THE DETERMINATION OF CARBON FIBRE BUNDLE SIZES BY ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

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Abstract
Electrochemical Impedance Spectroscopy (EIS) 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 80-300 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

1.1. Recycling carbon fibre reinforced polymers

In order to effectively recycle carbon fibre reinforced polymers it is necessary to design a holistic process that considers the destination of the recycled/degraded resin and that of the carbon fibres. One challenge is the separation of the various reaction products and the isolation of the fibres free of resin, another challenge is the characterisation both the organic phase and the carbon fibres[1]. One method of characterising the carbon fibres is to consider their tensile properties[2]. By carrying out tensile testing on a single fibre it is possible to obtain the tensile strength of the said fibre, providing that the diameter, and therefore, cross-sectional area is known. The tensile strength may be calculated according to equation 1.

$$\sigma_f = \frac{F_{\max}}{A_f} \quad (1)$$

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

The tensile strength obtained for a reclaimed fibre may then be compared to that of a virgin fibre and the quotient may be used as an indication as to the effects of the processing conditions. The challenge faced in carrying out characterisation by this method is that there is a requirement to ensure that the number of single fibres tested is representative of the bundle of fibres from which they originate. This necessitates the tensile testing of hundreds, if not thousands, of single carbon fibres from a single sample. As the bundle size increases it becomes unfeasible to test carbon fibres by this method due to the physical limitations of isolating and preparing thousands of single fibres.

Another approach that would help alleviate the requirement to test large numbers of single fibres would be to test fibres bundles. Providing that the carbon fibres are of the same type and have been processed in the same way it would be possible to carryout tensile testing on carbon fibre bundles, using statistics to describe the behaviour of the fibres within the bundle[3]. Tensile testing would yield a maximum tensile force exerted on the bundle and, by integration of the curve, the work done by the Universal Testing Machine (UTM). Calculating the tensile strength of the average fibre in the bundle would require accurate determination of the number of fibres in the bundle. Given that carbon fibres are readily conductive, by using Electrochemical Impedance Spectroscopy (EIS) it is possible to exploit this property to determine, with precision, the number of fibres in the bundle.

Electrochemical Impedance Spectroscopy 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. This paper shows how EIS may be used to measure the impedance of a single carbon fibre and further, that the impedance of a bundle of fibres is a function 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. The technique developed herein allows for the tensile testing of bundles of carbon fibre, with the intention of negating the requirement to carryout extensive tensile testing of individual carbon fibres, which has been carried out in previous work[2]. In doing so the tensile strength of recycled fibres may be readily calculated for bundles of fibres and potentially used as a means of classification.

1.1. Materials

Carbon fibre having a diameter of 7 μm (manufacture's data), T700S 50E, was 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.

1.2. 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 25μm 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.

1.3. 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 0V 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 Figure 1.

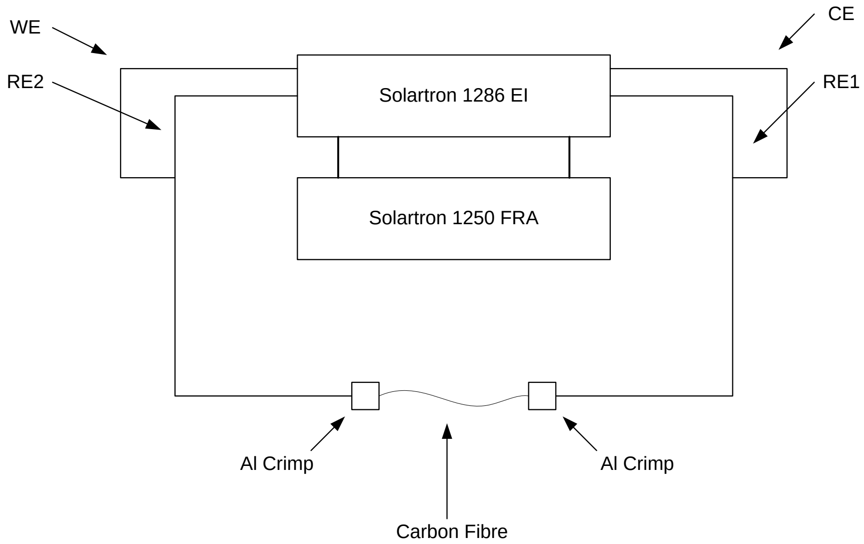

Figure 1: EIS Schematic Diagram, working electrode (WE), counter electrode (CE), reference electrode (RE)

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

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

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 EIS 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 measure using a steel rule with 1 mm graduation intervals. The frequency sweep conditions were preserved and each measurement was repeated 9 times.

1.4. 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.

2. 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 Figure 2.

Figure 2: EIS Equivalent circuit, system impedance (R_S), carbon fibre impedance (R_1)

2.1. Analysis on Single Fibres

The presence of single carbon fibres was confirmed by SEM, see Figure 4. It was necessary to conduct the EIS 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 consequently the impedance measured at any frequency throughout the sweep would be sufficient to calculate the fibre diameter.

Sweep Number	Z (Ω) (1.5kHz)	Z (Ω) (10Hz)
1	42168	42557
2	43081	42889
3	43032	42086
4	42821	42626
5	42615	43140
6	43370	43393
7	43491	43407
8	43077	43039
9	42975	42932
10	42748	42703
Mean	42938	42877
SD	359	383

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

It is explicit from equation 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. Figure 3 shows the linear increase in impedance observed with fibre length.

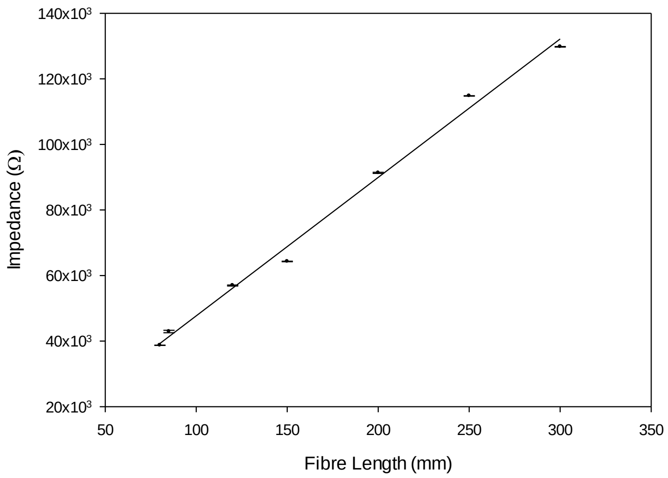

Figure 3: Impedance as a function of fibre length for single carbon fibres

The use of optical and scanning electron microscopy has been used to ratify the results obtained by EIS. By transposition of equation 2, the cross-sectional area, and therefore the fibre diameter, may be elucidated. The single fibre diameters calculated from EIS and measured by SEM are presented in Table 2 for different fibre lengths.

Nominal Length (mm)	Diameter EIS (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

Table 2: Single fibre diameter as calculated by (EIS) and measured by (SEM)

Analysis of Table 2 shows that the EIS 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 Figure 4. When measured by SEM the diameter of a 150 mm single fibre was found to be 6.85 μm, the diameter calculated by EIS was 6.89 μm. The diameter of the 120 mm carbon fibre determined by SEM was 6.72 μm, the diameter calculated using the EIS method was 6.55 μm, 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 diameter standard deviation was less than 0.05 for any single fibre and 0.13 for the entire sample population. Variation within a fibre is therefore not thought to be a significant source of error.

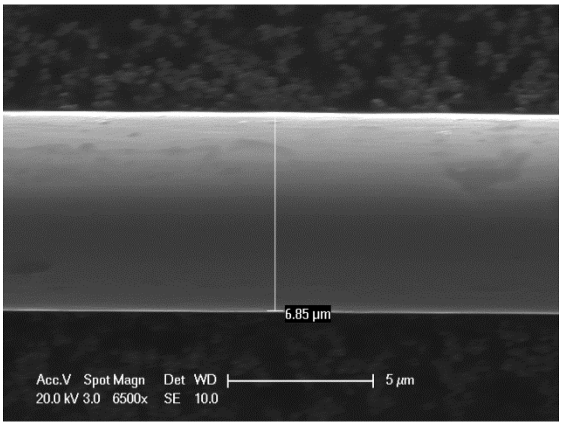

Figure 4: SEM Micrograph of single carbon fibre, 150mm in length

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

2.2. 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 impedance for bundles containing 2 to 20 fibres, that were again 85 mm in length. These data are collated in Figure 5.

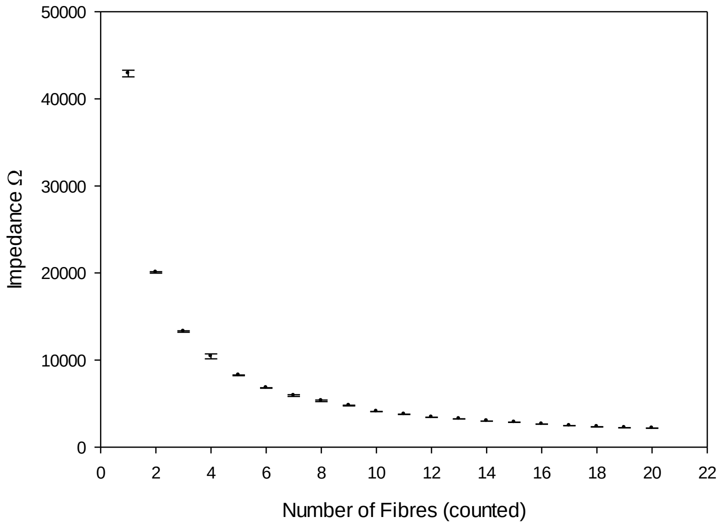

Figure 5: Overall impedance as a function of number of fibres

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 equation 3.

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

It follows from equation 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 by transposition of equations 2 and 3. It also follows that for a given fibre length, if the number of fibres is doubled the impedance is halved (see Figure 5).

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 EIS, see calculation 1. This in turn allows for the tensile testing of carbon fibre bundles and, as long as the length is measured prior to the test, the number of fibres may be determined.

2.2.1. Calculation 1

By EIS measurement, $Z_T = 2858.3\ \Omega$

$Z_S = 42907.5\ \Omega$

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

And therefore; $\frac{Z_S}{Z_T} = \frac{42907.5}{2858.3} = 15.01$

Where: Z_T = Impedance of the bundle, Z_S = Impedance of a single fibre, ρ = Resistivity, A_T = Total cross-sectional area of conduction, A_S = Cross-sectional area of a single fibre

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 the, as the number of fibres is now known, the tensile strength of the fibres may be deduced from equation 1.

3. Conclusion

The Electrochemical Impedance Spectroscopy 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. 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. 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.

References

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