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Measuring the Electrical Conductivity of Single Fibres

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The methodology required to measure the conductivity of single fibres is outlined, the basic underlying physics discussed and recent pertinent examples culled from the scientific literature are presented for illustration. Indications of where the field will progress are presented.

Keywords: Electrical conductivity measurement ; single fibre conductivity; electrospinning

1. INTRODUCTION

Textiles are ubiquitous. Not only do they protect but they also have aesthetic appeal and cultural importance. Recent technological advances have allowed the traditional functionality of textiles to be extended. Advances in materials nanoscience have added intelligence to textiles and created smart apparel which can sense and react to environmental conditions or stimuli from, for example, mechanical, thermal, chemical, electrical, or magnetic sources. Such textiles find uses in numerous applications ranging from military and security to personalised healthcare, hygiene and entertainment [1-3].

Smart textiles may be deemed passive or active. A passive smart material monitors the wearer's physiology or the external environment such as a shirt with in-built thermistors to log body temperature over time. If actuators are integrated the textile material becomes an active smart material as it may respond to a particular stimulus such as in the temperature aware shirt may automatically roll up the sleves when the body temperature becomes elevated.The fundamental components in any smart textile are sensors and actuators. Interconnections, power supply and control units are also needed to complete the system. These components must all be integrated into textiles while still retaining the usual tactile flexible and comfortable properties which one expects from a textile. Materials such as

metals, optical fibres and conductive polymers may be integrated into the textile structure, thus supplying electrical conductivity, sensing capability and data transmission capability to the material.

The experimental determination of both the thermal and electrical conductivity of single fibres presents a challenge. Fibres are characterized by having one very long dimension and the other two very small. This makes the determination of their mechanical and physical properties far from trivial. In particular, determination of their transverse properties, i.e. in the direction of the fibre diameter, can be difficult. In the present document we report on recently developed yet readily attainable methodologies by which such important physical measurements may be made. Much work has been reported on carbon based fibres in recent years and the recent papers written by Zhang and co-workers [4], Safarova et al [5], Sundaray et al [6], Monkman and co-workers [7], Fujii et al [8] and Imai et al [9] are of note.

2. MEASUREMENT PROTOCOL

The electrical resistivity (or conductivity) of a fibre can be measured in the most basic scheme by means of simple resistance probe. Both two probe and four probe variants are often utilized when measuring the resistivity of a material sample (figure 1). In the former method a uniform current density is applied across the specimen sandwiched between two electrodes located on parallel faces (fig.1(A)) and measure the potential drop across the latter electrodes.

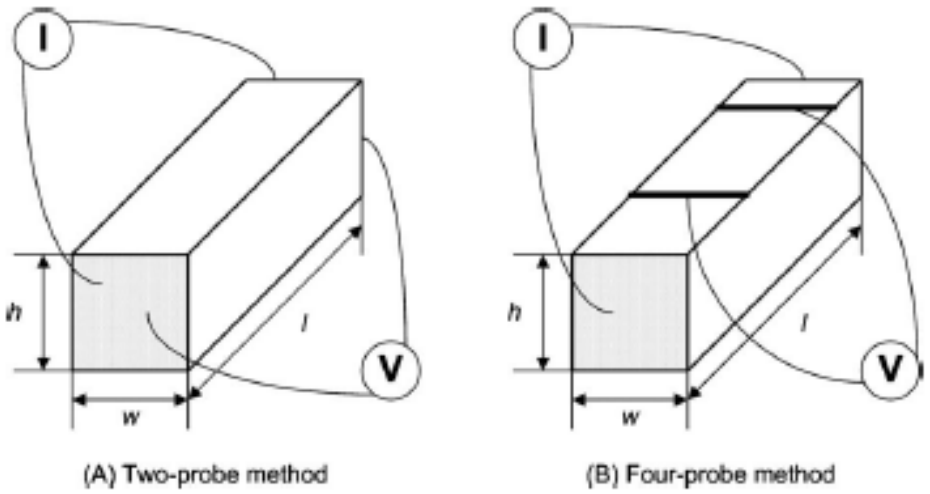

Figure 1. Schematic representation of resistance measurement across a solid material sample of defined geometry. (A) Two-probe method and (B) four probe method.

Here current injection and voltage measurement are made at the same two electrodes. Note that the two probe method is highly sensitive to the contact resistance at the current injection electrodes because the measured potential difference includes the potential drop across the electrode and its

interfaces. As noted using the resistance measured between two electrodes, the resistivity can be measured when the material specimen has a simple geometry of known dimensions. In the four probe method (see fig.1(B)), one pair of probes is used for the current injection at a pair of electrodes while the othe pair is used for voltage measurements on a different set of electrodes. Again the resistance is determined using Ohm's law and the resistivity can be calculated if the applied current is uniform and the specimen dimensions are known. The four probe method provides results which are more reliable because it is not sensitive to contact resistance issues.

These approaches for measuring the resistivity are well suited for simple blocklike specimens subjected to uniform currents, where the electrodes cover the entire sides of the specimen. The measurement of resistivity in conductive polymer composites has got quite sophisticated [10,11].

A simple methodology for determining the resistance of a section of a single fibre can be visualized as follows in fig.2 . The fibres are hung by a weight to keep them taut. Electrical resistance is measured at specific intervals of length. The slope of a plot of electrical resistance against length gives the resistivity. The electrical resistivity denoted by ρ (unit: Ωm) is given by:

$$\rho = \frac{RA}{L} = \frac{\pi d^2 R}{4L}$$

Here d represents the fibre diameter, R is the electrical resistance (unit: Ω) and L denotes the fibre length (unit: m). Note that the fibre diameter d (unit: m) is a very important parameter in characterizing a fibre. One can make a direct measurement of fibre diameter by means of an optical or scanning electron microscope. There is an ASTM standard (D 578) for this purpose. The main problem with direct measurement is that fibre diameter may not be uniform along the length. An indirect method that gives an average fibre diameter is to weigh a known length of fiber and use the following simple relationship:

$$d = \sqrt{\frac{w}{\pi \rho_m L n}}$$

where w denotes the mass of the fibre sample, ρ_m represents the density of the fibre and n is the number of filaments per tow, if a tow of fibre is being used. Often fibres have an irregular cross-section. The above method will give an equivalent diameter of a fibre having an irregular cross-section. One can also take a photograph of such a fibre and measure the area planimetrically. Note that the fibre diameter can also be measured by laser beam. The technique can be rapid and systematic. The radius of the fibre is given by:

$$r = \frac{\lambda s}{\delta}$$

where λ denotes the wavelength of the laser beam, s is the distance between the fibre and the projection screen and δ denotes the distance between the first intensity minima of the diffraction

pattern. The density of a fibre can be measured by the same techniques as those used for conventional, bulk materials. The Archimedes method can be used in a straight forward manner to measure this simple but important property. One weighs the mass of a fibre sample in air and in a liquid of known density, for example, ethanol. Then the following expression gives the density:

$$\rho_m = \left\{ \frac{m_a}{m_a - m_\ell} \right\} \rho_\ell$$

Here m_a, m_ℓ denote the mass of the fibre sample in air and in the liquid respectively, and ρ_ℓ denotes the density of the liquid.

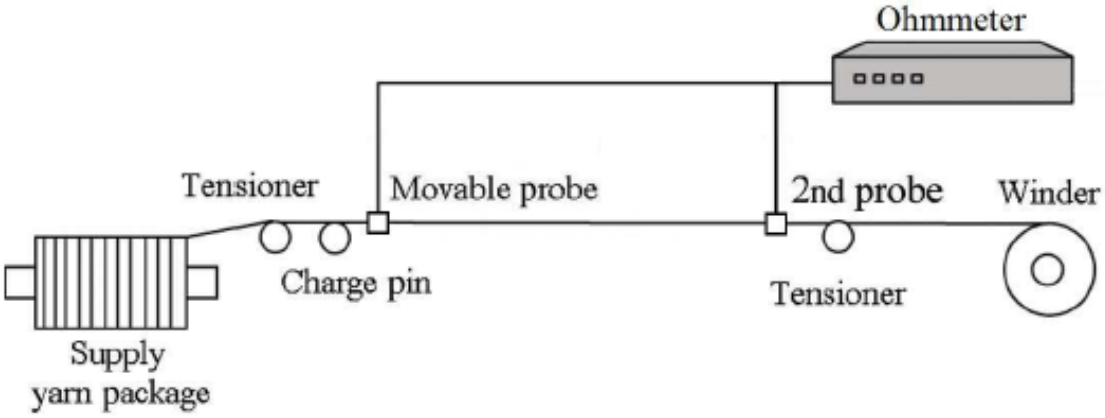

Figure 2. Schematic representation of resistance measurement of single fibre.

The two probe technique of fibre resistance measurement reported by Safarova et al [5] is illustrated schematically in fig.2. Here the resistance of the fibre is measured as a function of distance x along the fibre which represents the distance between the two probes, one which is fixed which serves as a zero and the other moveable. Hence we note that:

$$R = \frac{\rho x}{A} = \frac{4\rho}{\pi d^2} x$$

Hence a plot of R versus x should be linear with a slope yielding the resistivity of the fibre given by $S = 4\rho / \pi d^2$.

Electrospinning [12] uses an electrical charge to draw very fine (typically on the micro or nano scale) fibres from a liquid. Electrospinning shares characteristics of both electrospraying and conventional solution dry spinning of fibres. The process is non-invasive and does not require the use of coagulation chemistry or high temperatures to produce solid threads from solution. This makes the process particularly suited to the production of fibers using large and complex molecules. Electrospinning from molten precursors is also practiced; this method ensures that no solvent can be

carried over into the final product.The electrospinning process is the only established method for producing continuous polymeric fibres with diameters in the nanometer range (figure 3). When a threshold voltage is applied to the polymer solution, electrostatic forces overcome the surface tension of the pendant droplet to form nanofibres.

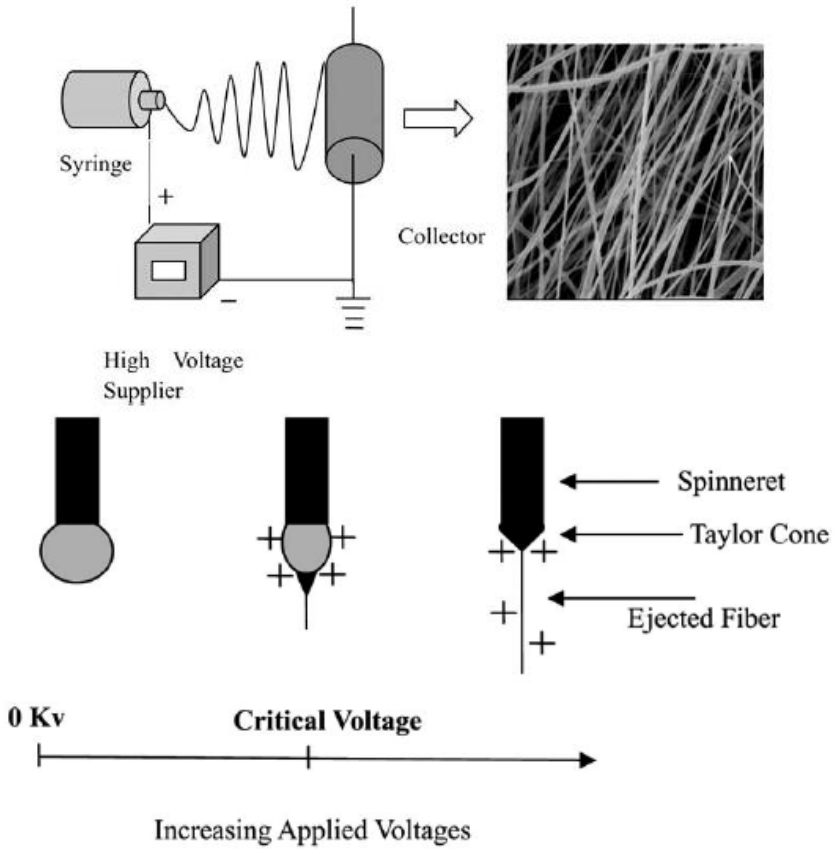

Figure 3. Schematic representation of electrospinning setup.

When a sufficiently high voltage is applied to a liquid droplet, the body of the liquid becomes charged, and electrostatic repulsion counteracts the surface tension and droplet is stretched, at a critical point a stream of liquid erupts from the surface. This point of eruption is known as the Taylor cone. If the molecular cohesion of the liquid is sufficiently high, stream breakup does not occur (if it does, droplets are electro-sprayed) and a charged liquid jet is formed. As the jet dries in flight, the mode of current flow changes from ohmic to convective as the charge migrates to the surface of the fibre (figure 4). The jet is then elongated by a whipping process caused by electrostatic electrostatic repulsion initiated at small bends in the fibre, until it is finally deposited on the grounded collector. The elongation and thinning of the fibre resulting from this bending instability leads to the formation of uniform fibres with nanometer nanometer-scale diameters (figure 5). The theoretical description of the electrospinning process is complex and has been examined for many years. Seminal work was conducted by Taylor [13] and more recently by Reznik [14] and Hohman [15]. A useful review has been provided by Doshi and Reneker [16].

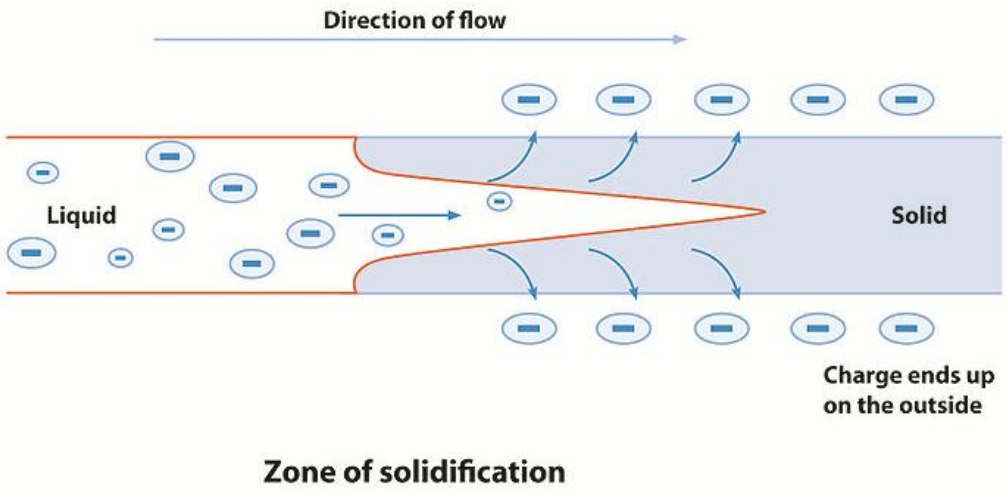

Figure 4. Illustration of the manner in which the distribution of charge in the fibre changes as the fibre dries during flight (adapted from [12]).

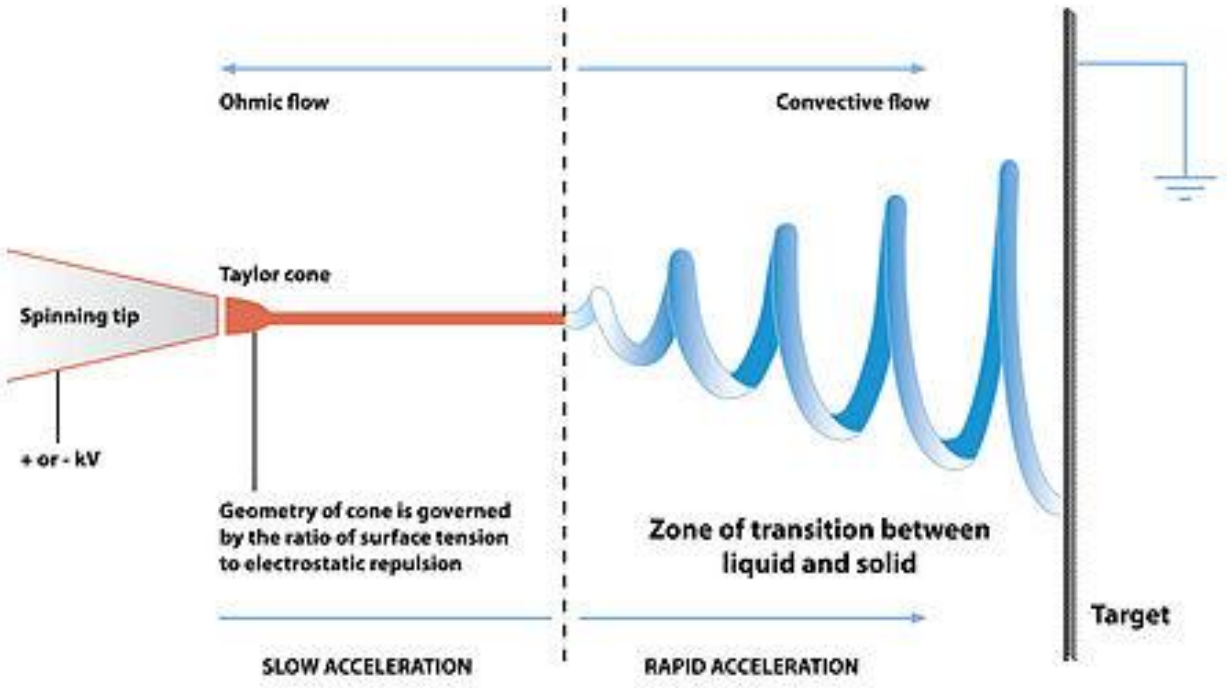

Figure 5. Illustration of fibre formation during electrospinning (adapted from [12]).

A particularly nice example of what can be done with electrospun single fibres is taken from the recent work reported by Natarajan et al [17] who examined the electrical properties of electrospun poly(ethylene oxide)/poly(pyrrole) composite fibres of diameters in the range 100 nm – 10 mm. They obtained well defined current-voltage curves using a two point probe technique using gold electrodes (figures 6 and 7). From such measurements the DC conductivity of a single PEO/PPY fibre (50 %

w/w) is typically of the order 10^{-4} S cm^{-1}. The conductivity typically increases with wt % of PPy as it should (fig.4(B)). These workers have extended their study to electrospun fibres of poly(methyl methacrylate)/single wall carbon nanotube composites [18], and extended the experiments to consider the temperature variation of electrical conductivity.

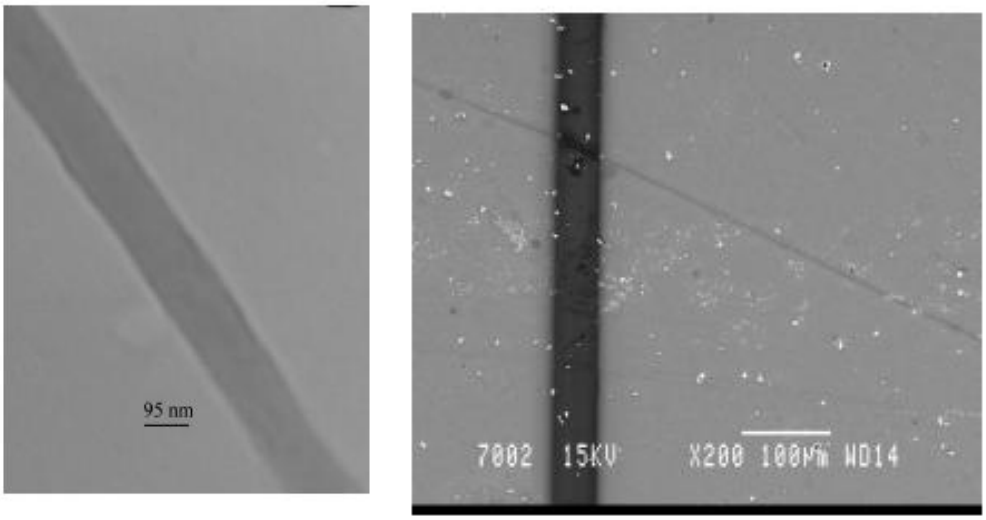

Figure 6. (A) Bright field TEM image of PEO/PPY composite fibre. (B) SEM image of fibre laid across Au electrodes in setup for two probe conductivity measurement (adapted from [17]).

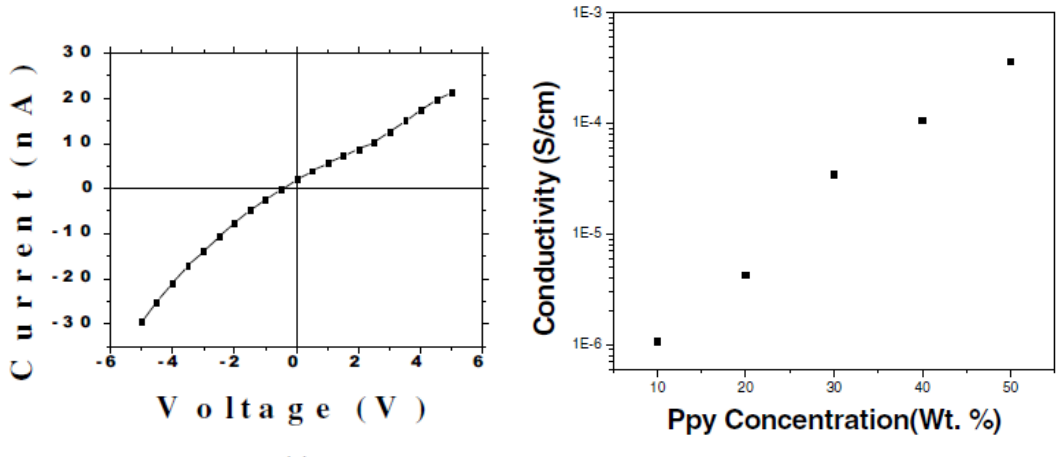

Figure 7. (A) Typical current vs potential curve obtained for PEO/PPy composite fibre for 50 wt % PPy in PEO (Ohmic response). (B) Variation of single fibre conductivity with weight % PPy (adapted from [17]).

The work reported by Zhang and co-workers [19] provides another elegant example of state of art. These workers reported four probe charge transport measurements on individual vertically aligned carbon nanofibres. The experimental setup and typical current vs voltage curves are outlined in figure

8. Typical linear Ohmic behavior is observed. Typical average VACNF resistivity was 4.2 x $10^{-3}\Omega$ cm, a value which lies in between that of graphite parallel to the basal plane (4 x 10^{-5} Ω cm) and perpendicular to the basal plane (4 x 10^{-2} Ω cm) is consistent with a simple model of charge transport where electrons travel mainly from one graphitic plane to another along the length of the nanofibre. It is interesting to note the difference in slope obtained when the two probe and the four probe methods are compared as in figure 8(b). This clearly indicates that the method adopted for resistance measurement, whether two probe or four probe is important.

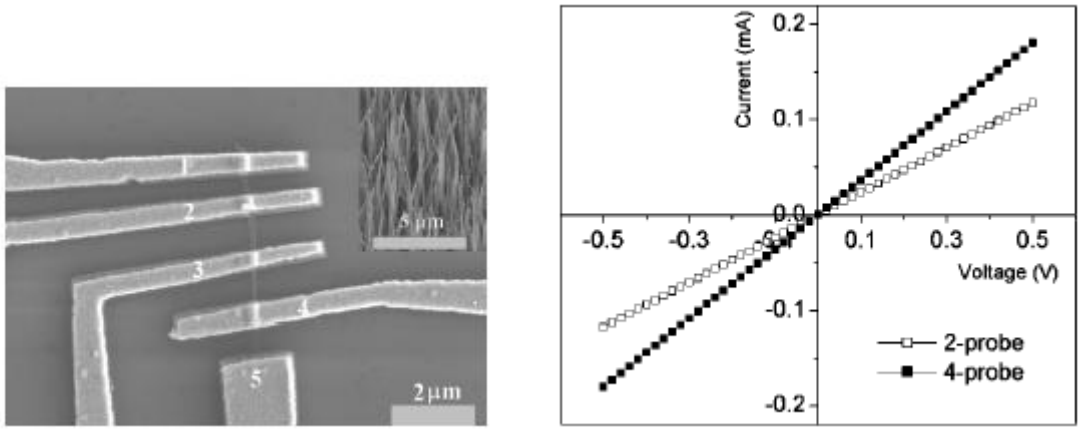

Figure 8. (a) SEM image of a completed vertically aligned carbon nanofibre (VACNF) structure with five metal electrodes (200 nm each of Ti/Au) contacting the nanofibre. Inset is a SEM image of as grown VACNF forest. (b) Typical current/voltage curves obtained from four probe (electrodes 1-4) of the device illustrated in (a) and two probe (electrodes 2,3) measurements on VACNF sample (adapted from [19]).

3. CONCLUSIONS

In this brief paper we have indicated the methodology required to measure the DC conductivity of well defined single fibres formed by a variety of methods. The experiment is far from trivial and requires sophisticated high resolution imaging equipment. Once the connection between the fibre and the injecting/measuring electrodes is set the physical determination of the corresponding current vs voltage response curve is straightforward. The field is still in its infancy and the examples chosen are typical of the results obtained in the literature.

The topic of single fibre conductivity measurement has only begun over the last few years. Much of the work presented in the literature is set at the level of the analysis of current vs potential response curves. This type of dc analysis is all well and good, but more sophistication in the experimental protocol adopted and data analysis employed is clearly needed. One obvious extension is to employ the well established technique of complex impedance spectroscopy [20] to the analysis both of single fibres and arrays of the latter when incorporated into garments. In this type of measurement the electrical impedance of the fibre is measured as a function of frequency over a wide range of values of the latter, and the electrical characteristics of the material characterized in terms of a suitable

electrical equivalent circuit. Indeed the technique could be envisaged as forming the basics for examining the properties and potential of conductive textiles for the electrical transmission of data. Thus the bringing to fruition of this type of development and extension in experimental methodology will assist in the ushering in of the era of truly smart fabrics and wearable computers [21].

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