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Counting carbon fibres by electrical resistance measurement

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ABSTRACT

Electrical Impedance Measurement has been used to measure the diameter of single carbon fibres to within 3% of the actual value measured by Scanning Electron Microscopy (SEM). The precision of the technique developed also allows for the accurate determination of the number of fibres present in a carbon fibre bundle, such data are important for the calculation of fibre tensile strength from the tensile force applied to carbon fibre bundles. The impedance of a single carbon fibre and carbon fibre bundles of up to 20 fibres have been measured, with results showing good agreement with theoretical values. The impedance of multiple lengths of carbon fibres ranging from 80 to 300 mm has also been studied, with the impedance being directly proportional to the fibre length, as per electrical theory. This technique will be suitable for determining the number of fibres in a virgin or recycled carbon fibre bundle.

1. Introduction

The tensile strength of carbon fibre is an important parameter when considering the application of the final composite product. With a growing requirement to recycle composite material [1] there will be an increasing need to design a system of classification of recycled fibres. Determination of the tensile strength requires the user to know the tensile force applied and the area to which the said force is applied as shown by Eq. (1) [2]. Calculation of the cross-sectional area of a single carbon fibre is trivial, however the cross-sectional area of a bundle of fibres is difficult to determine. This difficulty arises from the uncertainty in the number of fibres within the bundle. The number of fibres may range from tens of fibres to thousands and consequently manually counting the fibres becomes unfeasible, and approximating by mass is inaccurate owing to the low mass of a single fibre.

Tensile strength:

$$\sigma_f = \frac{F_{max}}{A_f} \tag{1}$$

where

- σ_f = Tensile strength
- F_{max} = Maximum tensile force applied
- A_f = Area over which force is applied

Electrochemical Impedance Spectroscopy (EIS) is a technique that applies a small potential difference, usually in the mV range, to an electrical or electrochemical system and accurately measures the current flowing through the system. In doing so physical systems may be represented by a number of electrical components, such as resistors, capacitors and inductors. By representing systems in this way it is possible to monitor perturbations in the physical system by analysing changes in the electrical properties. EIS has found wide and varied use in high performance materials applications, such as the measuring the quality of polyetheretherketone (PEEK) coatings in aqueous media [3] and the characterisation of Nafion coated micro-electromechanical systems [4]. The deployment of EIS in demanding applications may be, in part, attributed to the accuracy, precision and repeatability of the technique. Fundamental to the use of EIS is the generation of an equivalent circuit, a process by which a physical system is modelled by a set of electrical components. In doing so, the capacitive, resistive and inductive elements of the system are represented with changes to the physical system being modelled by changes in the values of the equivalent circuit components. The generation of the equivalent circuit may not be trivial for many systems, especially for dynamic systems [5]. Owing to the precision of the technique, EIS has also been used to measure the solubility of organic molecules in supercritical fluids, such as the solubility of naphthalene [6], 4-phenyltoluene, phenylboric acid, and iodobenzene in supercritical CO_2 [7].

This paper shows how Electrical Impedance Measurement (EIM) may be used to measure the impedance of a single carbon fibre and further, that the impedance of a bundle of fibres is a func-

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tion of the number of fibres and their length. In addition, the impedance of a single fibre may be used to calculate the diameter of the fibre with precision providing that the length of fibre is well determined. If the phase angle between the voltage and current is 0, it would be possible to measure the resistance of the fibres and therefore negate the use of more sophisticated equipment such as Electrochemical Impedance Spectrometers. The technique developed herein allows for the tensile testing of bundles of carbon fibre, with the intention of negating the requirement to carryout tensile testing of individual carbon fibres, which has been carried out in previous work [8]. In doing so the tensile strength of recycled fibres may be calculated and potentially used as a means of classification.

2. Experimental

2.1. Materials

Carbon fibres having a diameter of 7 μm (manufacture's data), T700S 50E, were obtained from Toray Carbon Fibres America Inc. and used without further treatment. Molybdenum (Mo) wire having a diameter of 25 μm, 99.95%, was obtained from Advent RM and used without further treatment.

2.2. Sample preparation

Individual carbon fibres were separated from the bulk carbon fibre tow manually under a magnifier and confirmed as single fibres by means of optical microscopy and ImageJ imaging software, using 25 μm Mo wire as a reference. Individual carbon fibres were then measured and cut to the specified length prior to being crimped with aluminium foil at either end to provide a gripping surface and electrical contact.

2.3. Electrochemical Impedance spectrometer setup

A Solartron 1250 Frequency Response Analyser and 1286 Electrochemical Interface were used to scan individual carbon fibres through a range of frequencies from 1.5 kHz to 10 Hz, with an amplitude of 25 mV, 25 cycle integration and 0 V DC potential. Data were recorded using Zplot and analysed using Zview software (Scribner Associates Inc.). A schematic representation of the 2-point experimental setup is provided in Fig. 1.

Each frequency sweep was repeated 9 times on each fibre sample prior to changing the setup. Using Eq. (2) the diameter of the individual carbon fibres was then calculated.

Resistivity of material:

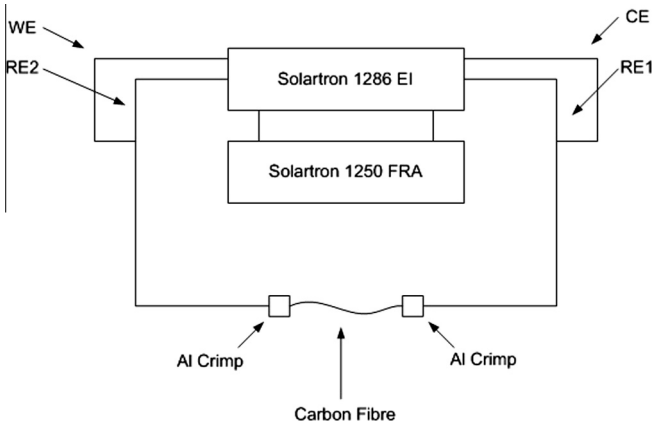

Fig. 1. EIS schematic diagram, working electrode (WE), counter electrode (CE), reference electrode (RE).

$$\rho = R\frac{A}{l} \quad \text{Since } R = Z \quad \text{when} \quad \phi = 0 \quad \rho = Z\frac{A}{l} \tag{2}$$

where

- ρ = Resistivity (Ω m)
- R = Resistance (Ω)
- A = Area (m^2)
- l = Length (m)
- Z = Impedance (Ω)
- ϕ = Phase angle (^{O})

After obtaining the impedance of an individual carbon fibre an additional carbon fibre was prepared and added to the EIM system. The impedance sweep was then carried out and repeated 9 times. This process was carried out on between 1 and 20 fibres.

In a separate experiment the length of the carbon fibre was varied from 80, 120, 150, 200, 250 and 300 mm, using single fibres. The fibre length was measured using a steel rule with 1 mm graduation intervals. The frequency sweep conditions were preserved and each measurement was repeated 9 times.

2.4. Scanning electron microscopy (SEM)

Samples were imaged using a Philips XL30 FEG ESEM. Since carbon fibres are conductive Gold sputtering was not necessary. Sections of individual carbon fibres were cut to approximately 15 mm and mounted directly onto adhesive stub mounts. Once mounted onto stubs, samples were loaded individually into the SEM and the sample chamber was evacuated. All images were taken with a working distance of 10.1 mm, an acceleration voltage of 20 kV and a magnification of 6500 times.

3. Results and discussion

The system impedance, including the aluminium crimps, was measured as 0.018 Ω. For low impedance measurements this value should be subtracted from the impedance measured. However, since the system impedance represents less than 0.01% of the impedance measured in all cases, it is deemed to be negligible and is consequently neglected. The equivalent circuit for the system is presented in Fig. 2.

3.1. Analysis on single fibres

Fig. 3 shows a single carbon fibre as observed by optical microscopy with a 25 μm Mo wire for scale. Bundles of fibres (from 2 to 20) were constructed by obtaining single fibres individually and compiling them. Since the fibres are obtained individually and added to the bundle, the number of fibres present in the bundle was always known.

The presence of single carbon fibres was also confirmed by SEM, see Fig. 4. It was necessary to conduct the impedance measurements prior to the SEM since the fibres were not recoverable from the SEM stage. For this reason, single fibres were initially identified by optical microscopy.

The impedance data measured at 1.5 kHz and 10 Hz for a single 85 mm fibre (Toray T700S 50E) have been tabulated and are presented in Table 1. It is noted that the impedance value is independent of the frequency (since the fibres act as resistors), and

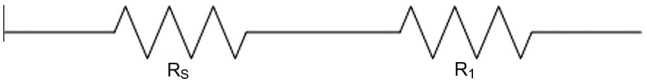

Fig. 2. EIS equivalent circuit, system impedance (R_S), carbon fibre impedance (R_1).

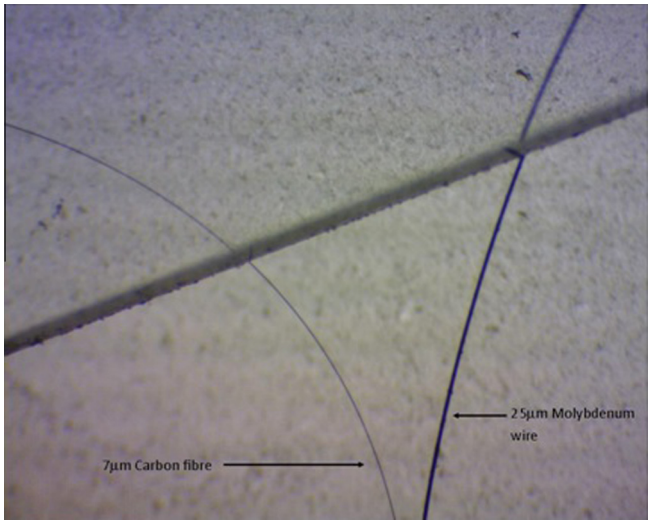

Fig. 3. Single carbon fibre with 25 μm Molybdenum wire for scale (optical microscope) (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.).

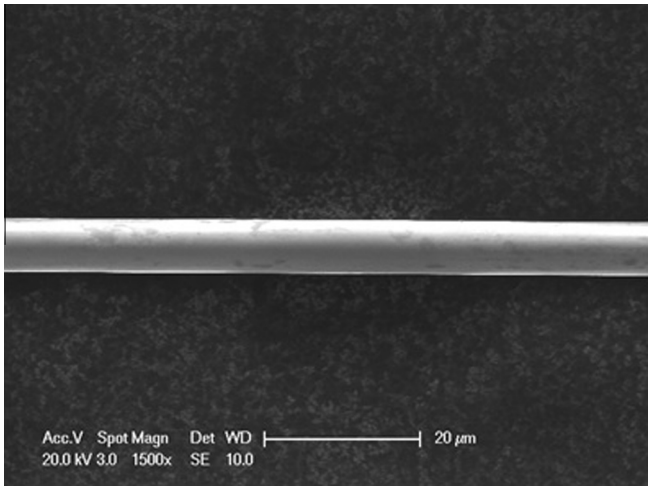

Fig. 4. SEM micrograph of the single carbon fibre, 150 mm in length (full length not shown), subsequently used in Electrical Impedance Measurements.

Table 1
Impedance at 1.5 kHz and 10 Hz for a single 85 mm carbon fibre.

Sweep number	Z (Ω) (1.5 kHz)	Z (Ω) (10 Hz)
1	42,168	42,557
2	43,081	42,889
3	43,032	42,086
4	42,821	42,626
5	42,615	43,140
6	43,370	43,393
7	43,491	43,407
8	43,077	43,039
9	42,975	42,932
10	42,748	42,703
Mean	42,938	42,877
SD	359	383

consequently the impedance measured at any frequency throughout the sweep would be sufficient to calculate the fibre diameter.

It is explicit from Eq. (2) that the impedance will increase linearly with increasing length of fibre. In order to verify this single carbon fibres of varying lengths, 80, 85, 120, 150, 200, 250 and 300 mm, were prepared and their impedance measured. Fig. 5 shows the linear increase in impedance observed with fibre length.

The presence of a single carbon fibre, observed by use of an optical microscope, was confirmed by SEM. The use of optical and scanning electron microscopy has been used to ratify the results obtained by EIM. By transposition of Eq. (2), the cross-sectional area, and therefore the fibre diameter, may be elucidated. The single fibre diameters calculated from EIM and measured by SEM are presented in Table 2 for different fibre lengths.

Analysis of Table 2 shows that the EIM technique presented provides a 'reasonably good' fit with the manufacturer's datasheet (7 μm) when applied to single fibres, especially when considering fibres with a length in excess of 150 mm. This increase in accuracy is thought to be attributed to the reduced percentage error in the length measurement which is done by hand. However, the small amount of error present in the length measurement is inconsequential for fibre bundles since all of the fibres are crimped at the same point and thus the length through which the current flows is essentially the same. Only the effect of the number of fibres therefore is observed.

The diameter of single carbon fibres was confirmed by means of SEM, see Fig. 6. When measured by SEM the diameter of a 150 mm single fibre was found to be 6.85 μm, the diameter calculated by EIM was 6.89 μm. Furthermore, the diameter of the 120 mm carbon fibre determined by SEM was 6.72 μm, (see Fig. 7), while the diameter calculated by EIM was 6.55 μm, again showing good agreement between the values. The variation in fibre diameter along the length of a fibre was investigated by means of SEM. Diameter measurements were taken at 5 arbitrarily selected points for 5 arbitrarily selected fibres. The standard deviation was less than 0.05 for any single fibre and 0.13 for the entire sample population. Full data are available in Table A1. The greatest variation observed within a fibre (0.09 μm) represents an error of less than 3%. Therefore, variation within a fibre is not thought to be a significant source of error.

It is therefore evident from both optical and Scanning Electron Microscopy that the Electrical Impedance Measurement approach presented is sufficiently accurate, precise and sensitive to detect and measure the dimensions of individual carbon fibres.

3.2. The approach for fibre bundles

The mean impedance of an 85 mm single carbon fibre was obtained to be 42.9 kΩ ± 2%, Table 1. The impedance value for each sweep at 1.5 kHz and 10 Hz was used to generate the average

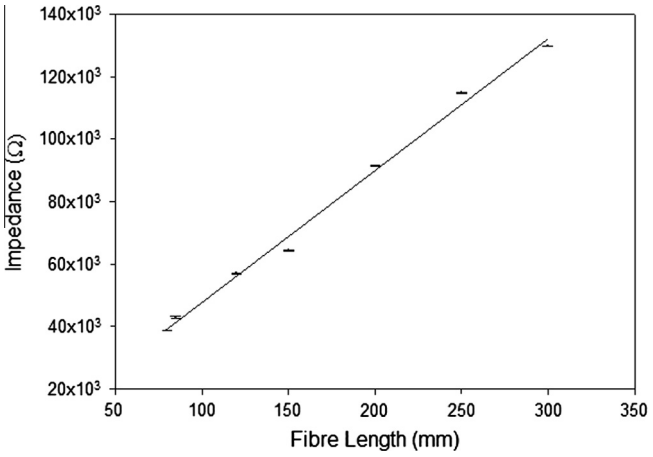

Fig. 5. Impedance as a function of fibre length for single carbon fibres.

Table 2
Single fibre diameter as calculated by (EIM) and measured by (SEM).

Nominal length (mm)	Diameter EIM (μm)	Diameter SEM (μm)	Difference (%)
80	6.49	6.52	0.46
85	6.35	6.46	1.70
120	6.55	6.72	2.53
150	6.89	6.85	0.58
200	6.68	6.22	7.40
250	6.66	6.48	2.78
300	6.86	6.55	4.73

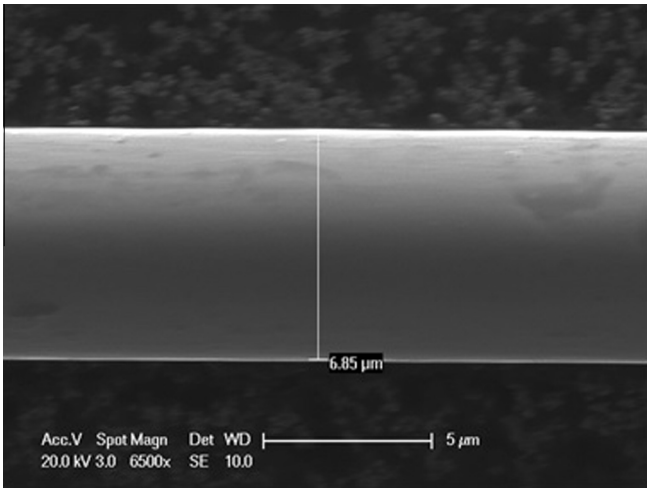

Fig. 6. SEM micrograph of single carbon fibre, 150 mm in length.

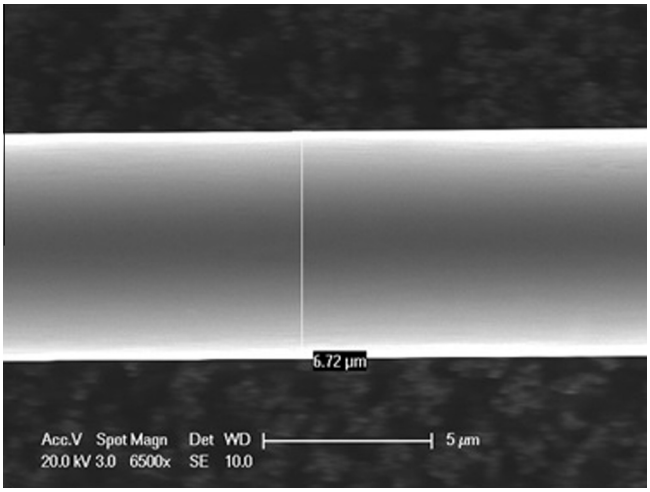

Fig. 7. SEM micrograph of a single carbon fibre, 120 mm in length, used in the Electrical Impedance Measurement.

impedance for bundles containing 2–20 fibres that were again 85 mm in length. These data are collated in Fig. 8.

Since the phase angle (ϕ) between the voltage (V) and current (I) is approximately 0, and hence no frequency dependence of the impedance, single carbon fibres behave as resistors. The total impedance of the system may therefore be determined by Eq. (3).

System total impedance, resistors in parallel:

$$\frac{1}{Z_T} = \frac{1}{Z_1} + \frac{1}{Z_2} \cdots \frac{1}{Z_n} \tag{3}$$

It follows from Eq. (3) that for a constant fibre length the total impedance of the system is a function of the number of fibres. As the fibre length is known the number of fibres may be calculated

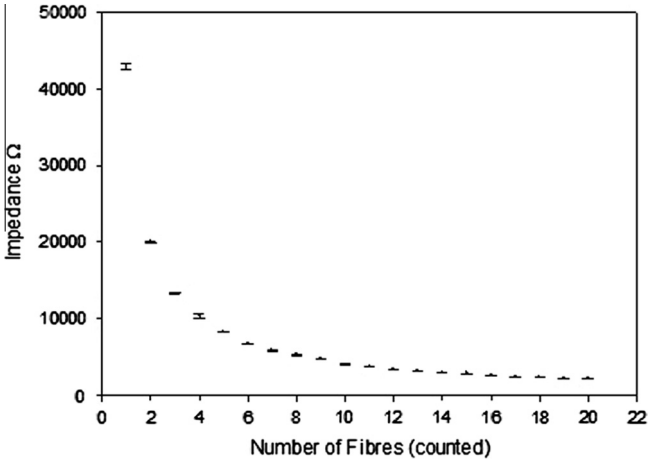

Fig. 8. Overall impedance as a function of number of fibres.

by transposition of Eqs. (2) and (3). It also follows that for a given fibre length, if the number of fibres is doubled the impedance is halved (see Fig. 8).

It has been possible to take a bundle of fibres, cut and crimped to a specified length, and calculate the number of fibres present by means of EIM. This in turn allows for the tensile testing of carbon fibre bundles and, as long as the bundle length is measured prior to the test, the number of fibres may be determined as shown by calculation 1 below. This is significant since it allows the tensile strength of the individual fibres within the bundle to be calculated, a step which is not possible without knowing the number of fibres present.

Calculation 1, determining the number of fibres in a bundle;
Since;

For bundles;

$$\rho = Z_T \frac{A_T}{l}, \quad \text{when} \quad \phi = 0,$$

$$\frac{\rho l}{A_T} = Z_T$$

For single fibres;

$$\rho = Z_S \frac{A_S}{l}, \quad \text{when} \quad \phi = 0,$$

$$\frac{\rho l}{A_S} = Z_S$$

where

- Z_T = Total impedance of bundle (Ω)
- Z_S = Impedance of single fibre (Ω)
- A_T = Total cross-sectional area of conduction through bundle (m^2)
- A_S = Cross-sectional area of single fibre (m^2)
- ρ = Resistivity (Ωm)
- l = Length of fibre (m)

where A_T is the total cross-sectional area through which conduction takes place in a bundle, A_S is the cross-sectional area of a single fibre. Measuring the total impedance of a carbon fibre bundle allows the number of fibres present to be determined by the quotient A_T/A_S. For a carbon fibre bundle consisting of an unknown number of 85 mm fibres, the number of fibres present may be deduced in the following way:

By EIM, $Z_T = 2858.3\ \Omega$

$$Z_S = 42907.5\ \Omega$$

$$\frac{Z_S}{Z_T} = \frac{\rho l A_T}{\rho l A_S} = \frac{A_T}{A_S} = \#fibres$$

And therefore;

$$\frac{Z_S}{Z_T} = \frac{42907.5}{2858.3} = 15.01$$

Thus, it is determined that the bundle consists of 15 fibres which was verified by optical microscopy. This bundle of fibres may then be prepared for tensile testing and, as the number of fibres is known, the tensile strength of the fibres may be deduced.

Aside from the fibre length measurement, one source of error with this technique is that there could be instances when there are a small number of fibres not connected to an Al crimp. In such instances, the fibres that are not in contact with either the crimp or another fibre would not transmit the current, and therefore would not contribute to the impedance of the system. However, in a bundle of fibres should one end of a fibre become disconnected it is likely that the fibre will remain in contact with many of the other fibres by proximity alone. The majority of the fibre's length is subsequently available for conduction and the overall effect on the impedance measurement is small. This effect of touching carbon fibres providing an alternative current pathway has been identified previously in work by Irving and Thiagarajan [9]. This error is further reduced in large bundles, where a bundle may commonly consist of several hundred fibres. In such instances the loss of a small proportion of a single fibre, or small number of fibres, becomes negligible on impedance and related measurements as shown by Eq. (4).

Effect of fibre loss on impedance of bundles:

$$\%Z_{inc} = \left(\frac{n_L}{n_I} \times 100\right) \tag{4}$$

where

- $\%Z_{inc}$ = Percentage increase in impedance due to reduction in number of fibres
- n_L = Number of fibres removed, assuming entire fibre length is non-conducting
- n_I = Initial number of fibres within bundle

3.3. Limit of detection

In principle, as long as the potential is applied to all the fibres the impedance of the system may be measured. This work demonstrates that the removal of a single fibre from a bundle of 20, amounting to a change of 5% in the bundle size, is easily detected (Fig. 8). This technique readily detects changes as small as 5% in the bundle size. The limit of detection is concerned with the particular experimental setup. The largest bundle of 80 mm fibres that may be analysed is 215,000. Bundles in excess of this size would have an impedance within 10% of the system impedance (excluding carbon fibre), at which point the contribution of the Al crimps and associated cabling to the overall system impedance may not be negligible. The equivalent circuit shown in Fig. 2 would no longer accurately model the system and a new equivalent circuit would need to be developed. Given that the impedance of a carbon fibre is directly proportional its length, the limit of detection (LOD) is also a function of the fibre length. The LOD increases with increasing fibre length as shown in Table 3.

The work herein demonstrates that there is no frequency dependence when measuring the impedance of carbon fibres, and as such, carbon fibres behave as resistors. Consequently it would be possible to conduct this procedure using less sophisticated equipment, operating with Direct Current (DC) as opposed to Alternating Current (AC) as performed here. DC measurements of carbon fibres yield resistance, where the magnitude of the resistance is identical to the magnitude of the impedance since the phase angle is 0. The approach presented was necessary to demonstrate the applicability, reliability and validity of the method.

Table 3
Limit of detection (LOD) for fibre bundles.

Nominal length (mm)	LOD (# of fibres)
80	215,000
85	235,000
120	315,000
150	355,000
200	505,000
250	635,000
300	720,000

4. Conclusion

The Electrical Impedance Measurement technique presented may be used to measure the diameter of individual carbon fibres. This information may in turn be used to identify, with precision, the number of individual carbon fibres present in a bundle. The current alternative is to manually count the fibres or approximate the number of fibres by mass, which when considering bundles consisting of thousands of fibres becomes impractical. Knowledge of the number of carbon fibres present facilitates calculation of the area over which the tensile force is applied during tensile testing by means of universal testing equipment, and thus provides the user with the average tensile strength of the carbon fibres tested.

Table A1
Fibre diameter variability with respect to length.

Fibre number	Position number	Diameter (μm)	Mean (μm)	Standard deviation (fibre) (μm)
1	1	6.90	6.91	0.01
	2	6.90		
	3	6.90		
	4	6.92		
	5	6.92		
2	1	6.94	6.90	0.03
	2	6.88		
	3	6.88		
	4	6.92		
	5	6.86		
3	1	7.17	7.16	0.01
	2	7.17		
	3	7.15		
	4	7.15		
	5	7.15		
4	1	6.88	6.85	0.04
	2	6.81		
	3	6.81		
	4	6.86		
	5	6.90		
5	1	7.13	7.11	0.02
	2	7.12		
	3	7.12		
	4	7.08		
	5	7.08		
Minimum		6.81		
Maximum		7.17		
Mean		6.98		
Standard deviation		0.13		

In doing so, the mechanical testing of individual carbon fibres is alleviated and the mechanical testing of fibre bundles is possible. This is a significant step forward in standardising recycled fibres given that the attractive mechanical qualities of carbon fibre are often associated with fibre bundles and seldom individual fibres. Future work in this area will assess the viability of extending this technique to directly assessing the mechanical properties of carbon fibre bundles.

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Appendix A

See Table A1.

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