A simple method to characterize the electrical and mechanical properties of micro-fibres

A Castellanos-Gomez

Kavli Institute of Nanoscience, Delft University of Technology, Lorentzweg 1, 2628 CJ Delft, The Netherlands
E-mail: a.castellanosgomez@tudelft.nl

Received 14 August 2013, in final form 13 September 2013
Published 22 October 2013
Online at stacks.iop.org/EJP/34/1547

Abstract

A procedure to characterize the electrical and mechanical properties of micro-fibres is presented here. As the required equipment can be found in many teaching laboratories, it can be carried out by physics and mechanical/electrical engineering students. The electrical resistivity, mass density and Young’s modulus of carbon micro-fibres have been determined using this procedure, obtaining values in very good agreement with the reference values. Young’s modulus has been obtained by measuring the resonance frequency of carbon fibre-based cantilevers. In this way, one can avoid common approaches based on tensile or bending tests which are difficult to implement for microscale materials. Despite the simplicity of the experiments proposed here, they can be used to trigger in the students interest regarding the electrical and mechanical properties of microscale materials.

(Some figures may appear in colour only in the online journal)

1. Introduction

The characterization of the electrical and mechanical properties of novel materials is a milestone in material science and mechanical and electronic engineering. While for macroscopic materials mechanical and electrical tests are well defined [1–3], when the dimensions of the material are reduced down to the micron-scale, characterization of these properties becomes challenging [4, 5].

Here we present an experimental procedure to characterize the electrical and mechanical properties of carbon micro-fibres that can be carried out in a teaching laboratory by physics and mechanical/electrical engineering students. Although the methodology developed here can be applied to other micron-scale materials, we choose carbon fibre because of the emerging technological interest in it. For instance, carbon micro-fibres are regularly employed as fillers in composite materials to engineer their electrical and mechanical properties. Note that the characterization of Young’s modulus and electrical resistivity of individual micro-fibres is...
crucial to predict the properties of the composite material after adding the filler. Among different micro-fibres, we chose carbon fibres because of the availability in the market of many kinds of carbon fibres with different electronic and mechanical properties. Moreover, carbon fibres are very affordable and despite their small dimensions they can be handled with ease using normal laboratory tweezers. In the following sections, the characterization of the electrical resistivity, mass density and Young’s modulus are described.

2. Electrical characterization

A single carbon fibre is placed lying down straight onto a laboratory glass slide. Electrical contacts are made by means of small droplets of silver-loaded conductive epoxy adhesive. In order to discount for the effect of the contact resistance ($R_C$) in the electrical measurements, four-terminal measurements are typically carried out. In this scheme, a current source is employed to inject current between two outer electrodes while a voltmeter is used to measure the voltage drop between two inner electrodes. An alternative and simpler approach consists of measuring the two-terminal resistance (directly with a multimeter) between contacts separated by an increasing distance. Figure 1 shows the measured two-terminal resistance ($R$) as a function of the distance between electrical contacts ($L$). Both the contact resistance and the electrical resistivity ($\rho_{el}$) can be extracted from the $R$ versus $L$ relationship:

$$ R = (\rho_{el}A^{-1}) \cdot L + R_C, $$

where $A$ is the carbon fibre cross section ($A = \pi r^2 = 38.5 \, \mu m^2$). Therefore, fitting the measured data to a linear relationship, one can determine the contact resistance from the interception with the vertical axis and the electrical resistivity from the slope. The value obtained for the electrical resistivity is $\rho_{el} = (1.3 \pm 0.3) \times 10^{-5} \, \Omega \, m$, while the contact resistance value is about $338 \, \Omega$.

1 Silver-loaded epoxy. Purchased at RS-Online with part number 186–3616.
3. Mechanical characterization

3.1. Mass density

The mass density of carbon fibres can be determined by measuring the mass for a given material volume. Nonetheless, due to the combination of small dimensions of carbon fibres (typically 5–10 μm in diameter) with their low values of mass density (ranging from 1.5 g cm\(^{-3}\) to 2.5 g cm\(^{-3}\)), the mass of a single carbon fibre cannot be easily determined with conventional teaching laboratory equipment. In order to solve this limitation, one can exploit the fact that carbon fibres are usually supplied in bundles composed by thousands of individual fibres whose diameter is well defined and provided by the manufacturer. For instance, the carbon fibre employed in this work was supplied in bundles composed by 12,000 individual fibres 7 μm in diameter. Therefore, one can estimate the mass density by weighting a bundle with length \(L\):

\[
M = (N \cdot \rho A) \cdot L + M_0,
\]

where \(N\) is the number of individual fibres composing the bundle (number provided by the manufacturer), \(\rho\) is the mass density, \(A\) is the cross section of an individual fibre \((A = \pi r^2 = 38.5 \, \mu \text{m}^2)\), \(L\) is the length of the fibre bundle and \(M_0\) is the tare weight of the measurement. A better estimate of the mass density can be obtained by weighting bundles with different lengths and plotting the measured mass as a function of the filament length. To perform the \(M\) versus \(L\) measurement, a fibre bundle ∼130 mm long was employed. One of the free ends was wrapped with adhesive tape to avoid losing individual fibres during the measurement. After weighting the bundle, few millimetres of the bundle are cut with a blade. The new length of the bundle is measured with a calibre and it is weighted again. This process is repeated until several \(M\) versus \(L\) datapoints are obtained. The mass density can be easily determined from the slope of the \(M\) versus \(L\) relationship and the tare weight, discounting the tare weight.

Figure 2 shows the measured mass for a filament of 12,000 carbon fibres with different lengths. The mass follows a linear trend with the filament thickness, as expected from equation (2). From the slope of the \(M\) versus \(L\) relationship, one can determine the mass density \(\rho = 1.71 \pm 0.06 \, \text{g cm}^{-3}\), which is in good agreement with the value provided by the manufacturer, \(\rho = 1.8 \, \text{g cm}^{-3}\).

3.2. Young’s modulus

Young’s modulus measurements on fibres are typically carried out by means of tensile and bending tests. These methods, however, are difficult to implement for micro-fibres in a teaching laboratory. Here we propose to employ a resonant method to determine Young’s modulus of micron-fibres that can be implemented in most physics teaching laboratories. The method consists of the measurement of the resonance frequency of cantilevers made from carbon micro-fibres. For circular cross section cantilevers, the relationship between Young’s modulus \((E)\) and its resonance frequency \((f)\) is given by [6]

\[
E = \frac{(1 - \nu^2) \cdot (4/3) \cdot \rho \cdot (\pi f^2 \cdot L^2 \cdot r^{-1})^2}{},
\]

where \(\nu\) is the Poisson ratio \((\nu = 0.27 [7])\), \(\rho\) is the mass density, \(L\) is the cantilever length and \(r\) is the fibre radius. Therefore, Young’s modulus of the carbon fibres can be obtained by simply measuring the resonance frequency of a carbon fibre cantilever with a certain length. The measurement of the resonance frequency is carried out with the experimental setup depicted in figure 3(a). The carbon fibre cantilever is fabricated by placing a long carbon fibre (few centimetres long) overhanging on a metal block. The fibre is fixed to the block by means of
Figure 2. Measurement of the mass of a carbon fibre bundle composed by 12,000 individual fibres. The bundle has been successively cut to obtain several pairs of mass and length values. The inset indicates how the tare weight can be determined from the interception with the vertical axis.

Figure 3. (a) Schematic diagram of the experimental setup employed to measure the resonance frequency of carbon fibre-based cantilevers. (b) Optical microscopy image of the free end of the carbon fibre cantilever in the static and on-resonance situations. (c) Resonance spectrum of a carbon fibre cantilever measured by determining the oscillation amplitude from optical microscopy images acquired while exciting the cantilever at different frequencies.

silver-loaded conductive epoxy. After curing the epoxy, the overhanging part of the fibre is cut to the desired length (typically 1–15 mm) with a blade. Then, the cantilever is driven by the electrostatic force between a metallic plate (typically a blank piece of printed circuit board connected to a signal generator) and the cantilever (grounded). The oscillation of the cantilever can be easily detected by inspecting the free end of the cantilever under an optical microscope. Figure 3(b) shows an optical microscopic image of the free end of a carbon fibre cantilever. When the cantilever is excited at its resonance frequency, the free end of the cantilever becomes blurred and the oscillation amplitude can be obtained directly from the image. Moreover, using stroboscopic illumination, one can capture the oscillation of the carbon fibre cantilever with
A simple method to characterize the electrical and mechanical properties of micro-fibres

Figure 4. Relationship between the resonance frequency of the carbon fibre cantilevers and their length. (Inset) Same relationship but represented on a linear scale.

Young’s modulus of carbon micro-fibres can be determined with higher accuracy by measuring the resonance frequency on several cantilevers fabricated with different lengths. We have measured the resonance frequency of three carbon fibre cantilevers. After measuring the resonance frequency, the cantilevers have been cut with a blade, the new length has been determined by means of an optical microscopy image and the new resonance frequency has been measured. This process has been carried out with each of the cantilevers to increase the statistics. Figure 4 shows the resulting measured resonance frequencies for cantilevers made from different carbon fibres (datapoints with different colours), reducing their length in steps. Young’s modulus obtained from equation (3) and the experimental $f$ versus $L^{-2}$ relationship is $E = 246 \pm 8$ GPa.

4. Conclusions

In summary, we presented a simple approach to characterize the electrical and mechanical properties of micro-fibres that can be carried out by physics undergraduate students in most teaching laboratories. We applied this procedure to study carbon fibres obtaining values of the electrical resistivity, mass density and Young’s modulus very close to the reference values.

5. Materials and methods

The carbon fibres used in this work are polyacrylonitrile (PAN), manufactured by Hercules Inc. with part number AS4–12K. The fibres are supplied in a bundle of 12,000 individual carbon fibres. The diameter of the individual fibres is $7 \mu m$. These kinds of carbon fibre bundles are
Table 1. Physical properties of the carbon fibres employed in this work. The value with (*) has not been provided by the manufacturer and approximate values based on similar carbon fibres have been used as an estimate.

| Property                  | Value (reference) | Value (this work) |
|---------------------------|-------------------|-------------------|
| Electrical resistivity ($\rho_{el}$) | $1.4 \times 10^{-5} - 1.6 \times 10^{-5}$ $\Omega\text{m}$ $^*$ | $(1.3 \pm 0.3) \times 10^{-5}$ $\Omega\text{m}$ |
| Mass density ($\rho$)      | $1.8 \text{g cm}^{-3}$ | $1.71 \pm 0.06 \text{g cm}^{-3}$ |
| Young’s modulus ($E$)      | $248 \text{GPa}$    | $246 \pm 8 \text{GPa}$    |

typically about 10 € per metre. Table 1 summarizes the physical properties of the carbon fibre used in this work.

The length of the carbon fibre cantilevers and their oscillation amplitude have been determined with an optical microscope (Nikon Eclipse LV-100), previously calibrated by imaging samples of known dimensions (such as the divisions of a calibre or the diameter of the carbon fibre itself). Nonetheless, due to the dimensions of the carbon fibres, one could employ a low-cost digital microscope\(^2\) to perform the characterization presented here.

Acknowledgments

The author would like to thank A Lara-Quintanilla (TU Delft) for carefully proof-reading the paper. AC-G acknowledges financial support through the FP7-Marie Curie Project PIEF-GA-2011-300802 (‘STRENGTHNANO’).

Appendix

An alternative method to determine Young’s modulus of the carbon fibres consists of measuring the resonance frequency of a carbon fibre cantilever before and after loading it with test masses [10]. Before the mass load, the resonance frequency of the cantilever can be written as

$$f_0 = \left(\frac{1}{2\pi}\right) \cdot \left(\frac{k}{m_{\text{eff}}}\right)^{1/2}, \quad (A.1)$$

where $k$ is the spring constant of the cantilever and $m_{\text{eff}}$ is its effective mass. After loading the cantilever with a test mass ($\Delta m$), the resonance frequency changes:

$$f = \left(\frac{1}{2\pi}\right) \cdot \left[\frac{k}{(m_{\text{eff}} + \Delta m)}\right]^{1/2}. \quad (A.2)$$

The spring constant of the cantilever can be extracted from expressions (A.1) and (A.2) as follows:

$$k = \left(\frac{\Delta m/4\pi^2}{\left(f_0^2 - f^2\right)}\right) \left[\left(f_0^2 - f^2\right) / \left(f_0^2 - f^2\right)\right]. \quad (A.3)$$

For a cantilever with circular cross section, the relationship between the spring constant and Young’s modulus is given in [6]:

$$E = 2(1 - \nu^2)(L^3/r^4)k. \quad (A.4)$$

Therefore, using equations (A.3) and (A.4), one can determine the carbon fibre Young modulus in an alternative way.

We have measured the resonance frequency of a 1 mm long carbon fibre cantilever before and after attaching two tin microspheres (see figure A.1). The microspheres are extracted from SN62 MP218 solder paste. The test masses naturally stick to the cantilever due to the presence of a small amount of flux covering them. Nonetheless, the mass attachment procedure can be

\(^2\) www.dino-lite.com/
A simple method to characterize the electrical and mechanical properties of micro-fibres

Figure A1. Optical microscopy image of the free end of a carbon fibre cantilever loaded with two tin microspheres.

Table A1. Values employed for the determination of Young’s modulus following the alternative approach described in the appendix.

| Value                              |                  |
|------------------------------------|------------------|
| Frequency before \((f)\)           | 4550 Hz          |
| Frequency after \((f_0)\)          | 1390 Hz          |
| Cantilever length \((L)\)          | 1 mm             |
| Test masses \((\Delta m)\)         | 0.24 \(\mu\)g   |
| Cantilever spring constant \((k)\)| 0.02 N m\(^{-1}\) |
| Young’s modulus \((E)\)            | 231 ± 18 GPa     |

cumbersome. Once the sphericity of the particles is checked under the optical microscope, their masses are determined by measuring their diameter using the density of the bulk material (8.824 g cm\(^{-3}\)). We have estimated that the flux increases the mass load by less than 1%

Table A1 summarizes the values employed for the calculation of Young’s modulus. The obtained value \(E = 231 ± 18\) GPa is in very good agreement with both the reference value and the value obtained in the analysis described in the main text.

References

[1] Turvey K 1989 Investigation of the frequencies of in-plane modes of a thin circular singly clamped ring with application to Young’s modulus determination *Eur. J. Phys.* 10 111
[2] Rafael M D 2008 Flexural vibration test of a cantilever beam with a force sensor: fast determination of Young’s modulus *Eur. J. Phys.* 29 589
[3] Velasco S, Román F and White J 2010 A simple experiment for measuring bar longitudinal and flexural vibration frequencies *Am. J. Phys.* 78 1429
[4] Acosta J C et al 2011 A tuning fork based wide range mechanical characterization tool with nanorobotic manipulators inside a scanning electron microscope *Rev. Sci. Instrum.* 82 035116
[5] Castellanos-Gomez A et al 2012 Elastic properties of freely suspended MoS\(_2\) nanosheets *Adv. Mater.* 24 772–5
[6] Finot E, Passian A and Thundat T 2008 Measurement of mechanical properties of cantilever shaped materials *Sensors* 8 3497–541
[7] Krucinska I and Spycka T 1991 Direct measurement of the axial Poisson’s ratio of single carbon fibres *Compos. Sci. Technol.* 41 1–12
[8] Castellanos-Gomez A, Agrait N and Rubio-Bollinger G 2009 Dynamics of quartz tuning fork force sensors used in scanning probe microscopy Nanotechnology 20 215502

[9] Castellanos-Gomez A 2013 A simple strobe to study high-order harmonics and multifrequency oscillations in mechanical resonators Eur. J. Phys. 34 1

[10] Cleveland J et al 1993 A nondestructive method for determining the spring constant of cantilevers for scanning force microscopy Rev. Sci. Instrum. 64 403–5