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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.
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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:

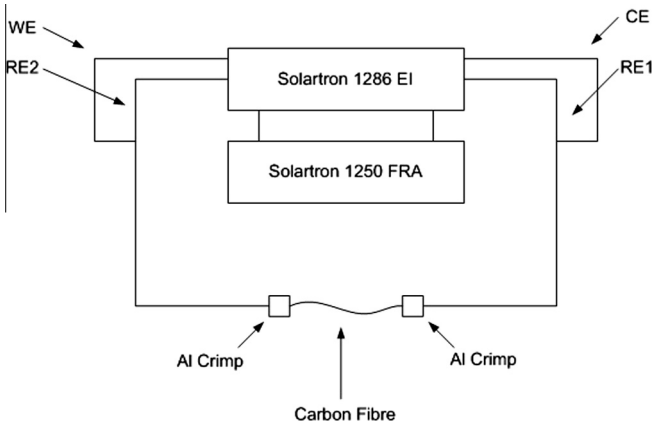

Fig. 1. EIS schematic diagram, working electrode (WE), counter electrode (CE), reference electrode (RE).

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

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

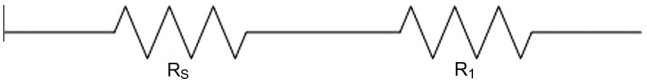

Fig. 2. EIS equivalent circuit, system impedance (R_S), carbon fibre impedance (R_1).

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