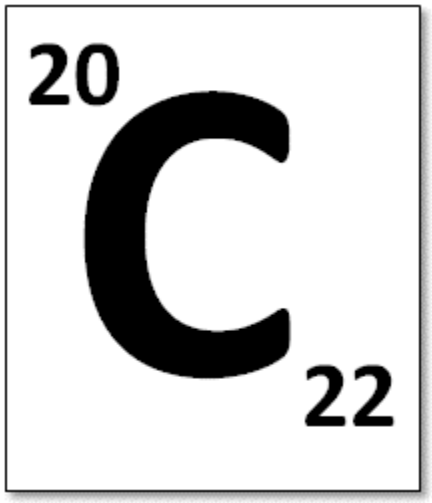


Carbon 2022

The World Conference on Carbon
'Carbon for a Cleaner Future'

3rd July – 8th July 2022
at
Imperial College London
UNITED KINGDOM

Extended Abstract Book

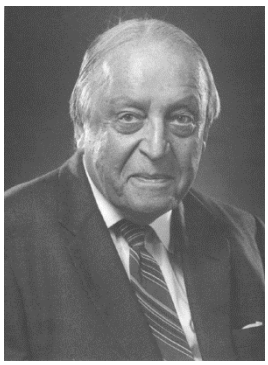
www.carbon2022.org

organised under the auspices of
The British Carbon Group

The
British Carbon Group

Carbon 2022 is organised by the British Carbon Group. The British Carbon Group is a special interest group of the Royal Society of Chemistry, the Institute of Physics and the Society of Chemical Industry devoted to the advancement of carbon science. It is also a member of the European Carbon Association.

Preface

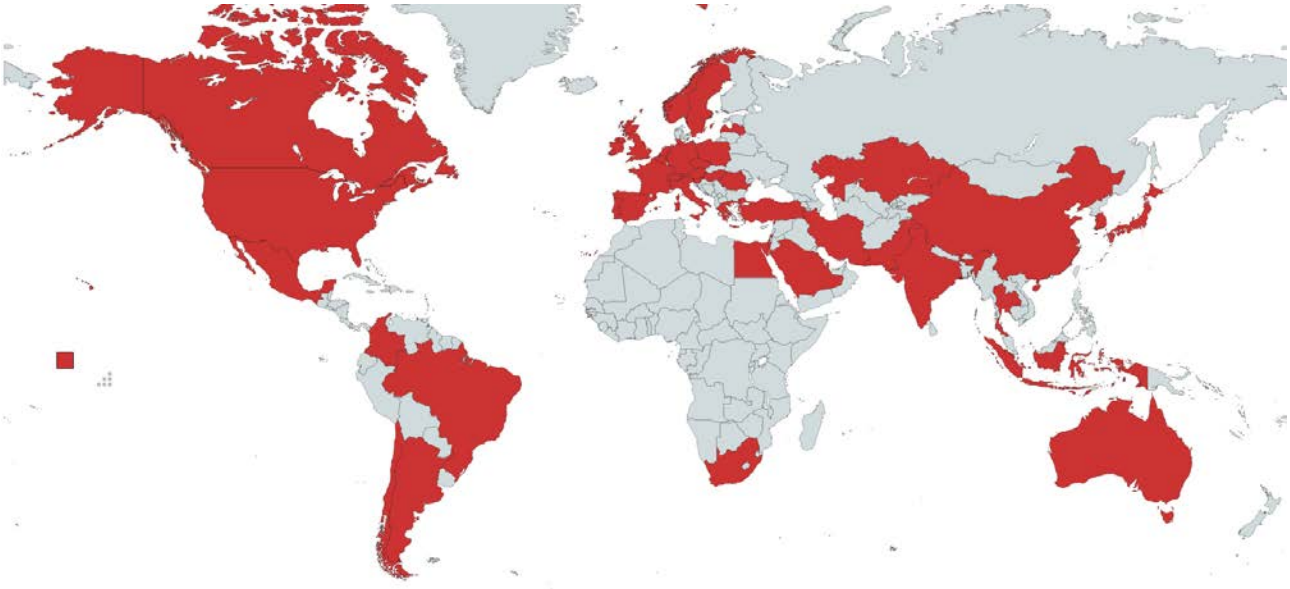


After a 2-year absence, the British Carbon Group was delighted to organise the 21st edition of the World Conference on Carbon at Imperial College London. Since the first World Conference on Carbon in 2000, scientists from around the globe have met annually to discuss and celebrate advancements in the field of carbon science - together in-person - and we continued that tradition in 2022. It was especially appropriate that we met at Imperial College London. This was the institution where Professor A. R Ubbelohde established his Carbon Research Laboratory and organised the First UK Industrial Carbon and Graphite Conference (one of the precursors to the World Conference on Carbon), held in 1957 in London – 65 years ago.

Alfred R. J. P. Ubbelohde

The United Kingdom has a long history of carbon science starting with the 'discovery' of plumbago (natural graphite) in Cumbria in 1564 during the reign of Elizabeth I – the word graphite didn't really exist until several centuries later when proposed by Werner and Harsten in 1879. Carbon science in the UK has a long tradition associated with the rich coal fields found in the North of England and through this led to many achievements including Rosalind Franklin's very influential work on graphitizing and non-graphitizing carbons. Certainly, the familiarity with industrial carbon materials influenced the UK's decision to focus on graphite-moderated civil nuclear power since the 1940s. This era of the Advanced Gas-cooled fleet of nuclear electricity generation is coming to an end which creates new challenges. Indeed, the UK has had a long association with industrial carbons, from major production of anode carbons in Sheffield until the 1990s, to modern composites technology especially in sports such as Formula One. Later the work by Sir Harry Kroto and colleagues on Buckminsterfullerene led to the 1996 Nobel Prize, with a second Nobel prize in 2010 for Sir Andre Geim and Sir Konstantin Novoselov, for their work on graphene. Carbon science has a rich and influential history, but this conference showed it has a bright future too.

We had over 300 registered attendees with 45 countries represented, and over 200 talks and 150 posters presented. This extended abstract book represents a summary of some of the excellent work seen over the week. We look forward to the discussions continuing in 2023 in Mexico!



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- Hiden
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- Ingevity
- Institute of Physics
- MDPI C – Journal of Carbon research
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C2022 Summary Programme

	Morning		Afternoon		Evening
	09:00 - 10:10	10:40 - 13:00	14:00 - 15:00	15:30 - 17:30	
Sunday 3rd July					
Queens Tower Rooms				Registration, Exhibition Set-Up	Welcome Reception (16:00-18:30)
Monday 4th July					
Session		1	2	3	
A Great Hall	08:30 Opening Ceremony, Plenary & Brian Kelly Award	Carbon in Energy	Nanoscale Carbons	Carbon in Energy	SCI Drinks Reception at Belgrave Square (19:00)
B Pippard		Graphite	Innovative Carbons for Sustainability	Carbon in Biology, Medicine & Health	
C Read		Physicochemical Carbon Properties	Environmental Carbon	Heterocarbons	
QT Rooms		Set-up Posters (Session 1)			
Tuesday 5th July					
Session		4	5	6	
A Great Hall	Plenary & French Carbon Group Awards	Carbon in Energy	Carbon in Energy	Elsevier Carbon Journal Workshop	
B Pippard		Nanoscale Carbons *(sponsored by C Journal)*	Carbon in Biology, Medicine & Health *(sponsored by C Journal)*		
C Read		Nuclear Graphite *(sponsored by NNL Ltd)*	Carbon Fibres & Composites	Nanoscale Carbons	
QT Rooms				Poster Session 1	
Wednesday 6th July					
Session		7	8	9	
A Great Hall	Plenary & Attonuclei Prize	Carbon in Energy	Carbon in Energy	Carbon in Energy	
B Pippard		Carbon Fibres & Composites	Graphite	Amorphous Carbons	
C Read		Environmental Carbon	Nanoscale Carbons	Nanoscale Carbons	
QT Rooms		Set-up Posters (Session 2)			
Thursday 7th July					
Session		10	11	12	
A Great Hall	Plenary & ECA Award	Carbon in Energy	French Carbon Group Awards (SFEC2020-22)	Miscellaneous Carbons	Conference Banquet at Butler's Wharf (19:15 for 19:30 departure)
B Pippard		Innovative Carbons for Sustainability	Innovative Carbons for Sustainability		
C Read		Environmental Carbon	Environmental Carbon		
QT Rooms				Poster Session 2	
Friday 8th July					
Session		13	14	15	
A Great Hall	Plenary (Presentation C2023)	Carbon in Energy	Carbon in Energy	Carbon in Energy	
B Pippard		Innovative Carbons for Sustainability	Physicochemical Carbon Properties	Nanoscale Carbons	
C Read		Environmental Carbon	Nanoscale Carbons	Carbon Fibres & Composites	

Abstracts are listed alphabetically by presenting author.

Carbon 2022

The effect of nacre-inspired coating on the tensile properties of carbon fibre composite

Rupam Gogoi[a#], Francois De Luca[b], David B. Anthony[a,c], and Milo Shaffer[a,d*]
a: Department of Chemistry, Imperial College London, UK
b: Department of Chemical Engineering, Imperial College London, UK
c: Department of Aeronautics, Imperial College London, UK
d: Department of Materials, Imperial College London, UK
Presenting author, E-mail: rgogoi@ic.ac.uk
* Corresponding author, E-mail: m.shaffer@imperial.ac.uk

Keywords
Nanostructured coating; Bio-mimicking; Fibre-matrix interphase

INTRODUCTION

Carbon fibre (CF) has shown tremendous potential as an alternative material for the construction of lightweight and high-strength composites commonly known as CF reinforced polymer (CFRP). Yet, there is a concerning design limitation in such material which arises from its poor toughness leading to a catastrophic failure. Although, energy absorption in composite materials is manifested through various toughening mechanism such as fibre-matrix debonding and friction post debonding, fibre pull-out, stress relief at the interphase etc., yet it is brittle in nature with very low strain to failure.
In addition to low toughness, CFRP do not reflect the true inherent strength of the CF, implying limited exploitation of the reinforcing fibre. The strength of a composite material depends on the effective load transfer from the matrix to the fibre under the application of load. The effective load transfer is therefore dependent on the interphase and the fibre-matrix interfacial adhesion. From previous studies, it has been observed that surface modifications of CF have the potential in improving the fibre-matrix interfacial adhesion, and hence, bulk mechanical properties of composites under tensile and flexural loads (Sharma, 2014). However, increasing the interfacial strength improves composite strength and stiffness it reduces the toughness. To tackle both these drawbacks, an optimized nanostructured interphase, inspired by natural nacre was developed in the current study to increase the toughness by alleviating local stress concentration, whilst maintaining effective load transfer (De Luca, 2018). The nanostructured interphase in the form of a coating on CF was produced by layer-by-layer (LbL) deposition of hexagonal layered double hydroxide (LDH) platelets as the inorganic phase and soft poly (sodium 4-styrene sulfonate) (PSS) polyelectrolyte as the organic phase, thus mimicking a brick-and-mortar nanostructure analogues to the natural nacre system. The hypothesis is that an ordered multilayer structure can absorb the energy released by fiber breaks, via multiple crack deflections in the layered interphase structure, spreading along the length of the fibers; progressive fiber debonding/slippage, mediated by strain hardening of the interphase layer in shear, can then allow local stress relaxation without excessive debonding. Together, these effects may potentially delay the correlation of fiber breaks in a composite and hence increase its ultimate strength and exhibit a pseudo-ductile failure.

RESULTS AND DISCUSSION

The LbL process is based on electrostatic forces and requires the substrate to have a high surface charge density. For successful deposition and adequate adhesion, the surface charge density of the as revied industrially oxidized, unsized CF was increased by oxidation using O_2 plasma treatment initially and subsequent immersion in 0.1 M $KMnO_4$ (Fig. 1A). At high pH, the oxidises obtained on the CF surface dissociate, resulting in an increased negative surface charge density. The measured

ζ-potentials increased in magnitude from −19 mV for as received CF to −58 mV observed for the oxidised CF. To have a high electrostatic interaction among fibre surface and coating the pH was maintained at 10 for all the solutions and suspensions in LbL deposition, where the ζ-potential was most negative.

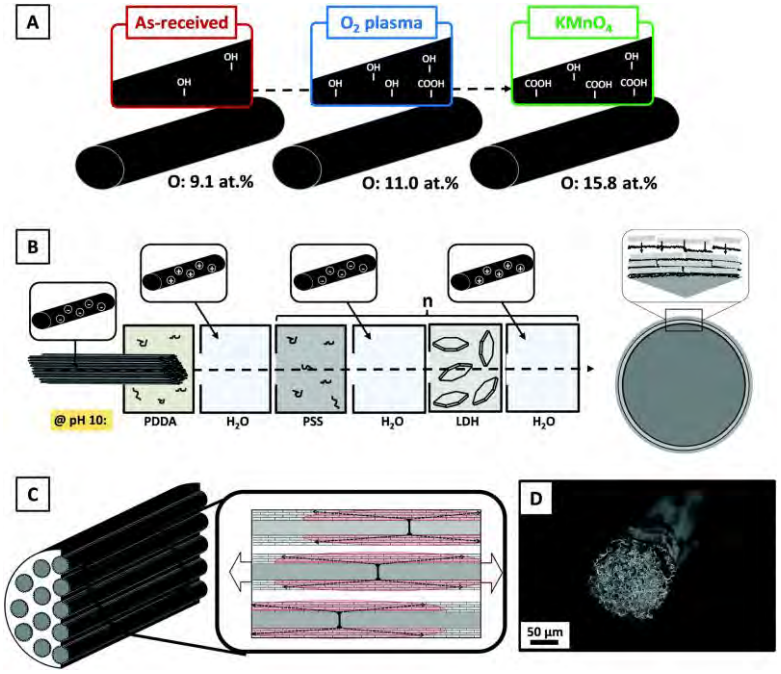

Fig. 1. Schematic diagram showing the modification of the as-received unsized carbon fibers, coating deposition and small composite manufacture: the as-received carbon fiber surfaces were functionalized with low-pressure O_2 plasma treatment and subsequently further oxidized in KMnO4 (A). PDDA(PSS/LDH)n "brick-and-mortar" nanostructures were assembled, by LbL at pH 10, on all the individual carbon fibers within a bundle (B). The composite containing coated carbon fibers was intended to fail via crack deflection (in red) and sliding within the volume of the anisotropic nanostructured interphase (C). SEM micrograph of a composite cross-section made of coated carbon fibers (D).

Coating of CF was done by sequential LbL dipping process, as illustrated in Fig. 1B. A soft, cationic poly (diallyl dimethyl ammonium chloride) (PDDA) precursor layer was found to promote the adhesion of the coating to the fiber, avoiding direct contact between the stiff, positively charged platelets and rigid carbon fiber surfaces. The negatively charged fiber surfaces were first coated with a positive PDDA buffer layer, before repeatedly applying (PSS/LDH) bilayers.

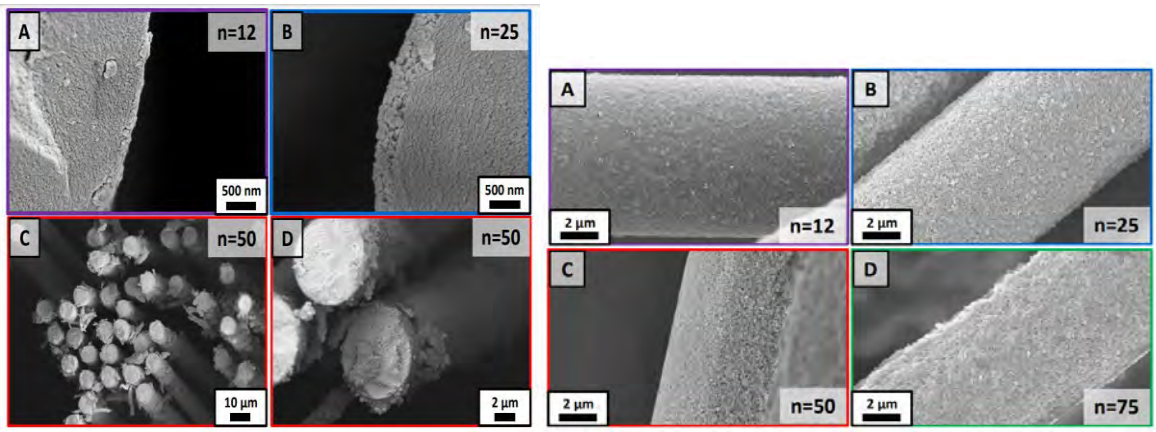

Fig. 2: Cross-section (left image) and surface (right image) of nanostructured coatings deposited on a bundle of unsized CF. SEM micrographs of carbon fibers coated with PDDA/$(PSS/LDH)_n$ coating. n=12 (A), n=25 (B) and n=50 (C and D).

SEM in Fig. 2 showed that the PDDA/(PSS/LDH)$_n$ coatings were homogeneous across all the fibers within the bundles. The self-limiting nature of the LbL process means that each individual fiber within the bundle can be coated simultaneously, accelerating production and allowing small unidirectional model composites, consisting of several hundred coated carbon fibers in epoxy to be manufactured (Fig. 1C and D).

The apparent interfacial shear strength (IFSS) and ability of the fiber to progressively slide during the debonding process were investigated from single-fibre pull-out (SFPO) test (Fig. 3A and B). Stable fiber slippage was quantified by defining the debonding length ratio (DLR) as the ratio between the distance that the fiber slides prior to full debonding (l_d) over the fiber length embedded in epoxy (l_e). The IFSS was determined from a plot of maximum force against embedded area (Fig. 3A). Both IFSS and DLR were significantly improved by the presence of the nanostructured coating, relative to the control fibers, reaching maxima of 59.5 ± 3.9 MPa (+84%) and 0.17 ± 0.02 (+89%), respectively, for the fibers coated with a 0.4 µm thick PDDA/(PSS/LDH)$_{25}$ layer. Further increases in the interphase thickness resulted in a relative drop of the IFSS and DLR, most likely due to a reduction of the radial clamping force exerted on the fiber by the epoxy matrix, as a result of the compliance of the coating.

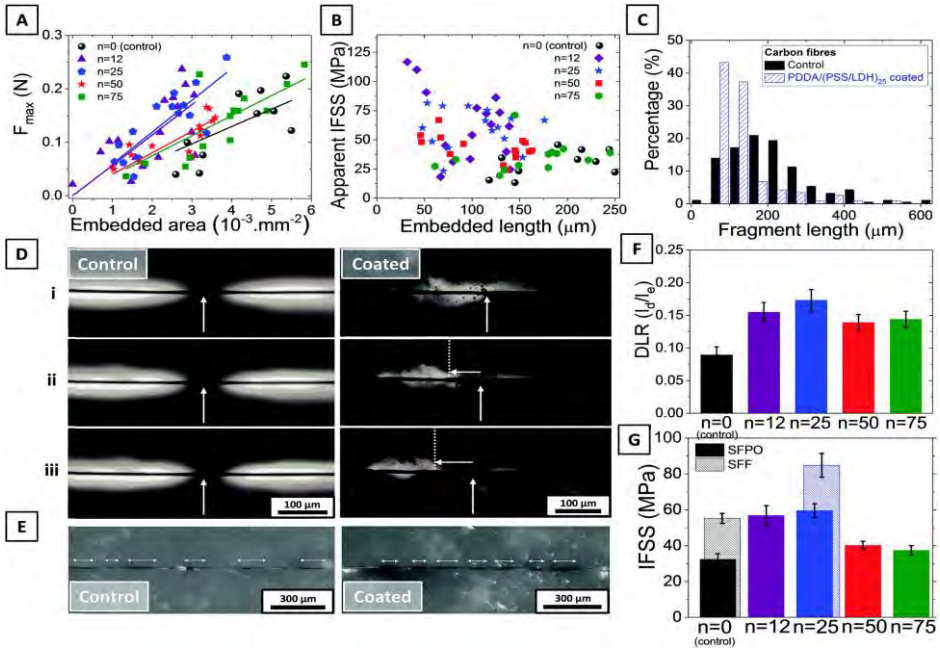

Fig. 3. Maximum force (F_{max}) applied to single fibers as a function of fiber embedded area in the epoxy matrix (A), apparent IFSS = $f(l_e)$ of control fibers and fibers coated with PDDA/(PSS/LDH)$_n$ of varying thickness (B) and histogram distribution of fiber fragment length for control and PDDA/(PSS/LDH)$_{25}$ coated fibers obtained by single fiber fragmentation tests (C). Optical images of in situ fragmentation of control and PDDA/(PSS/LDH)$_{25}$ coated (D) carbon fibers using cross-polarized light in transmission at +1.2% and +2.4% strain (i, ii and iii, respectively); vertical arrows pinpoint fiber fragments and horizontal arrows and the dashed line highlight progressive sliding of the fiber. Optical images of control and PDDA/(PSS/LDH)$_{25}$ coated carbon fibers using non-polarized light in transmission mode after the fragmentation test (E); horizontal arrows show fiber fragments. Debonding length ratio (DLR) and IFSS of carbon fibers coated with different PDDA/(PSS/LDH)$_n$ coating thicknesses determined by single fiber tests in epoxy (F and G, respectively).

Under single fiber fragmentation tests (SFFT), the control fibers exhibited an intense and large stress field near each fiber break, which remained relatively unchanged when strain increased (Fig. 3D(i–iii)); in contrast, the PDDA/(PSS/LDH)$_{25}$ coated fibers presented a less intense stress field, spread along the length of the fiber (Fig. 3C and D). Upon increasing the macroscopic strain, the stress field was observed to propagate progressively along the fiber, accompanied by a noticeable further reduction in intensity of the stress field. The weaker initial stress field can be attributed to crack

deflection within the nanostructured "brick-and-mortar" interphase, while the reduction in its intensity can be attributed to progressive slippage of the fiber, through plastic deformation of the interphase. IFSS values estimated using Kelly-Tyson model (Kelly, 1965), which were 55.2 ± 2.8 and 84.8 ± 6.6 MPa respectively for the control and PDDA/(PSS/LDH)$_{25}$ coated fibers.

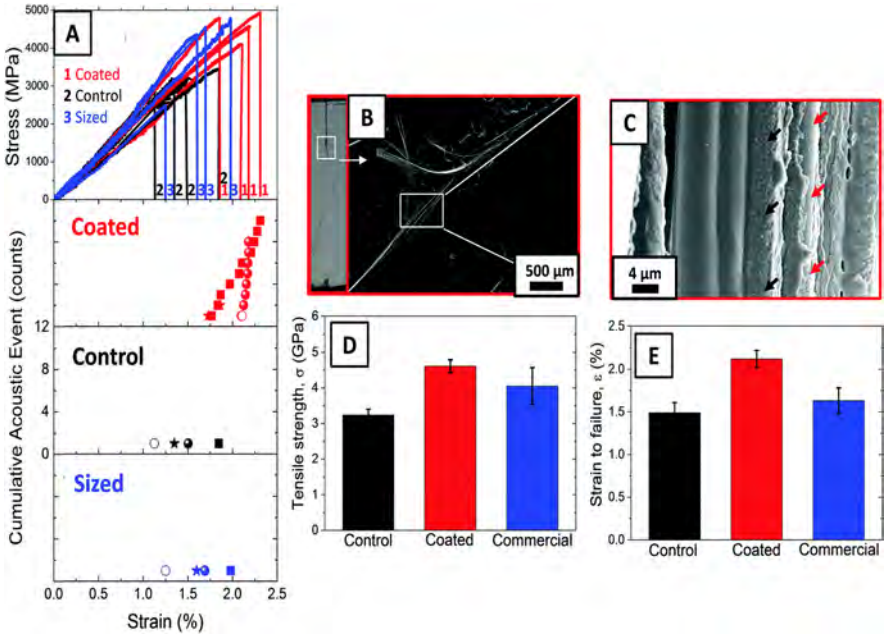

Fig. 4. Stress–strain curves and associated cumulative distributions of acoustic emission events occurring during tensile tests of impregnated fiber tow composites containing control fibers, coated fibers (with "brick-and-mortar" PDDA/(PSS/LDH)$_{25}$ nanostructured coating), and sized fibers (A). Low and high magnification SEM micrographs, accompanied by high-speed video stills, of fracture surfaces of impregnated fiber tow composites containing coated fibers (B and C, respectively) – red and white arrows pinpoint locations where the coating is removed and still adhered to the surface of the fibers, respectively. Tensile strength and strain-to-failure of the composites containing control, coated and sized fibers (D and E, respectively).

The tensile properties and fracture behavior of small unidirectional model composites were compared against control fibers, and the optimum PDDA/(PSS/LDH)$_{25}$ coated fibers, as well as additional samples prepared with as-received commercially sized PAN-based fibers (called "sized fibers" in the following). The tensile stress–strain responses of these tow composites were recorded and correlated with acoustic events (AEs), indicative of fiber breaks (Fig. 4A) occurring during the tensile test. In order to minimize any effect of varying fiber volume fractions and, therefore, allow for a fair comparison of the different composite specimens, the load applied to the material was converted into a stress normalized to the cross-sectional area of the total fiber content in the composite. This approach provides a clearer indication of the fiber dominated tensile properties, which determine the tensile properties of unidirectional tow composites. The elastic modulus was determined to be similar for all types of fiber composites, roughly 230 GPa; the similarity to the expected elastic modulus of AS4 carbon fibers (231 GPa) confirms the robustness and consistency of the composite preparation and testing. The tensile strength of the tow composites containing the nanostructured interphase increased from 3235 ± 160 MPa to 4607 ± 391 MPa, as compared to composites containing the control fibers (Fig. 4D). The increased tensile strength of the coated fibers was accompanied by an increased strain-to-failure from 1.49 ± 0.12% to 2.12% ± 0.10% (Fig. 4E). More importantly, the strength and strain to failure of the coated fiber composites also exceeded the values for the sized fibers (4048 ± 523 MPa and 1.63 ± 0.15%, respectively). Only a single AE event was detected for the composites based on control or sized fibers, which was associated with the final composite failure. However, for the composites containing coated fibers, additional AE

events were detected, prior to failure. This observation is consistent with a larger number of independent fiber breaks occurring in the hierarchical composites, due to a successful reduction in local stress concentrations in neighbouring fibers. The associated delay in the formation of a critical cluster of fiber breaks can explain the improved ultimate tensile strength and strain to failure of the composites.

CONCLUSIONS

An entirely new class of nanostructured fiber sizing was designed and successfully implemented. A 0.4 µm thick "brick-and-mortar" PDDA/$(PSS/LDH)_{25}$ nanostructured coating was found to offer the greatest improvements in IFSS and DLR, as determined by single fiber pull-out. In fragmentation tests, the local stresses associated with fiber fragmentation appeared reduced and diffused. When the optimum coating was used as the interphase in unidirectional impregnated fiber tow model composites, the new system provided higher ultimate strength and strain-to-failure than composites containing either bare unsized treated carbon fibers ("control fibers") or the as-received commercially sized carbon fibers ("sized fibers"). Acoustic emission recorded a higher number of fiber breaks within the hierarchical composites prior to the final catastrophic failure, indicative of the occurrence of multiple isolated fiber breaks. The nanostructured "brick-and-mortar" interphase appears to have isolated fiber breaks within the composite, delaying the formation of a critical cluster, leading to improved tensile properties compared to the composite containing commercial fibers.

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