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Measuring the through-plane electrical resistivity of bipolar plates (apparatus and methods)

Nicolas Cunningham *, Michel Lefèvre, Guy Lebrun, Jean-Pol Dodelet

INRS-Energie, Matériaux et Télécommunications, 1650 boulevard Lionel Boulet, Varennes, Qué., Canada J3X-1S2

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Abstract

In this paper, we will describe an apparatus for measuring the through-plane electrical conductivity and also discuss its calibration. This paper describes operating procedures that were used and their effects on conductivity measurements. The following factors were found to affect the measurements accuracy and reproducibility: (i) the method used to polish the copper electrodes, (ii) the nature of the disk used to ensure a good electrical contact between the electrodes and the sample and (iii) whether these disks were reused or not.

A 2 μohm drift was observed in the resistance measurements on a 24-h period. Two calibration methods were studied. The first calibration method was developed to calibrate the conductivity apparatus using power resistors and we were able to determine that a systematic error of 60 μohm was present. A second calibration method was then used to measure the electrical conductivity of two Poco graphite samples. Using our apparatus, the electrical conductivity of AXF-5Q and DFP-2 Poco graphite samples were both (60–210 μohm cm) lower than their reported statistical value of 1470 and 1500 μohm cm, respectively.

Keywords: DC conductivity; Poco graphite conductivity; PEM fuel cells

1. Introduction

In order to move towards commercialization, fuel cell components must become cheaper and easier to produce. Replacing machined graphite bipolar plates by molded composites (C-polymers) has been extensively studied and there is a large amount of patents on this subject. As Steele and Heinzel [1] pointed out, C-polymer composites have generally a lower conductivity than isotropic high-quality graphite (from Poco Inc., for example). A through-plane sheet resistance of less than 0.01 ohm cm^2 has been referred to by many authors [2,3] to minimize resistive losses in a stack. If one would like an idea of what this number actually means, the calculation could be based on a current density between 1 and 2 A cm^{-2} and a surface of between 200 and 400 cm^2 which are typical values characterizing industry fuel cell stacks prototypes. Using these numbers, the voltage drop per plate would range from 10 to 20 mV. This can be compared to a voltage drop of 50 mV/cell for a well-humidified Nafion® membrane (100 μm thick operating at 1 A cm^{-2})[2,4].

The through-plane sheet resistance ($R_{z\ \mathrm{bulk}}$) expressed in ohm cm^2 [5] is, therefore, of importance for engineers working with fuel cells, allowing them to easily calculate power losses in a stack of known dimensions. The electrical through-plane sheet resistance can be expressed using Eq. (1) where $\rho_{z\ \mathrm{bulk}}$ is the through-plane electrical resistivity and L the thickness of the plate

$$R_{z\,\mathrm{bulk}} = \rho_{z\,\mathrm{bulk}} L \qquad (1)$$

We know that the resistance of a material of thickness L and area A is given by the following equation:

$$R_{\mathrm{Material}} = \frac{\rho_{z\mathrm{bulk}} L}{A} \qquad (2)$$

* Corresponding author. Present address: Royal Military College of Canada, Department of Chemistry and Chemical Engineering, P.O. Box 17000 Stn Forces, Kingston, Ont., Canada K7K-7B4.
E-mail address: nicolas.cunningham@rmc.ca (N. Cunningham).

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Nomenclature

$R_{z\ \text{bulk}}$ through-plane sheet resistance (ohm cm^2)
$\rho_{z\ \text{bulk}}$ through-plane electrical resistivity (ohm cm)
R_{Material} resistance of a block of a given material (ohm)
R_{Meas} measured resistance (contains R_{Material} and R_{System}) (ohm)
R_{System} system resistance (contains R_{Inst} and $R_{\text{Interfaces}}$) (ohm)
R_{Inst} systematic error caused by instruments (ohm)
$R_{\text{Interfaces}}$ interfacial contact resistances + intrinsic resistances of carbon paper or indium disks (ohm)
R_{Plate} total contact resistance caused by one bipolar separator plate (can also be one plate sandwiched between two electrodes) (ohm cm^2)
$R_{\text{Set-up1}}$ resistance of one carbon paper disk (1 in. diameter) (ohm)
$R_{\text{Set-up2}}$ resistance of a resistor sandwiched between two carbon paper disks (ohm)
$R_{\text{System method 1}}$ system resistance obtained using the first calibration method (ohm)
$R_{\text{Cu–CP}}$ resistance caused by an interface copper–carbon paper (ohm)
$R_{\text{System method 2}}$ system resistance obtained using the second calibration method (ohm)
$R_{\text{Cu–In}}$ resistance caused by an interface copper–indium (ohm)
$R_{\text{CP–Poco}}$ resistance caused by an interface carbon paper–Poco graphite (ohm)
$R_{\text{CP–CP}}$ resistance caused by an interface Poco graphite–Poco graphite (ohm)
R_{Resis} resistance of the resistor measured using the press (ohm)

During a standard experiment, the following equation is always true:

$$R_{\text{Meas}} = \frac{V_{\text{Meas}}}{I_{\text{Meas}}} = R_{\text{Material}} + R_{\text{System}} \tag{3}$$

where V_{Meas} and I_{Meas} are the voltage and current measured. Unfortunately, the measured resistance is not only caused by the sample (R_{Material}) but can also originate from contact resistances between various components of the system and from systematic measurements errors. R_{System} will, therefore, be dependent on the type of materials used; for example whether a carbon paper or an indium disk is used to ensure a good electrical contact between the electrode and the sample. A general equation can be developed for R_{System} and is given underneath:

$$R_{\text{System}} = R_{\text{Inst}} + R_{\text{Interfaces}} \tag{4}$$

where R_{Inst} is the systematic error caused by the instruments (voltage and current values) and $R_{\text{Interfaces}}$ encompasses all the interfacial resistances and the intrinsic resistances of the carbon paper disks for example.

Some researchers have used Eq. (5) [6] to characterize the electrical conductivity of their bipolar plates.

$$R_{\text{Plate}} = \frac{V_{\text{Meas}} A_{\text{Plate}}}{I_{\text{Meas}}} \tag{5}$$

In this case, it becomes impossible to distinguish the resistance caused by the system (R_{System}) from the bulk resistance of the measured material. R_{Plate} is, nonetheless, useful because it is representative of the resistance that would occur in a fuel cell stack. For those interested in measuring $R_{z\ \text{bulk}}$ precisely, a method must be developed to be able to isolate the bulk resistance. Researchers at NREL [6,7] have described a method to correct the measured resistance, which allowed them to measure the value of the resistance cause by a stainless steel carbon paper interface. This work is an attempt to go one step further by testing and fully characterizing a four-point-probe apparatus to measure the through-plane electrical conductivity. Two methods will be employed to isolate the bulk resistance. Special attention will be devoted to the accuracy of the system and to the effect of the operating procedures and methods used.

2. Experimental

2.1. Apparatus

The apparatus built in our lab is a computer-controlled pneumatic press. The air going into the 10.16 cm (4 in.) Bimba cylinder piston is supplied by a Marsh Bellofram type T3000 electropneumatic controller that can deliver compressed air up to 689.5 kPa (100 psi). The applied force which was up to 5000 N is measured by a load cell, model Rice Lake RLS 1000 (4000 lbs full scale), and using 2.54 cm (1 in.) copper cylinder to apply the required pressure. The voltage drop between the two copper cylinders is measured using a Keithley model 2700 multimeter, while the current is supplied by a Kepco power supply model BOP 100-1M. Alumina disks were used to electrically isolate the copper electrodes from the rest of the apparatus.

Fig. 1A shows a picture of the press and the power supply, while Figs. 1B and C show the copper electrodes during sample measurements. On Fig. 1C, the sample (black area between the two copper electrodes) is visible because the upper Teflon® ring has been retracted. The latter can be moved up and down to greatly facilitate the alignment of the sample and the carbon paper. Fig. 2 presents the engineering drawings of the copper electrodes.

Fig. 3 presents the engineering drawings of the capped resistors that have been mounted in such a way as to be able to fit in the press. The resistances were mounted in a hollow

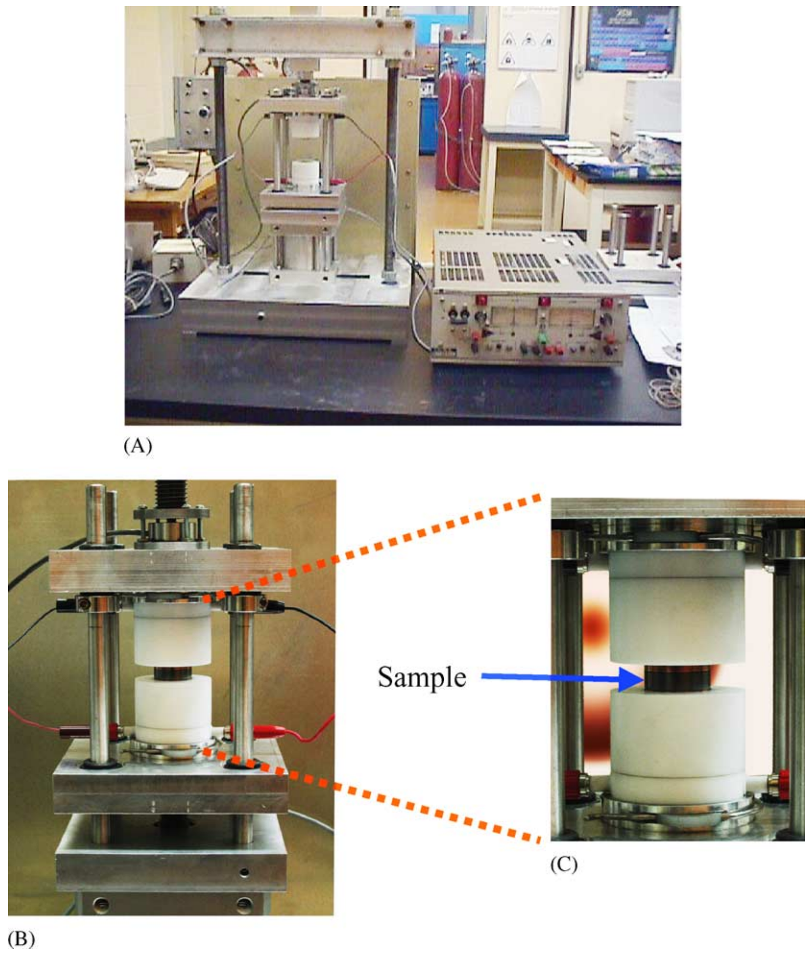

Fig. 1. Pneumatic press with copper blocks.

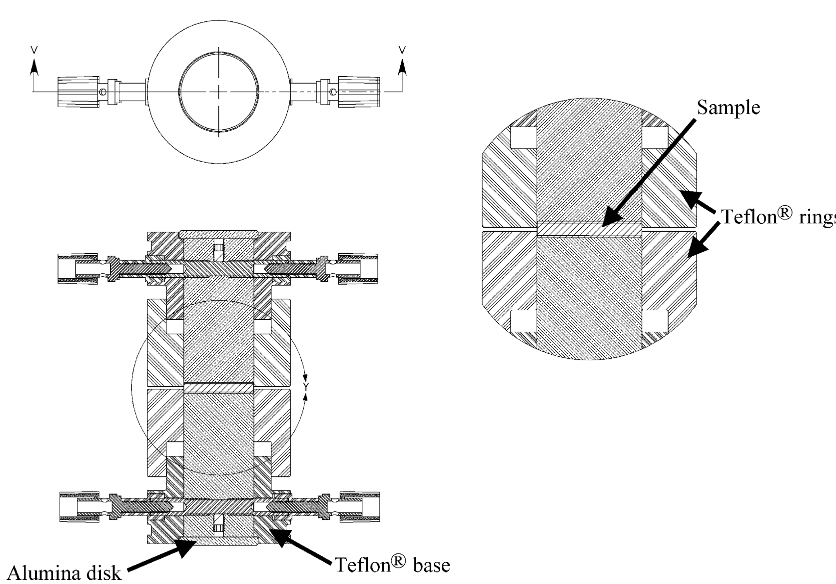

Fig. 2. Drawing showing the electrode assembly with the sliding rings.

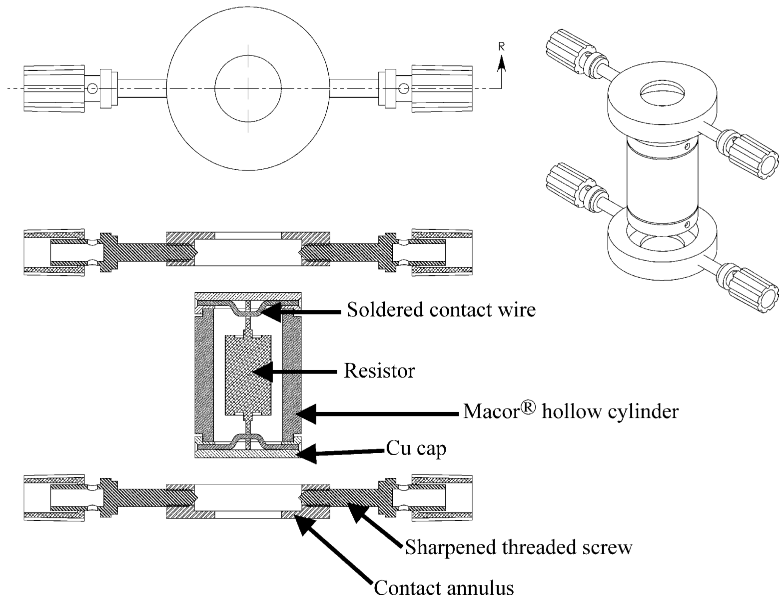

Fig. 3. Drawings of the capped resistors with the two parts needed to calibrate the resistors.

Macor® cylinder and capped with two copper disks. The resistances ranging from 0.05 to 1 ohm were soldered to the copper disks before they were glued unto the hollow cylinders. The two additional parts that are shown on Fig. 3 are needed to calibrate the resistors without having to place them in the press. On each part, two standard electrical connectors mounted on the contact annulus are able to make contact with the capped cylinder using threaded screws. The screws were sharpened to ensure a good electrical contact with the capped cylinder and thus insuring proper calibration.

2.2. Materials

AXF-5Q and DFP-2 graphite blocks from Poco graphite were machined to form disks (about 2.54 cm o.d.). The electrical resistivity given by Poco for these two materials is 1.475 and 1.5 mohm cm, respectively. Two disks made of plain carbon paper grade 2050 from Spectracorp or Indium (99.99%) from Indium Corporation of America were used to ensure good electrical contact between the two copper electrodes and the measured sample.

2.3. Experimental method

The calibration of the resistors was carried out using the sharpened screws (see Fig. 3) to ensure a good electrical contact between the wire and the resistor. The slope of the graph presenting the voltage drop versus the applied current was used to calculate the value of resistors. This procedure was repeated at least five times during a 24-h period to assess the stability of the resistors and of the equipment. In this experiment, the electrical contact was insured by the two contact annuluses with the sharpened screws, the resistors were therefore not placed in the press.

For the experiments requiring the pneumatic press, all copper surfaces are first polished using a diamond paste (typically 1 μm) from Bueler. Then, disks of 2.54 cm diameter (1 in.) are punched in carbon paper or indium. Using the sliding Teflon® ring, the sample and/or disks (carbon paper or indium) are aligned and compressed using a force of around 600 N. The press is then switched to automatic mode where it records the voltage drop and current. Fig. 4 presents the evolution of the voltage drop during a typical test. At the end of each pressure step, the current was varied between 0 and 1 A and the voltage drop recorded. The resulting variation on the voltage drop can be better observed in the insert on Fig. 4. The value of the resistance caused by the sample and/or disks can be obtained using the slope of the voltage drop ver-

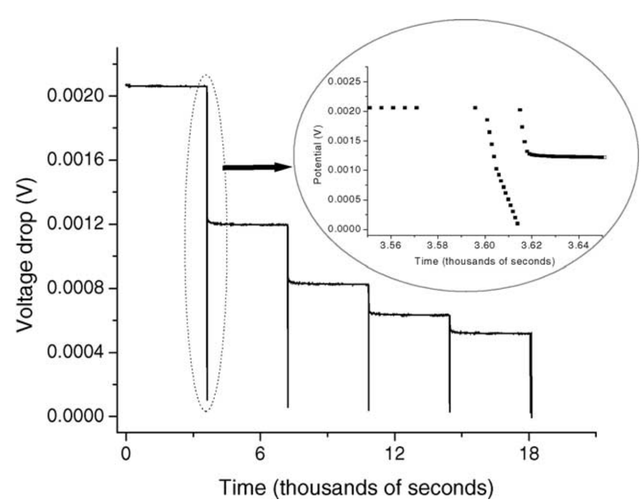

Fig. 4. Evolution of the voltage drop during a typical test with the insert showing the end of the first pressure step at around 3600 s, and the measurement period (between 3600 and 3615 s) during which the current was varied between 1 and 0 A.

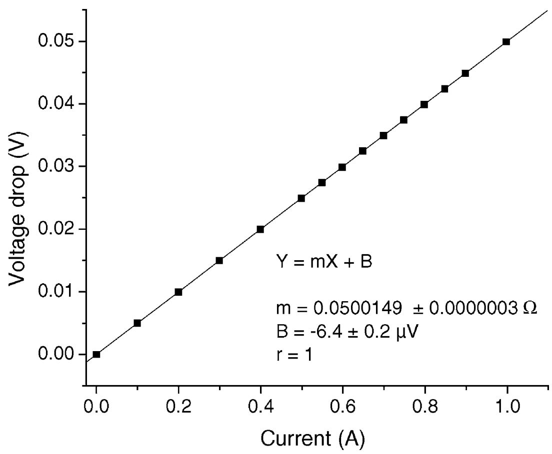

Fig. 5. Voltage drop across a nominal resistance of 0.05 ohm when the electrical contacts are made using machined screws. *m*, *B* and *r* are, respectively, the slope, intercept and correlation coefficient of the slope.

sus the applied current. An example of a slope is given in Fig. 5.

Two calibration methods were used to try to isolate the value of the bulk and that of the system resistance from one another. The first one was of particular use for the resistor's measurements. Fig. 6 presents the two set-ups used for the first calibration procedure. In Set-up 1, the resistance of one carbon paper is measured between two copper electrodes while in Set-up 2, carbon paper disks are placed between the copper electrodes and the top and bottom of the resistor. Since the resistors are capped with copper, the carbon paper disks are therefore pressed between two copper surfaces. This is similar to what was measured in Set-up 1 (see Fig. 6) were the carbon paper disk was placed between the two electrodes. Assuming that all copper surfaces were polished to produce the same surface finish, the value of the resistor could be extracted easily. The value of the resistor was calculated by removing twice the resistance of Set-up 1 from the total resistance of Set-up 2. The same procedure was repeated using indium disks instead of carbon paper disks. Eq. (6) summarizes the first calibration method:

$$R_{\text{Material}} = R_{\text{Set-up2}} - 2R_{\text{Set-up1}} \qquad (6)$$

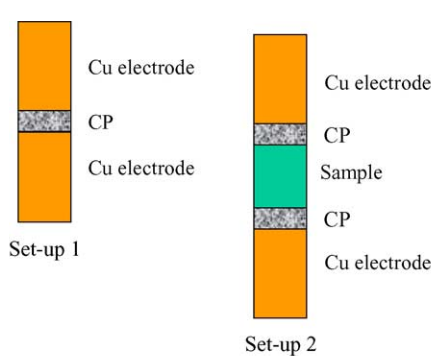

Fig. 6. Drawing showing the two set-ups used: Set-up 1 with only one carbon paper (CP) and Set-up 2 with the sample sandwiched between two pieces of carbon paper.

In this particular case, $2R_{\text{Set-up 1}}$ is taken to be equal to $R_{\text{System method 1}}$ which can be further developed to give the following equation:

$$R_{\text{System method1}} = 2\left(R_{\text{Inst}} + 2R_{\text{Cu–CP}} + \frac{\rho_{\text{CP}}L_{\text{CP}}}{A_{\text{CP}}}\right) \qquad (7)$$

$R_{\text{Cu–CP}}$ represents the resistance caused by the two contacts between each carbon paper (CP) and the copper electrode (Cu) and ρ_{CP}, L_{CP} and A_{CP} are, respectively, the through-plane resistivity, thickness and area of the carbon paper disk. Since we are removing twice R_{Inst}, any systematic error will need to be corrected for since it appears only once in the measurements made using Set-up 2.

The second calibration method consisted in measuring the resistance of AXF-5Q or DFP-2 cylinders of different thicknesses. It was then possible to plot the resistance of the different cylinders in function of their thicknesses. The resistance was represented by the slope of the graph while the system resistance was extrapolated from its intercept. This calibration method is very useful when the thickness of the material measured can be varied easily. Eq. (8) is representative of the second calibration technique.

$$R_{\text{Meas}} = \frac{\rho_{\text{Poco}}L_{\text{Poco}}}{A_{\text{Poco}}} + R_{\text{System method2}} \qquad (8)$$

where ρ_{Poco}, L_{Poco} and A_{Poco} are, respectively, the through-plane resistivity, thickness and area of the Poco graphite sample. $R_{\text{System method 2}}$ is given by Eq. (9):

$$R_{\text{System method2}} = R_{\text{Inst}} + 2\left(R_{\text{Cu–CP}} + R_{\text{CP–Poco}} + \frac{\rho_{\text{CP}}L_{\text{CP}}}{A_{\text{CP}}}\right) \qquad (9)$$

where $R_{\text{CP–Poco}}$ is the resistance caused by the interface between the Poco graphite sample and the carbon paper.

3. Results and discussion

3.1. Measurements reproducibility and accuracy

In order to properly calibrate the press, the first step was to determine the resistance of various resistors (see Fig. 3). Fig. 5 presents the voltage drop measured for the 0.05 ohm nominal resistance versus the applied current when the electrical contact was made using the sharpened screws. It is obvious that the linear fit is nearly perfect and that we will be able to rely on the calibration of the resistors. This procedure was repeated five times for each resistor to assess the reproducibility of the set-up and the results are presented in Table 1. The error on each resistor was calculated to be from 2 to 5 μohm, which is about 10 times more than the error on a single resistor measurement (see the linear fit in Fig. 5). We will compare the value of the error on the resistor calibration with other errors later on in this study; we can already anticipate that it will not be significant.

Table 1
Measured values of two resistances (average of five tests) and their absolute and relative error

Nominal value (ohm)	Measured value (ohm)	Error (ohm)	Error (%)
0.05	0.050013	0.000002	0.004
0.10	0.099775	0.000002	0.002
0.50	0.497581	0.000005	0.004

The accuracy of the resistance measurements using the pneumatic press was carried out next. However, before going any further, a few factors that could influence resistance measurements were assessed. A first set of experiments is presented on Fig. 7 and was carried out to study the effects of the copper electrode surface finish. The curves labeled 1 μm (A) are the highest curve measured and 1 μm (B) the lowest using electrodes polished using a 1 μm diamond paste. It should also be noted that polishing the electrodes using a paste with different particle size abrasive may not yield the same results. This can be observed on Fig. 7 where the curve labeled mirror polish was obtained with electrodes polished using a 0–0.1 μm diamond paste. The voltage drop values obtained with the paste containing finer particles was higher than when 1 μm particles were used. We did not assess the effects of polishing using other particles sizes or compounds so we cannot give more explanations on this phenomenon.

At 5000 N, the value of the measured voltage drop is nearly identical for both tests (A and B) using electrodes polished using the 1 μm paste, even if it was not when lower forces were applied. In the case of the experiment labeled not polished, it was carried out right after experiment labeled 1 μm (A) without re-polishing the electrodes. Even when 5000 N is applied, its voltage drop is always higher than the voltage drop for polished electrodes. When indium disks replaced carbon paper disks, the same type of results is obtained. In the end, it was decided to polish each electrode using the 1 μm diamond paste to minimize the voltage drop and insure reproducibility.

A similar set of experiment was carried out to assess the effect of reusing carbon paper and indium disks. As stated above, the copper electrodes were always polished before each test. No trends were detected in the results when the disks were reused for up to five times. The voltage drop measured could increase or decrease varying from test to test. A slight decrease in the reproducibility of the measurements

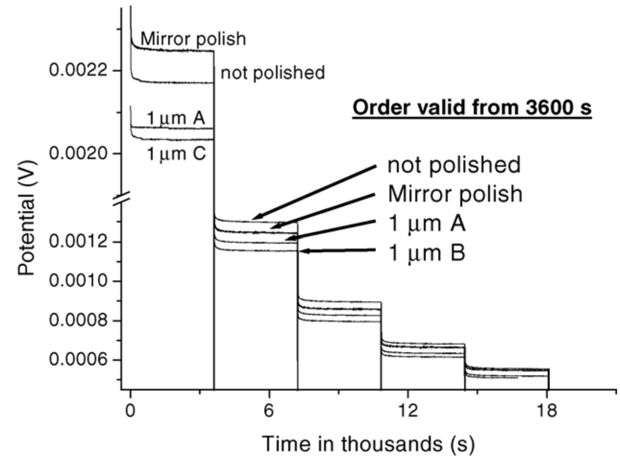

Fig. 7. Effect of the surface finish on the value of the voltage drop.

was detected but not enough to warrant a detailed analysis of the results. Nonetheless, it was decided, as a preventative measure, to use only new carbon paper or indium disks and that principally to lower the number variables that could potentially affect the measurements.

3.2. System resistance determination using the first calibration method

To obtain the necessary value to calibrate the system using the first method described above, the value of the resistances caused by placing one carbon paper or indium disks (see Fig. 6 Set-up 1) had to be precisely determined. Table 2 presents the average of at least three measurements with their absolute and relative errors. At first glance, both materials appear to produce less resistance as the pressure increases which is expected since the four contact resistances ($R_{Cu–CP}$ or $R_{Cu–In}$) will be lower when higher forces are applied. It can also be added that the value of the resistance caused by indium is around one order of magnitude lower than the resistance caused by carbon paper. We can then try to isolate the percentage of the measured resistance that is caused by the bulk resistance of the carbon paper or indium disk.

The accepted values for the through-plane resistivity of the carbon paper and indium are, respectively, 0.07 [8] and 84×10^{-6} ohm cm [9]. Using these resistivities and the dimensions of the disks, we can calculate the bulk resistance

Table 2
Values of the resistance caused by one disk of carbon paper or indium (see Fig. 5 Set-up 1)

Applied force (N)	Carbon paper			Indium		
	R (ohm)	Error[a] (ohm)	Error (%)	R (ohm)	Error[b] (ohm)	Error (%)
875	4.9×10^{-4}	2×10^{-5}	4.6	2.9×10^{-5}	5×10^{-6}	17
1850	3.5×10^{-4}	1×10^{-5}	4.0	2.6×10^{-5}	4×10^{-6}	16
2950	2.9×10^{-4}	1×10^{-5}	5.0	2.5×10^{-5}	3×10^{-6}	11
3900	2.5×10^{-4}	1×10^{-5}	5.6	2.4×10^{-5}	2×10^{-6}	10
4950	2.2×10^{-4}	1×10^{-5}	5.6	2.4×10^{-5}	3×10^{-6}	11

[a] Measurements made using carbon paper are limited by the reproducibility of the carbon paper itself.
[b] Measurements made using indium are limited by the apparatus (2×10^{-6} ohm for a 24-h period).

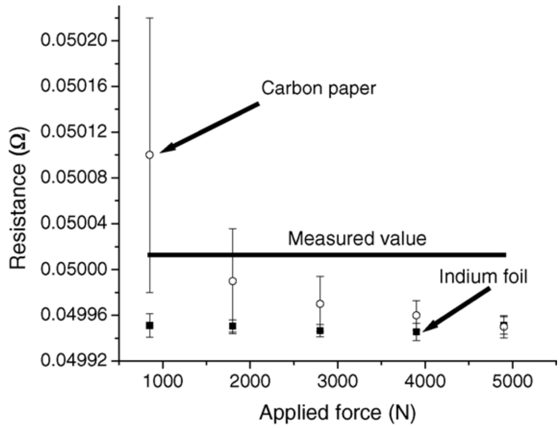

Fig. 8. Corrected value of the resistance measured for the 0.05 ohm (nominal) resistor.

caused by the disks. The results are astonishing since the bulk resistance of carbon paper and indium disk are, respectively, of 2.95×10^{-4} and 4.22×10^{-10} ohm. This means that the value of the resistance measured for the indium disk comes entirely from the contact resistance between the indium and copper. While the bulk resistance of indium is clearly insignificant, the same cannot be said for the carbon paper. The value of the bulk resistance using the uncompressed thickness and resistivity of carbon paper is higher than the measured resistance when the applied force is above 3900 N. However, during the course of a test, the carbon paper is compressed. This could affect its resitivity and reduce its thickness, which may explain why a lower resistance is measured.

One could attempt to measure the resistance of two carbon paper disks one on top of each other to obtain more information. Measurements using two pieces of carbon paper yield a resistance of 3.4×10^{-4} and 3.0×10^{-4} ohm, respectively, for applied forces of 3900 and 4950 N. This is an increase of around $0.8–0.9 \times 10^{-4}$ ohm over the resistance value obtained when only one carbon paper was measured. This increase in the resistance contains the bulk resistance of one compressed carbon paper plus the value of the contact resistance between one carbon paper and another ($R_{\text{CP–CP}}$). This shows that we cannot isolate the bulk resistance of a compressed carbon paper.

Next, the value of the resistance caused by a capped resistor sandwiched between two carbon paper disks was measured at five different pressures using the procedure described in Section 2. This was repeated three times for the two resistors calibrated earlier in this study (see Table 1) using either carbon paper disks or indium disks. The value of R_{Mat} (in this case the material is the resistor) calculated using Eq. (6) is presented on Fig. 8 where the measured value of the resistor (R_{Resis}) was added in the form of a straight line. The error on the value of the measured resistance is roughly represented by the thickness of the straight line on Fig. 8. The value calculated for the resistor is almost constant at (0.04995 ohm) when indium disks are used. The difference between the calibrated value of the resistor (0.050013 ohm) and the measured value is, therefore, of roughly 60 μohm for indium. When carbon paper disks are used, the accepted value falls within the error bars for applied forces of under 1800 N. When higher forces are applied, the value appears to converge to the value measured using indium disks (0.04995 ohm). Furthermore, when the nominal resistor of 0.1 ohm was used, similar curves were observed and again the offset between the measured value and the calibrated value was around 60 μohm for an applied force of 5000 N.

This offset of 60 μohm can be explained because as mentioned earlier, we are in fact removing twice the systematic instrumentation error. This can be more easily appreciated by developing Eq. (6), which gives:

$$R_{\text{Material}} = R_{\text{Resis}} + R_{\text{Inst}} + 2\left((2R_{\text{Cu–CP}}) + \frac{\rho_{\text{CP}} L_{\text{CP}}}{A_{\text{CP}}}\right) - 2\left(R_{\text{Inst}} + (2R_{\text{Cu–CP}}) + \frac{\rho_{\text{CP}} L_{\text{CP}}}{A_{\text{CP}}}\right) = R_{\text{Resis}} - R_{\text{Inst}}$$

This would mean that the systematic instrumentation error is in fact this offset of nearly 60 μohm. However, compared to the resistance of a typical fuel cell bipolar plate, this value is most likely insignificant. It also shows that the error (2–5 μohm) that was measured on the calibration of the resistors and their stability through a 24-h period is 30 times lower than the systematic error. Therefore, our assumption to neglect it was justified. On a side note, the bulk resistance of the carbon paper, calculated using uncompressed properties, is now below or roughly equal to the value of the measured resistance if the later is corrected for the systematic error. Such error could technically be caused by thermoelectric EMFs [10] but in our system, the voltage drop difference measured by reversing the polarity of the current source was under the 2 μV level, which is much below the offset measured. The influence of reversing the polarity of the current source in our system is inside the reproducibility that was obtained in a 24-h period. It can therefore be neglected. It might not be the case for all measurements systems and one may have to apply the proper corrections that are well explained in several Keithley Instrument's publications [10,11].

3.3. Second calibration method results and Poco graphite resistivity measurements

The first calibration method reaches its limits when the materials to be measured are not made of copper. When composites are measured, the calibration using the copper capped resistors cannot be used and one would need to find a way to obtain resistors that would be capped with the materials that is to be measured. The second calibration method is not restricted in that way. R_{System} is obtained by varying the thickness of the analyzed material, which means that one can isolate it as long as sample of different thicknesses are available. Furthermore, this second calibration method is not affected by systematic error since the slope is used in the calculations. Using carbon paper disks, the resistance of

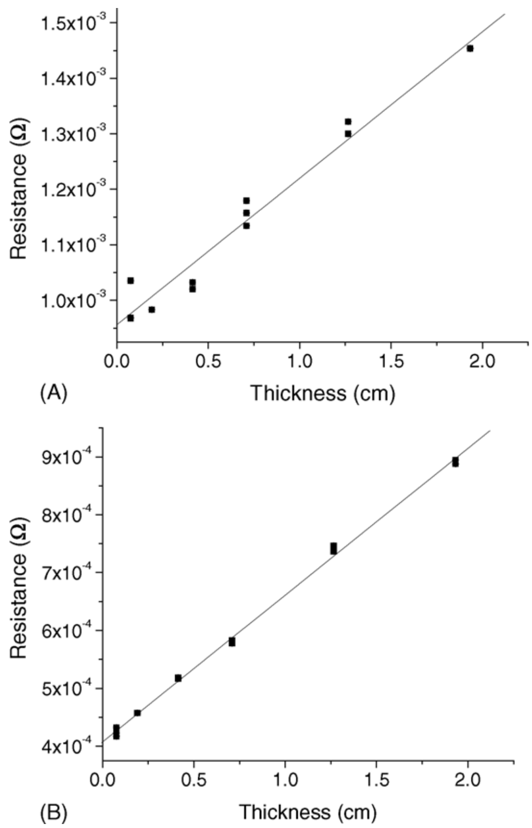

Fig. 9. Resistance of Poco graphite cylinders vs. their thicknesses at a pressure of (A) 875 and (B) 4950 N.

Poco graphite cylinders, from 0.0756 to 1.73 cm thick, and of DFP-2 (1500 μohm cm) or AXF-5Q (1470 μohm cm[12]) was measured. As described earlier in this paper, the resistance of the cylinders was plotted in function of the thickness and that for five different forces. The curves for two applied forces, 875 and 4950 N are, respectively, plotted on Fig. 9A and B. It is obvious that the linear fit on the data of Fig. 9A will be less accurate than the fit of the data on Fig. 9B. The intercept, slope and correlation coefficient of the graphs for each pressure can be found in Table 3. The numerical values of Table 3 confirm our observations since the errors on the intercept and slope decrease and correlation coefficient increase when the applied force is higher.

What is more disturbing is the fact that the value of the resistivity obtained for the Poco graphite is lower than its reported value. Our measured resistivity for Poco graphite is at most 1.34×10^{-3} ohm cm (or 1340 μohm cm) and this is obtained at the lowest force applied (875 N) when the error is the highest at 7×10^{-5}ohm cm. The measured resitivity is even lower at $1.28 \times 10^{-3} \pm 2 \times 10^{-5}$ ohm cm when the force applied reaches 4950 N. Since the slope is not affected by systematic instrumentation error, no correction needs to be applied. An error in the hundreds of μohm cm is not easy to explain. However, it can be advanced that for the thinner cylinders (0.0756 cm), the error made in measuring both their thickness and resistance is higher than when thicker cylinders are measured. This can be observed on Fig. 9A and B where the spread of the data point appears to be more important for the thinner cylinders. This may partially explain the error that was made in measuring the resitivity of Poco graphite DFP-2 because many cylinders used for the calibration were quite thin. There are actual limitations as to the sample thickness that the press can handle which is roughly 10 cm. Furthermore, when one tries to measure very thick cylinders, aligning the carbon paper disks, graphite and electrodes becomes problematic. The sliding ring can no longer be used to align everything and the reproducibility cannot be maintained. This in effect limits the thickness that one can measure using this apparatus to about 2 cm. So improving the results presented on Fig. 9 would have to be done by increasing the number of cylinders of different thicknesses that were measured.

However, the fact that Poco graphite does not monitor the resitivity of DFP-2 or AXF-5Q on a billet-by-billet basis [13] is much more important. This means that we have no actual way of checking the resitivity of the billet from which our samples came from but must rely on the statistical value given by the manufacturer. So if our sample was more conductive than the statistical reported value, we could be trying to explain a difference that, in reality, is much lower than what is presented in this paper.

In this study, the values of the resistance obtained using carbon paper are always higher than those obtained using indium. It is therefore logical to try to perform the second calibration method using indium disks. Unfortunately, this has proven very difficult to achieve. Fig. 10 shows the measured voltage drop and applied force in function of the time during a test made using carbon paper disks (A) and indium disks (B). On Fig. 10A, it can be observed that voltage drop first decreases rapidly at the onset of the test, then slowly stabilize around 16,000 s and increases from that point on. These variations could never have resulted from a current variation because in that time frame it increased by less than 5 μA. For a resistance of about 2.5×10^{-4} ohm, this would amount

Table 3
Values of the slope, intercept, through-plane resistivity of Poco graphite and correlation coefficient (r) for five different pressures measured using carbon paper disks

	(875 N)		(1800 N)		(2950 N)		(3900 N)		(4950 N)	
	Value	Error	Value	Error	Value	Error	Value	Error	Value	Error
Intercept (ohm)	9.6E-04	1E-05	6.55E-04	8E-06	5.31E-04	5E-06	4.58E-04	4E-06	4.07E-04	4E-06
Slope (ohm cm^{-1})	2.6E-04	1E-05	2.55E-04	8E-06	2.54E-04	5E-06	2.53E-04	4E-06	2.54E-04	4E-06
Resitivity (ohm cm)	1.34E-03	7E-05	1.29E-03	4E-05	1.28E-03	3E-05	1.28E-03	2E-05	1.28E-03	2E-05
r	0.9862	–	0.9946	–	0.9976	–	0.9985	–	0.9988	–

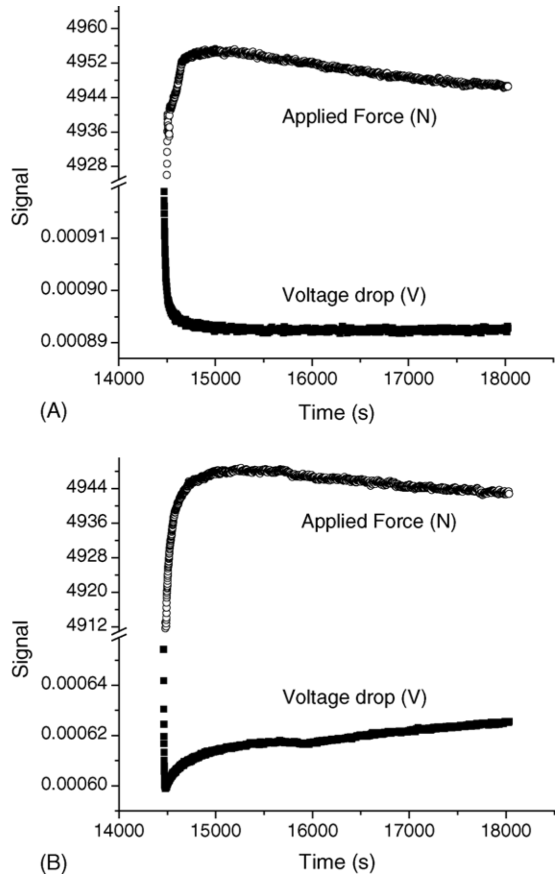

Fig. 10. Voltage drop for Poco graphite sample increasing through time when carbon paper (A) and indium (B) disks are used.

to a change of less than 1.25×10^{-9} V. The only other variable during the test was the applied force that is without any doubt responsible for the observed variation in the voltage drop measurements. On Fig. 10A, we can observe that the voltage drop and applied pressure are inversely proportionate. This is obvious at the onset of the test and after 16,000 where the voltage drop increases while the applied pressure slowly drops.

When indium disks are used (Fig. 10B), the voltage drop behaves in a totally different manner before the 16,000 s mark. For an unknown reason, the voltage drop increases from 14,500 to 15,500 s and that even if the pressure also increases in that time frame. This capacitive-like behavior was observed every time a sample of Poco graphite was measured using indium disks but does not appears to occur when measuring resistors or indium disks alone without any sample. This phenomenon remains unexplained at this time. One of the advanced hypotheses is that since samples of Poco graphite are intrinsically porous (20% porosity [10]), air can diffuse through the sample and react with the indium disk. Indium reacts readily with air to form oxides that are less conductive than the pure metallic indium. When, for a reason, the oxide layer is broken, the voltage drop is lowered.

4. Conclusions

This paper presents a system that was built to measure the through-plane electrical resistivity. An instrumented pneumatic press with copper electrodes and capped resistors were built and calibrated. The reproducibility of the measuring instrument and resistor over a 24-h period was measured to be 2 μohm cm. Several factors were found to influence the value and reproducibility of the measured voltage drops. In order to minimize their effects, new pieces of carbon paper or indium were used for every new sample. The copper electrodes were polisher prior to every measurement using a 1 μm diamond paste.

Two methods were used to try to isolate the resistivity of the analyzed material from the system resistivity. The first method showed that a systematic error of roughly 60 μohm cm was present and likely due to the instruments. It was also determined that the resistance measured for indium disks was lower than that of carbon paper disks by about one order of magnitude.

The first calibration method not only served to show that a systematic error was present in the system but also demonstrated the importance of contact resistances (R_{Cu-PC} or R_{In-PC}). For indium disk, all the measured resistance was due to the interfaces. This is important since the value of the contact resistances might be higher than the value of the bulk resistance if the latter is very low.

The second method was used to measure the conductivity of two Poco graphite samples: AXF-5Q and DFP-2. An error ranging from 60 to 210 μohm cm was always present when trying to measure their resistivities. This error was not caused by systematic errors and we have not, at this time, found its source. Using indium disks for measuring the conductivity of Poco graphite samples was deemed impossible because of a capacitive-like effect.

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