



Pozegic, T., Anguita, J. V., Hamerton, I., Jayawardena, K. D. G. I., Chen, J-S., Stolojan, V., Ballochi, P., Walsh, R., & Silva, S. R. P. (2016). Multi-Functional Carbon Fibre Composites using Carbon Nanotubes as an Alternative to Polymer Sizing. *Scientific Reports*, 6, [37334]. <https://doi.org/10.1038/srep37334>

Publisher's PDF, also known as Version of record

License (if available):
CC BY

Link to published version (if available):
[10.1038/srep37334](https://doi.org/10.1038/srep37334)

[Link to publication record in Explore Bristol Research](#)
PDF-document

This is the final published version of the article (version of record). It first appeared online via Nature at <http://www.nature.com/articles/srep37334>. Please refer to any applicable terms of use of the publisher.

University of Bristol - Explore Bristol Research

General rights

This document is made available in accordance with publisher policies. Please cite only the published version using the reference above. Full terms of use are available: <http://www.bristol.ac.uk/red/research-policy/pure/user-guides/ebr-terms/>

Multi-Functional Carbon Fibre Composites using Carbon Nanotubes as an Alternative to Polymer Sizing

*Thomas R. Pozegic, José V. Anguita, Ian Hamerton, Imalka Jayawardena, Jeng-Shiung Chen, Vlad Stolojan, Paolo Balocchi, Robert Walsh and S. Ravi P. Silva*¹.*

T. R. Pozegic¹, J. V. Anguita¹, I. Hamerton², K. D. G. I. Jayawardena¹, J-S. Chen¹, V. Stolojan¹, P. Balocchi³, R. Walsh³ and S. R. P. Silva*¹.

¹ Advanced Technology Institute, University of Surrey, Guildford, Surrey, GU2 7XH, U.K.

² Advanced Composites Centre for Innovation and Science, Department of Aerospace Engineering, University of Bristol, Bristol, BS8 1TR, U.K.

³ Bombardier, Airport Road, Belfast BT3 9DZ, Northern Ireland, U.K.

Supplementary Notes

Supplementary Note 1 | Electron Microscopy

The fuzzy fibre and unmodified carbon fibre plies were analysed by scanning electron microscopy (SEM, Quanta 200 from FEI) in fabric form with masking tape adhered around the edges to aid handling and prevent airborne fibres, which could potentially enter the vacuum pumps. SEM was performed using an FEI Quanta 200 and was operated under high vacuum with a secondary electron detector employed. Typical voltages were between 2 - 20 kV, spot sizes of 2 - 4 nm with a working distance of 10 mm.

For STEM analysis, a fuzzy fibres were placed in a vial containing isopropyl alcohol (5 ml) and ultrasonicated for 10 minutes to be subsequently drop casted on a carbon grid for imaging.

Supplementary Note 2 | Cross-sectional Analysis

Once the polishing process (Supplementary Table 1) was completed on the 4-ply thick fuzzy fibre composite (F-CFRP), a 7.5 nm gold film was deposited using a DC magnetron sputtering system prior to SEM analysis.

Supplementary Note 3 | Ultrasound Testing

*¹ Corresponding author. Tel: 01483 68 9825. E-mail: s.silva@surrey.ac.uk

Ultrasound testing was performed using an Olympus Omniscan phased array ultrasonic testing instrument with a MX 5 MHz probe in a water filled immersion tank. The sample tested was a single fuzzy fibre ply consolidated into a composite with a stack of unmodified carbon fibre plies.

Supplementary Note 4 | Raman Spectroscopy

Raman spectroscopy on unmodified carbon fibre ply, interlayer and catalyst-deposited carbon fibre ply, and fuzzy fibre ply samples were performed using the 514 nm and 782 nm argon-ion laser lines, operated at a laser power of 17 mW, and using a Renishaw Raman spectroscope with a Leica DM LM microscope. Scans were between 0 and 3000 cm^{-1} with a duration of 30 s and repeated twice (3 iterations). A microscope with a magnification lens of 50x, provided a spatial resolution of $\sim 5 \mu\text{m}$ and data analysed as discussed in the literature ¹.

Supplementary Note 4 | Determination of Fibre Volume Fraction via Thermogravimetric Analysis

The method employed was a modified version by Yee *et al.*², where a variable temperature rate (see inset of Supplementary Figure 2), was employed under a nitrogen flow (60 ml/min). Initially, poly matrix samples were tested to confirm decomposition where any remaining mass was attributed to residue and refined the fibre volume fraction calculation. In addition, the carbon fibres were tested to confirm stability.

The weight percentage of the fibre (f_f) was modified to account for residue after the decomposition of the polymer matrix²:

$$f_f = \frac{1}{1 - Y} \left(\frac{W_r}{W_c} - Y \right) \quad \mathbf{1}$$

Where W_r is the weight of the polymer matrix, W_c is the weight of the composite and Y is the percentage residue from thermally decomposing the pure polymer matrix. The fibre volume fraction (V_f) was calculated using:

$$V_f = \frac{\left(\frac{W_f}{D_f} \right)}{\left(\frac{W_f}{D_f} \right) + \left(\frac{W_r}{D_f} \right)} \quad \mathbf{2}$$

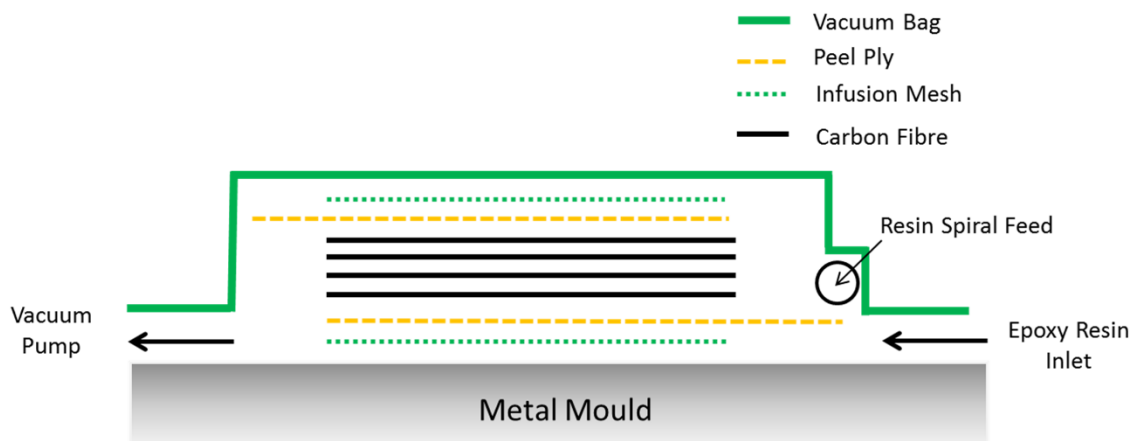
Where W_f is the weight of the carbon fibre; W_r is the weight of the polymer matrix, D_f is the density of the fibre and D_r is the density of the polymer matrix.

For the F-CFRP, the CNTs remain on the carbon fibres, hence are included in the fibre volume fraction:

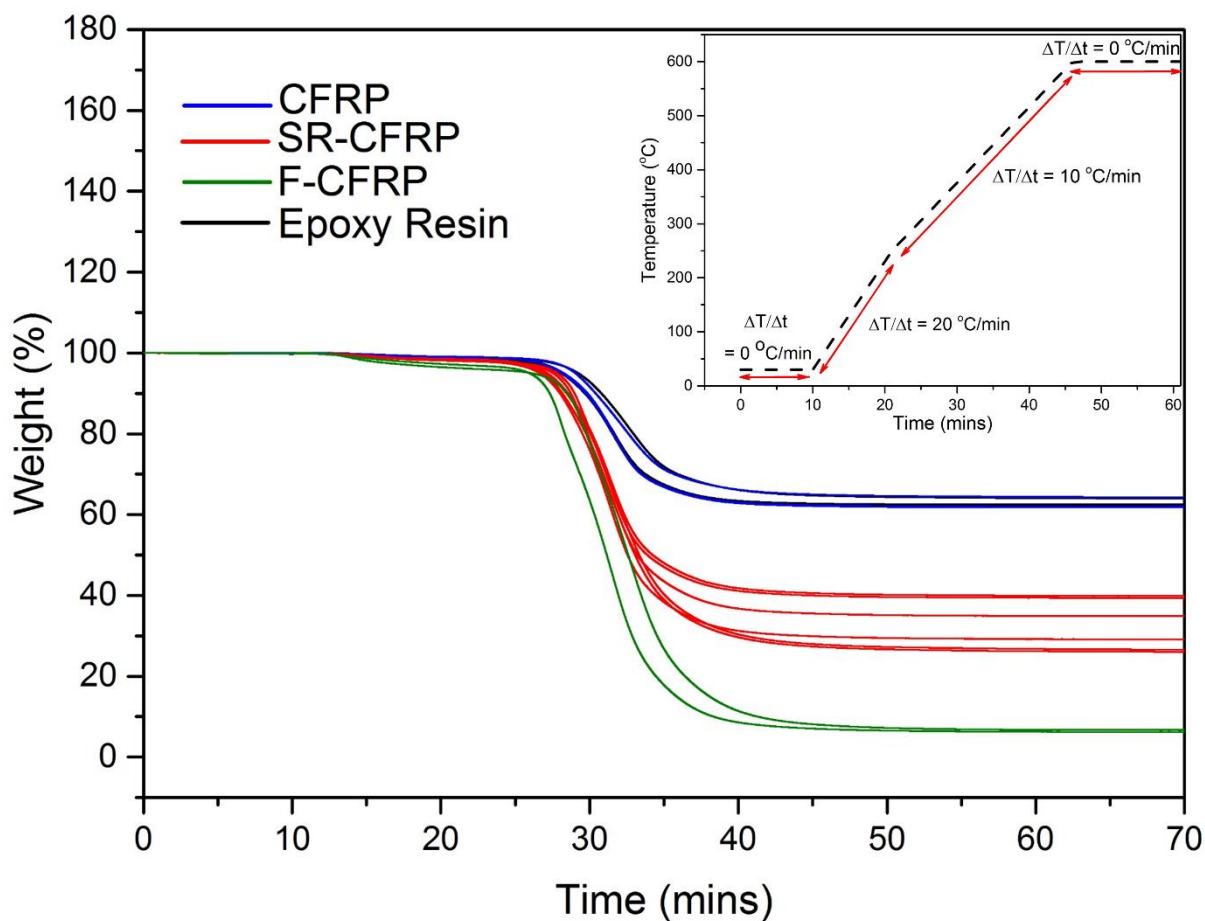
$$V_f = \frac{\left(\frac{W_f}{D_f}\right) + \left(\frac{W_{CNT}}{D_{CNT}}\right)}{\left(\frac{W_f}{D_f}\right) + \left(\frac{W_r}{D_r}\right) + \left(\frac{W_{CNT}}{D_{CNT}}\right)} \quad 3$$

Where W_{CNT} is the weight of the CNTs (see Figure 1a in article) and D_{CNT} is the density of the CNTs (1.74 g/cm³).

Supplementary Figures



Supplementary Figure 1 | Schematic diagram of the vacuum assisted resin transfer moulding (VARTM).



Supplementary Figure 2 | TGA data of weight % *versus* time to determine the fibre volume fraction of each composite. Percentages shown include residue weight. (Inset) TGA process used, carried out under a nitrogen flow (60 ml min^{-1})

Supplementary Tables

Supplementary Table 1 | Key peak positions and intensity ratios to characterise the CNTs on the carbon fibre

	<i>Stoke Shift (cm^{-1})</i>				<i>Intensity Ratios</i>	
	<i>RBM</i>	<i>D</i>	<i>G</i>	<i>2D</i>	<i>I_D/I_G</i>	<i>I_D/I_{2D}</i>
514 nm	-	1339	1575	2676, 2740	0.4	0.5
782 nm	231, 240	1308	1595	2575, 2619	0.7	3.5

Supplementary Table 2 | Polishing process for cross-sectional analysis.

<i>Polishing Stage</i>	<i>Consumable</i>	<i>Lubricants (Flow Rate, $ml\ s^{-1}$)</i>	<i>Polishing Speed (rpm)</i>	<i>Polishing Time (min)</i>
1	Struers A/S, SiC, 200 mm, 500 Grit	Water (13)	50	3
2	Struers A/S, SiC, 200 mm, 1200 Grit	Water (13)	50	3
3	Struers A/S, SiC, 200 mm, 2400 Grit	Water (13)	50	3
4	Buehler FiberMett, 200 mm, 3 μm	Water (1)	50	5
5	Buehler, 200 mm, 1 μm ,	Water (1)	50	5
6	Buehler Ultra-Prep, 200 mm, 0.5 μm	Water (1)	50	5
7	Buehler, 200 mm, 0.3 μm	Water (1)	50	5
8	Buehler FiberMet, 200 mm 0.05 μm ,	Water (1)	50	5

Supplementary References

1. Chen J-S, Stolojan V, Silva SRP. Towards type-selective carbon nanotube growth at low substrate temperature via photo-thermal chemical vapour deposition. *Carbon* **84**, 409-418 (2015).
2. Yee RY, Stephens TS. A TGA technique for determining graphite fiber content in epoxy composites. *Thermochimica Acta* **272**, 191-199 (1996).
3. Kim SH, Mulholland GW, Zachariah MR. Density measurement of size selected multiwalled carbon nanotubes by mobility-mass characterization. *Carbon* **47**, 1297-1302 (2009).