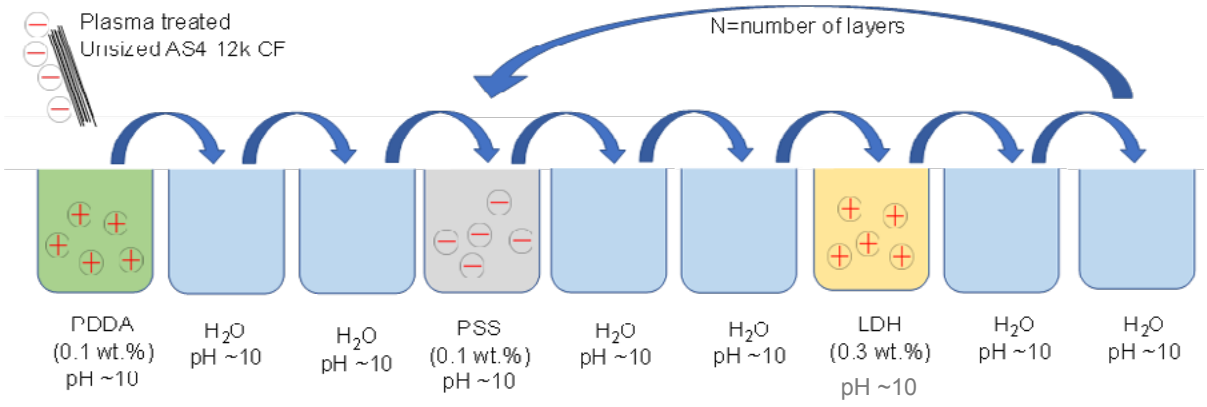

Figure 3. Schematic of the LbL method to coat LDH particles on a glass or CF substrate. {Adapted from [6]}.

The substrate is dipped into the PDDA solution for 10 min followed by 2 min of washing. The washing was carried out in two separate H_2O baths with 2 dips of 30 s in each bath. Subsequently, the substrate was dipped in PSS and LDH solutions for 10 min with 2 min of washing after each solution to obtain a monolayer. For multilayer coatings of LDH, the process was repeated for the desired number of layers as shown in Figure 3. PDDA and PSS aqueous solutions of 0.1 wt.% were used as the polycationic and polyanionic solutions. The first and the only layer of positively charged PDDA on the substrate acts as the buffer layer and avoids the solid-to-solid contact between LDH particles and the substrate (both positively charged). Whereas the negatively charged PSS acts as the soft matrix to glue the LDH particles together. The pH of all the solutions were maintained at ~10 using 0.1 M NaOH. The surfaces of the glass slides and CFs were both treated to improve the surface activity before coating. The glass slides were cleaned in a base bath containing 0.15 M KOH, followed by rinsing in deionized water. The AS4 12k CFs were plasma treated and subsequently dipped in 0.1 M $KMnO_4$ to increase the surface charge density of the fibres [5].

3. Results and discussion

3.1. LDH particle characterizations

The synthesized LDH particle solution had a pH of ~10 with a milky appearance. TEM analysis revealed a platelet-like 2D structure of LDH particles as shown in Figures 4 (a) and 4 (b) at low and high magnifications, respectively. The average zeta potential of the synthesised LDH solution was ~+35 mV as shown in Figure 4 (c), confirming a stable dispersion of the solution suitable for the LbL process. The width of the LDH particles was in the range from ~50 to ~300 nm, Figure 4 (d), with an average particle size of ~130 nm. The synthesis of phase pure LDH particles [3] was confirmed by the XRD diffractogram presented in Figure 4 (e). The thickness of the LDH particles was estimated using the Scherrer equation (Eq. 1) for the characteristic (003) XRD peak at 2θ = 11.48 º, Figure 4 (f).

$$t = \frac{k.\lambda}{\beta.cos\theta} \tag{1}$$

In Eq. 1, t represents the mean thickness of LDH platelets, k stands for a dimensionless shape factor (0.89), λ denotes the X-ray beam wavelength (1.5418 Å), β represents the full width at half maximum (FWHM) of the diffraction peak, and θ signifies the Bragg angle. Employing Eq. 1, the determined average platelet thickness is ~15 nm. Consequently, the aspect ratio of the synthesised LDH platelets is ~9. The LDH particle synthesized here falls in the range of suitable particle size required for a conformal coating on AS4 CF as reported in a previous study [5].

3.2. LDH coating quality

A monolayer of LDH coating on a glass slide was used to check the coating quality as displayed by the SEM images in Figure 5 at two different magnifications. At low magnification, Figure 5 (a), the coated glass slide was observed to have a homogenous coverage of LDH particles with no uncoated spots. At high magnification, Figure 5 (b), no significant agglomeration of the particles was observed, thus confirming a good quality of monolayer coating which can form the base of the consecutive layers in a multilayer coating system.

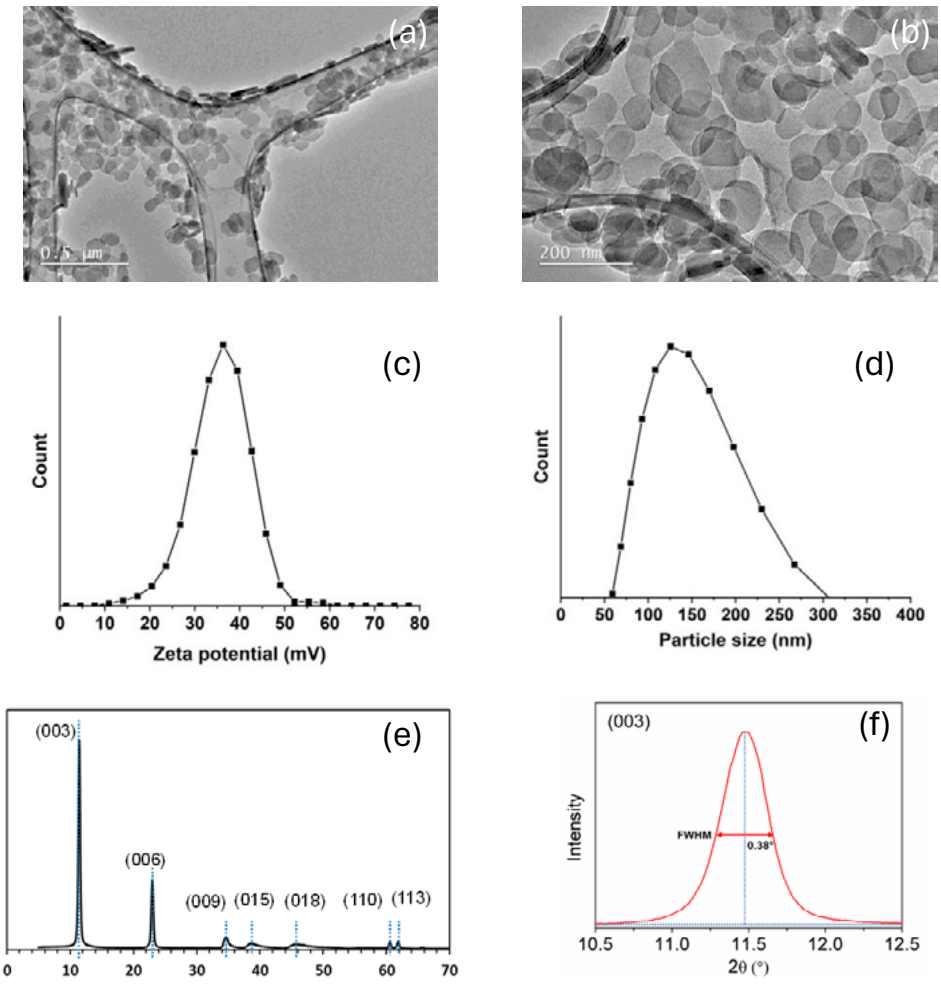

Figure 4. Illustration of various analyses employed to characterize LDH particles. (a) and (b) show TEM images of the particles at low and high magnifications, respectively. Particle size and zeta potential distribution curves obtained from dynamic light scattering are plotted in (c) and (d), respectively. XRD diffractogram to investigate the crystallographic structure of LDH is represented in (e) and the diffraction peak of the (003) plane is shown in (f) indicating the full width at half maximum (FWHM) used to calculate the particle thicknesses. {Adapted from [6]}.

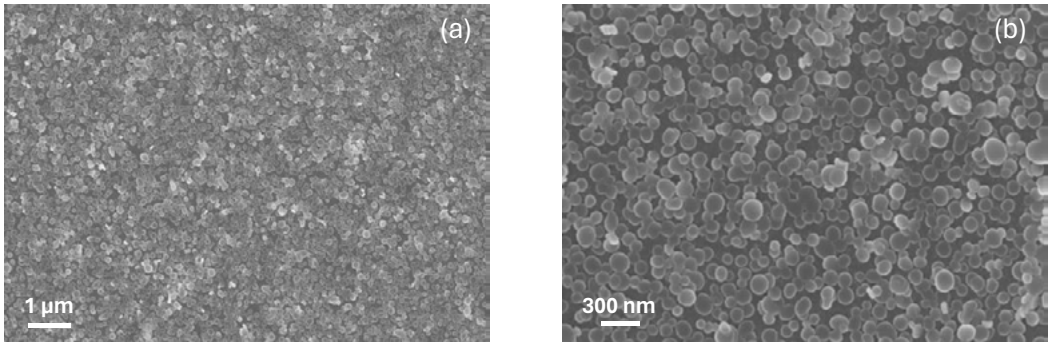

Figure 5. SEM images of a monolayer LDH coating on a glass slide at (a) low and (b) high magnifications.

3.3. Multilayer LDH coating on carbon fibre

An AS4 12k CF tow with 15 layers of LDH coating on individual fibres was obtained using the LbL method mentioned in Section 2.3. The coated fibre tow, examined under SEM at different locations, revealed uniform LDH coverage demonstrating a good coating quality with an example displayed in Figure 6 (a). At high magnifications, Figure 6 (b), the LDH particles can be seen to form a closely packed coating structure on the fibre surface. The self-limiting nature of the coating method and selection of suitable particle size facilitated the formation of this well-ordered layered structure with uniform coverage among individual fibres within the 12k tow. To check the multilayer coating coverage along the circumference of the individual fibres, the cross section of the coated fibres was examined by simply cutting the fibres with a scalpel and analysing under SEM. A uniform thin layer of coating running along the circumference of the fibre can be observed in Figure 7 (a). With a closer look at the coating, the formation of a well-ordered nacre-like layered architecture was revealed within the coating structure, Figure 7 (b).

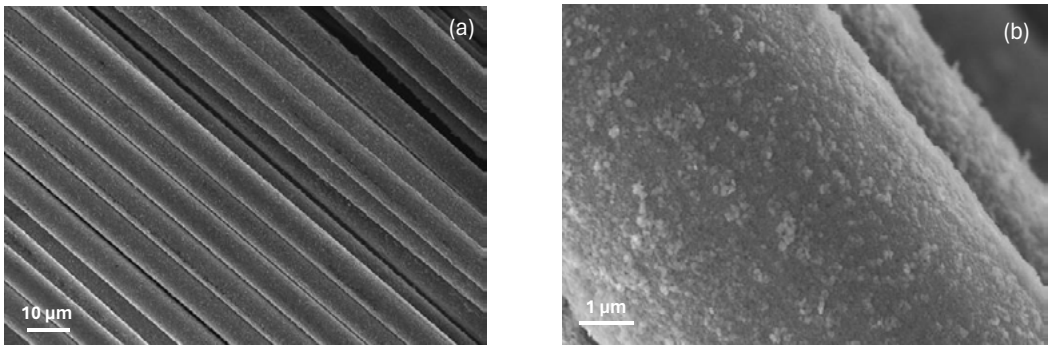

Figure 6. SEM image of (a) CF bundle having 15 layers of LDH coating on individual fibres and (b) zoomed image of a single CF within the fibre bundle.

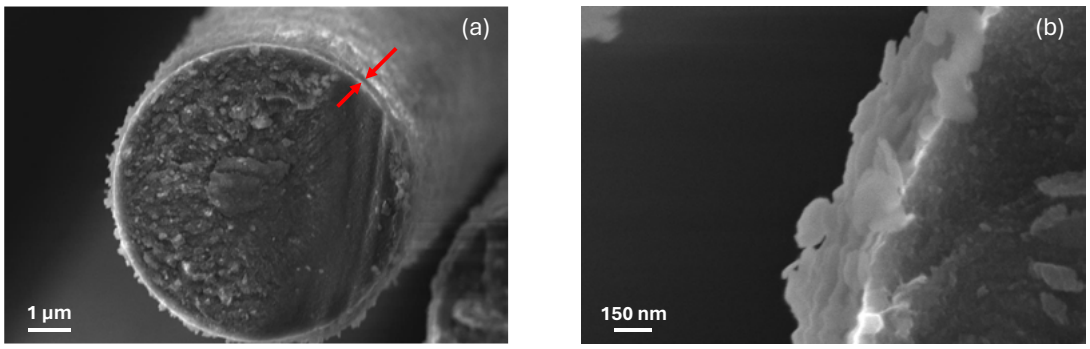

Figure 7. Cross-section SEM images of a single CF coated with 15 layers of LDH particles at (a) low and (b) high magnifications. The red arrows in (a) indicate the coated layer.

4. Conclusions and discussion

This study successfully developed a hierarchical nanostructured coating inspired by nacre for CFs. A stable aqueous suspension of LDH containing particles of suitable size was synthesized to coat glass slides and CFs. The monolayer LDH coating on the glass slides exhibited good quality with no particle agglomerations and uncoated spots. Subsequently, a 15-layer LDH coating was developed on a section of a long CF bundle (12k) using the LbL method. The morphological analysis confirmed a uniform coating on individual fibres throughout the bundle. A cross-sectional examination of the coated fibres verified a consistent coating layer around the fibre circumference. In future studies, these coated fibres will be utilized to fabricate unidirectional tow composite rods and subjected to innovative compression testing methods developed as part of the Next Generation Fibre-Reinforced Composites programme [7].

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