Polímeros
ISSN 1678-5169 (Online)

https://doi.org/10.1590/0104-1428.08720

SHORT COMMUNICATIONS

Accurate measurement of pitch-based carbon fiber electrical resistivity

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Abstract

This study investigated the appropriate methodology required to measure single carbon fibers electrical resistivity. Two- and four-probe methods were evaluated for this measurement. Comparing results for single filaments of pitch-based and PAN-based fibers shows that the two-probe method gives acceptable results for PAN-based fibers, but much higher deviations from adjusted resistivity for pitch-based fibers (>15%). The four-probe method shows small deviations (<1%) for both precursors and is the most suitable for measurements of pitch-based carbon fibers. The four-probe method gives higher accuracy than the two-probe for all samples tested.

Keywords: *carbon fiber, four-probe method, mesophase-pitch, electrical resistivity.*

How to cite: Guimarães, C. J. B., Aguiar, A. P., & Castro, A. T. (2021). Accurate measurement of pitch-based carbon fiber electrical resistivity. *Polímeros: Ciência e Tecnologia, 31*(1), e2021011. https://doi.org/10.1590/0104-1428.08720

1. Introduction

Mesophase pitch-based carbon fibers have higher transport properties than most polymers, because of the mesophase pitch's ability to form highly ordered graphite domains[1-7]. Therefore, they are used as thermal and electrical management materials in applications such as high thermal conductivity radiators[8,9], electronic packaging[10], electromagnetic interference shielding[11], heat storage[9], and radar absorption[12].

Volume resistivity is an important performance indicator for carbon fibers, applied to the evaluation of process parameters along its production steps: spinning[13-15], stabilization[16,17], carbonization[18,19], and graphitization[19-22]. It can also be applied in the evaluation of pre and post-processing steps such as intercalation[23,24], annealing[25], and coating[26-29]. Hence, many researchers use single fiber methods to find the correlation between electrical resistivity and other physical properties[30-32].

First proposed by Wenner[33] in 1915, and adjusted for small, fragile compounds by Coleman[34] in 1975, the four-probe method is commonly used by carbon fiber researchers[14-20,23-28]. However, the carbon fiber resistivity international standard method, ISO 13913, specifies a two-probe measurement[35], and many authors use which[36-42].

Despite being a simple alternative[43], the two-probe method may be sensitive to contact and lead resistances[44] (Figure 1). Some authors recommend this method only when resistance values are high[45] or when accuracy is not required, as it has a known systematic bias (20–800 Ω)[38-41]. Thus, this information suggests the reference standard single carbon-fiber resistivity test method has some limitations, and it could be inadequate for carbon fibers' electrical resistivity measurements with highly ordered graphite domains such as mesophase pitch-based carbon fibers.

To investigate whether the two methods used in literature are suitable, applied the two- and four-probe to measuring PAN- and pitch-based carbon fibers' electrical resistivity and testing the effect of contact resistance through the linear fitting sample resistances for different gauge lengths. The results were compared with the datasheet values and literature reports.

2. Materials and Methods

Standard and high-modulus grades of PAN- and pitch-based carbon fibers were selected (Table 1). For each sample, electrical resistivity measurements for ten single filaments, obtained at room temperature by the two- and four-point methods, were averaged and compared to the manufacturer datasheet values.

Individual filaments were straightened and glued to the specific mounting tab of each method. The two-probe mounting template is a 0.3 mm thickness cardboard, with a 25 mm hole cut out. The four-probe arrangement is a printed circuit board with four parallel copper conduction paths, with the two inner trails separated by 25 mm and the two outer trails by 35 mm. Each carbon monofilament lay on the standard support following a centerline of the mounting template, fixed with a conductive adhesive (Figure 2).

Table 1. Electrical resistivity (ρ_0), Young Modulus (E), and diameter (D) of commercial fibers specification[46-49].

Manufacturer	Name	ρ_0 (μΩ.m)	E (GPa)	D (μm)	Precursor
Cytec	K-1100	1.2	965	10.0	Pitch
Cytec	P-25	13	159	11.0	Pitch
Torayca	M46J	9	436	5.0	PAN
Torayca	T300	17	230	7.0	PAN

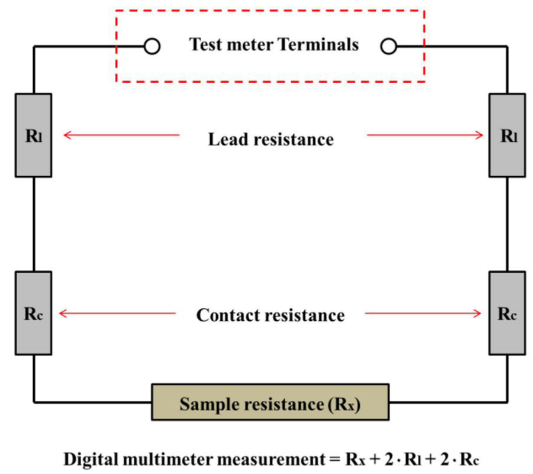

Figure 1. Two-probe method equivalent circuit representation.

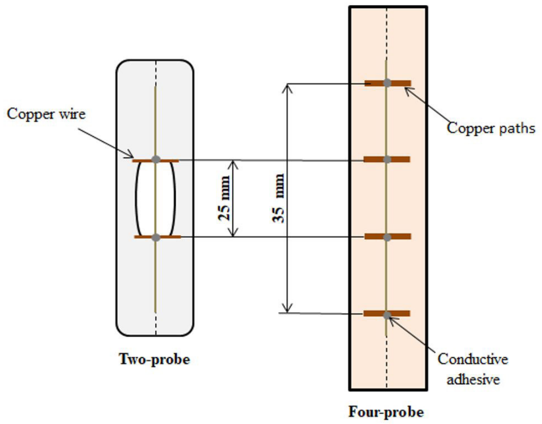

Figure 2. Two and four-probe mounting tab for single filament.

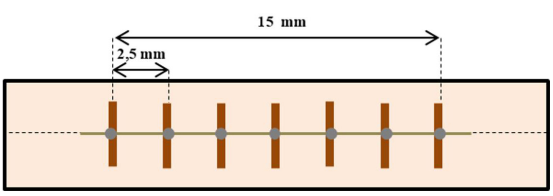

Figure 3. Mounting tab with different gauge length sample for two and four-probe method.

A Mitutoyo CD-6” AX-B digital caliper was used to measure the distances between the two inner points (L) at which the fiber no longer touches the conductive adhesive. An Olympus BX41 confocal microscope was used to measure the diameter (D) at three distinct points along the filament length at 1000x magnification. In the two-probe method, a Fluke 87 V digital multimeter the electrical resistance (R) of individual filaments. Meanwhile, in the four-probe method, the external contacts were connected through a Keithley’s 6221 DC source, and the internal contacts connected through a Keithley’s 2182A high impedance nano voltmeter connected the others. Thermal voltage’s effects were eliminated by reversing the polarity and averaging the two values[50]. Electrical resistance was obtained according to Equation 1.

$$\rho = \frac{\pi D^2}{4L} \cdot R \quad (1)$$

The resistance of each sample was also measured by both methods, at different lengths in the 2–15 mm range (Figure 3) to estimate the contact resistance (R_c) and the adjusted resistivity value (ρ_a). These parameters can be obtained by linearly fitting the resistance (R*) of different gauge lengths (L*), according to Equation 2, assuming that the cross-sectional carbon fiber area (A) and the contact resistance are constant. The adjusted electrical resistivity values were compared to the averages electrical resistivity at the fixed 25 mm distance.

$$R^* = \rho_a \cdot \frac{L^*}{A} + R_c \quad (2)$$

3. Results and Discussions

Table 2 shows the average electrical resistivity ($\bar{\rho}_i$) and relative deviation (D_i) from the manufacturers’ values (ρ_0) of each carbon fiber for both tested methods. For the two-probe method, the electrical resistivity relative deviations of the PAN-based fibers are less than 4%, while the pitch-based deviations exceed 10%. In contrast, all relative deviations for the four-probe method are less than 2%. Besides that, at 95% confidence interval Student’s t-test[51] results in no statistically significant difference between the manufacturers’ values (ρ_0) and the four-probe method electrical resistivity ($\bar{\rho}_{IV}$) (Table 2), since t-values (t_{IV}) modulus are less than the critical t-value (t_{crit} = 2.26[51]). On the other hand, the two-probe showed a significant difference between these values for K-1100 and P-25 fiber since the t-values modulus is higher than the t-critical. These results suggest that pitch-based carbon fiber manufacturers do not follow the single filament method proposed by ISO 13913 international standards.

The four-probe standard deviations are smaller than the two-probe for all samples (Table 2). Besides, the former, by statistical F-test[51], provide better precision at 95% confidence level, since all samples F-value (Table 2) are superior to the critical F-value (F_{crit} = 3.31[51]). These

Table 2. Average electrical resistivity ($\bar{\rho}_i$), manufacturers declared value (ρ_0), standard deviation (σ_i), relative deviation (RD_i) from manufacturers provided value, t and F values.

Name	Two-probes method				Four-probes method				F
	$\bar{\rho}_{II}$ (μΩ.m)	σ_{II} (μΩ.m)	RD_{II} (%)	t_{II}	$\bar{\rho}_{IV}$ (μΩ.m)	σ_{IV} (μΩ.m)	RD_{IV} (%)	t_{IV}	
K-1100	1.4	0.3	16.7	2.30	1.2	0.2	0.0	0.00	3.36
P-25	11.7	1.2	10.1	-3.43	12.8	0.6	1.9	-1.05	4.00
M46J	8.9	0.6	1.1	-0.53	8.9	0.3	1.1	-1.05	4.00
T300	16.4	1.7	3.5	-1.12	16.8	0.8	1.2	-0.79	4.52

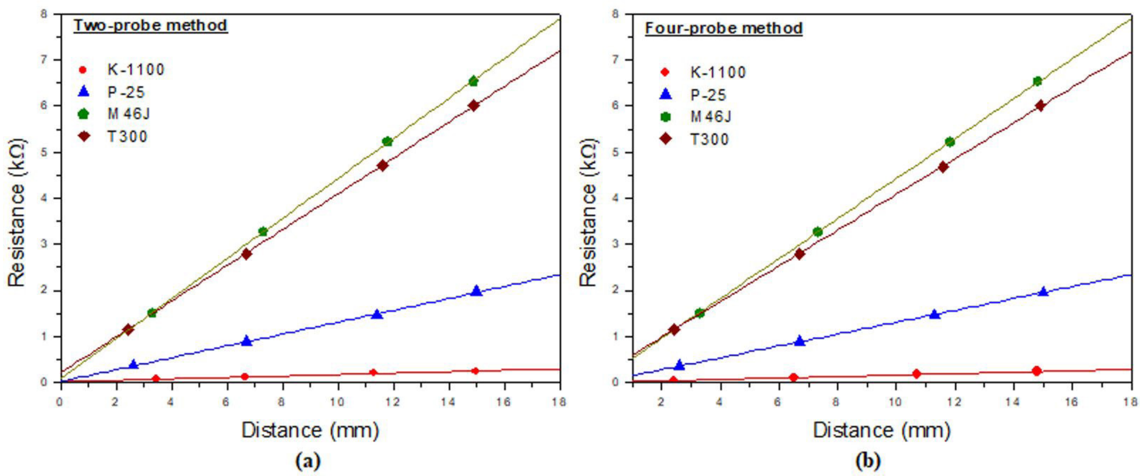

Figure 4. Commercial carbon fibers fit of (a) two-probe and (b) four-probe methods.

results suggest that the four-probe is more accurate than the two-probe method.

Table 3 shows each carbon fiber's literature data electrical resistivity (ρ_L). Comparing these to two- and four-probe the electrical resistivity (ρ_{II} *and* ρ_{IV}) by Student's t-test concludes that is no statistically significant difference in a 95% confidence interval (Table 3) since the t-values (t_{IV}) modulus are less than the critical t-value for all sample. On the other hand, there is a significant difference between literature data and two-probe electrical resistivity values for K-1100 fiber since t-values modulus is higher than the t-critical. This result indicates that the two-probe may not be a suitable method to estimate the pitch-based carbon fibers' electrical properties.

Figure 4 shows the correlations between measured electrical resistance and gauge length, fitted by a straight line, for both methods. Contact resistance is given by the vertical axis intercept, and electrical resistivity by the line slope (Table 4). All correlation coefficients (R^2) were higher than 0.999, representing a good fit.

Contact resistances varied from about 20 – 220 Ω, with the highest values from PAN-based carbon fibers. For these fibers, there was no significant difference between two and four probes contact resistance. However, for pitch-based carbon fibers, the contact resistance obtained by the two-probe method is significantly higher than by the four-probe method. These results confirm the higher accuracy of the four-probe method[44].

For all samples, the adjusted electrical resistivity obtained by the two and four-probe methods were identical.

Table 3. Literature data (ρ_L), standard deviation (σ_i), and t values for two- and four- probe results.

Name	ρ_L (μΩ·m)	t_{II}-value	t_{IV}-value
K-1100	1.17[52]	2.65	0.47
P-25	13.7[53]	-1.84	2.11
M46J	9.3[54]	-1.84	-1.95
T300	16.8[55]	-0.74	0.00

Comparison of average values for the adjusted electrical resistivity (ρ_a) and the electrical resistivity ($\bar{\rho}_i$) obtained by the four-probe method, by Student's t-test[51] show no statistically significant difference in a 95% confidence interval. For the two-probe method, on the other hand, there is a significant difference between these values for K-1100 and P-25 fibers, which are both pitch-based.

Besides having low resistivity, the P-25 and K-1100 fibers' electrical resistances are the lowest because they have the largest diameter (Table 1), so its values are more affected by contact resistance (Table 5). The lowest electrical resistance fiber, K-1100, presented the highest relative deviation from adjusted resistivity, while the highest electrical resistance fiber, M46J, presented the smallest difference. This effect is more prominent in two-probe measurements, which is the method that has higher contact resistances.

The coefficient of variation (Table 5), variability estimator, from pitch-based carbon fibers is higher than PAN-based; this occurs because pitch-based fibers tend to be more heterogeneous than PAN-based[56], which intensifies measurement noise.

Table 4. Contact resistance (R_c), adjusted electrical resistivity (ρ_a), and coefficient of determination (R^2)

Name	Two-probe method			Four-probe method		
	R^{II}_c (Ω)	ρ^{II}_a (μΩ·m)	R^2	R^{IV}_c (Ω)	ρ^{IV}_a (μΩ·m)	R^2
K-1100	17	1.2	0.99936	5	1.2	0.99999
P-25	25	12.9	0.99979	8	13.0	0.99966
M46J	90	8.9	0.99992	92	8.9	0.99973
T300	215	16.7	0.99993	211	16.7	0.99998

Table 5. Average electrical resistance ($\bar{R}$), relatives deviation (RD) from adjusted resistivity, and relative standard deviation (RSD).

Name	$\bar{R}$ (kΩ)	RD_{II} (%)	RD_{IV} (%)	RSD_{II} (%)	RSD_{IV} (%)
K-1100	0.5	16.7	0.8	23	15
P-25	2.8	9.3	0.5	10	5
T300	10.0	2.0	0.4	7	3
M46J	11.4	0.3	0.3	10	5

4. Conclusions

The two-probe method specified by ISO resulted in up to 2% relative deviation from adjusted resistivity for PAN-based fibers and over 15% deviation for pitch-based fibers. On the other hand, the four-probe method achieved less than 1% relative deviation from adjusted resistivity for all tested fibers, producing accurate and consistent results, even when measuring low resistances.

We conclude that the two-probe method is particularly inadequate for determining pitch-based carbon fiber's electrical resistivity due to its inability to measure low electrical resistances accurately. For PAN-based fibers, the two-probe method gives acceptable results, but with lower accuracy than the four-probe method unless its values are corrected by linear fitting of the resistance of different gauge lengths.

5. Acknowledgements

We thank the Brazilian Army Technological Center (Centro Tecnológico do Exército - CTEx) for supporting this work.

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Received: Sept. 29, 2020
Revised: Feb. 26, 2021
Accepted: Mar. 21, 2021