ACCURATE MEASUREMENTS OF ELECTRICAL CONDUCTIVITY OF METALS IN THE RANGE FROM 2 MS/m TO 14 MS/m

Parker, M.; Pokatilov, A; Raba, K. & Kübarsepp, T.

Abstract: The accurate method for electrical conductivity measurements has been established. In the realization of the method Van der Pauw’ DC-measurement technique is applied. The measurements were conducted for five metal plates whose electrical conductivity nominal values are in the range from 2 MS/m to 14 MS/m. The limiting factors of the accuracy in DC-measurements of electrical conductivity have been studied. The electrical conductivity measurements are directly traceable to the Estonian national standards for electrical units, for length and for temperature. The relative uncertainty estimates of the measured electrical conductivity values can be less than 0.3% (k=2).

1. INTRODUCTION

Electrical conductivity measurements are widely used in industry for production and inspection of metals. For example in aviation an electrical conductivity measurement is used to identify defects in metal products, e.g. aircraft wings. The electrical conductivity measurements are also used in coin industry where this kind of measurement is applied for quality assurance of metals and also for detecting counterfeited coins.

The electrical conductivity could be measured with different measurement methods. The widely used commercial devices are based on electromagnetic (eddy-current) method. By using this type of devices accurate measurements in relative terms can be obtained. However, in order to provide traceability to measurement units, eddy-current devices need to be calibrated with appropriate reference standards.

In this paper we describe accurate electrical conductivity measurements by using a well-known Van der Pauw method. We present measurement results and thoroughly studied uncertainty budget. The limiting factors in achieving high-accuracy results by using our measurement technique are briefly discussed.

2. THEORETICAL BACKGROUND

The Van der Pauw’ method could be used to determine electrical conductivity of a sample with any geometrical shape [1]. The electrical conductivity of a squared shaped test piece could be determined according to [2]: where \(d\) is the thickness of the sample

\[
\frac{1}{\sigma} = \frac{\pi d}{\ln 2} \frac{R_a+R_b}{2} f(r),
\]

and \(\sigma\) is the electrical conductivity of material, \(R_a\) and \(R_b\) are resistances measured at two different sides of the test piece, (Fig. 1). In Eq (1) the coefficient \(f(r)\) is equal to one with accuracy 0.001% when the resistance \(R_a\) and the resistance \(R_b\) differ from each other less than one percent [2]. In case the measured resistances \(R_a\) and \(R_b\) differ more than one percent, the coefficient \(f(r)\) should be, however, calculated [1].

In order to take into account several influencing effects in our measurements we have used following equation [3]:

\[
\frac{1}{\sigma} = \rho = \frac{\pi d}{\ln 2} \frac{R_a+R_b}{2} f(r)K
\]

where the coefficient \(K\) is [3].

\[
K = (1 + \delta_{\text{th}})(1 + \delta_{\text{cont}})(1 + \delta_{\tau})
\]
The coefficients $\delta_{\text{thr}}$, $\delta_{\text{cont}}$ and $\delta_t$ represent effects of thermal voltage, contact size and temperature on electrical conductivity measurements, respectively.

Some samples can exhibit differences in the measured resistances $R_a$ and $R_b$ even if the sample is of the square shape. To validate our developed method we measured the resistances $R_c$ and $R_d$ also. The resistances $R_c$ and $R_d$ are measured just at opposite sides of the resistances $R_a$ and $R_b$ respectively (Fig. 1).

3. MEASUREMENTS

The realization of electrical conductivity scale by the Van der Pauw’ (VdP) method requires accurate electrical, temperature and dimensional measurements.

For conductivity measurements by the VdP technique we used a dedicated measurement tool, which ensures firm contacts of measurement leads to the corners of samples, see Figure 2.

The electrical conductivity values of five square metal plates in the range from 2 MS/m to 14 MS/m have been measured by the Van der Pauw method. The presented measurements results have been obtained on the measurement equipment of AS Metrosert at the National Standards Laboratory for Electrical Quantities [4]. The performed measurements are traceable to the Estonian national measurement standards.

**Dimensional measurements**

For a symmetrical square plate only the accurate measurement of the thickness is necessary [2].

In order to test the squarness of our plates, we measured their sides along the X and Y axis (Fig. 1) by the electronic height gage TESA Micro Hite Plus M600 with the measurement uncertainty less than 40µm.

![Fig. 2. Measurement tool for the Van der Pauw method.](image)

![Fig. 3. The measured thickness change of the CuNi sample. The thickness of sides.](image)
denoted as 1-2 is 3.005 mm thick and 3-4 is 2.974 mm. The relative difference between the lengths \( L_a \) and \( L_b \) of the sides of the studied plates are presented in (Table 1).

The thickness of the sample \( d \) is the major dimensional parameter affecting accuracy of the conductivity value. The thickness has been measured at nine points for each plate by the universal length measurement machine ULM Opal 600 with the measurement uncertainty of less than 3 µm. For all plates the measured thickness changes across the plate are less than 10 µm. Except the CuNi plate, which has the highest change in thickness around 30 µm as illustrated in Figure 3.

**Resistance measurement**

The resistances \( R_a \) and \( R_b \) are determined from comparison to a 1 Ω standard resistor by use of the current range extender MI 6011B as a precision current transformer. The direct current of 10 A is supplied from the current source MI 6100A to the VdP fixture through the current transformer where it is divided to the 100 mA level, see Figure 4. The applied high current value ensures required sensitivity in the voltage drop measurement. However, it can cause a temperature gradient across the plate by increasing the temperature of the measurement leads and contacts. Temperature distribution around a contact of the VdP tool can be checked by the Infrared imaging and measurement system FLIR ThermaCAM SC 3000. In Figure 5 an infrared image of a contact of the VdP tool is shown. The temperature of the contact exhibit no significant change in temperature at the measurement current of 10 A. The increase in temperature is mainly observed in the measurement leads.

The resistance value is obtained from the ratio of two voltage drops \( U_x \) and \( U_s \) measured at the plate and standard resistor by the nanovoltmeter Agilent 34420A and multimeter Fluke 8508A respectively:

\[
R = \frac{U_x}{U_s} \cdot R_s \cdot k \tag{4}
\]

where \( R_s \) is the value of the standard resistor and \( k \) is the ratio of the current transformer. The thermal voltages arising in the measurement system in the junctions of dissimilar metals are cancelled out by reversing the current direction for each resistance measurement. The total measurement uncertainty of the resistance

\[
Rs- \text{ shunt, Vs-voltmeter Fluke 8508A, Vx-}\text{nanovoltmeter Agilent 34420A.}
\]
value is estimated to be less than 0.1 % of the measured value.

**Temperature measurement**

In the measurements the VdP measurement tool was placed in the air thermostat at the temperature of 20 °C. This was done to minimize the effect of temperature fluctuation on the electrical conductivity measurements. The temperature values have been measured at five points on the plate by the calibrated resistance thermometers with the measurement uncertainty less than 0.1 °C.

The voltage and temperature measurements performed in the study were automated by the specially developed software. By using the equation (2) the conductivity values of five metal plates have been determined. The conductivity values and relative measurement uncertainties are summarized in Table 1.

5. DISCUSSION

<table>
<thead>
<tr>
<th>ID</th>
<th>Material</th>
<th>Dimensions [mm]</th>
<th>La-Lb, %</th>
<th>Ra-Rb, %</th>
<th>Electrical conductivity</th>
<th>Uncertainty</th>
</tr>
</thead>
<tbody>
<tr>
<td>T178</td>
<td>Titanium</td>
<td>80.0x80.0x10.1</td>
<td>0.0</td>
<td>3.4</td>
<td>2.172 MS/m</td>
<td>0.1%</td>
</tr>
<tr>
<td>CuNi</td>
<td>CuNi</td>
<td>79.5x80.0x3.0</td>
<td>0.6</td>
<td>2.2</td>
<td>3.103 MS/m</td>
<td>0.7%</td>
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<tr>
<td>U178</td>
<td>NordicGold</td>
<td>80.1x80.0x10.1</td>
<td>0.0</td>
<td>0.5</td>
<td>9.571 MS/m</td>
<td>0.1%</td>
</tr>
<tr>
<td>NG2</td>
<td>NordicGold</td>
<td>80.0x80.0x3.0</td>
<td>0.0</td>
<td>0.5</td>
<td>9.973 MS/m</td>
<td>0.2%</td>
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<tr>
<td>C178</td>
<td>Brass</td>
<td>80.0x80.0x9.9</td>
<td>0.2</td>
<td>0.2</td>
<td>14.294 MS/m</td>
<td>0.2%</td>
</tr>
</tbody>
</table>

Table 1. Parameters of the metal plates measured in the present study.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
<th>Uncertainty</th>
<th>Sensitivity coefficient</th>
<th>Uncertainty [Ωm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness d</td>
<td>10.0581·10⁻³ m</td>
<td>2.81·10⁻⁶ m</td>
<td>4.58·10⁶ Ω</td>
<td>1.29·10⁻¹⁰</td>
</tr>
<tr>
<td>Constant</td>
<td>2.2662</td>
<td>2.89·10⁻¹⁰</td>
<td>4.60·10⁻⁷ Ωm</td>
<td>1.33·10⁻¹⁰</td>
</tr>
<tr>
<td>Resistance Rₐ</td>
<td>9.9256·10⁻⁶ Ω</td>
<td>1.02·10⁻⁹ Ω</td>
<td>2.28·10⁻² m</td>
<td>2.33·10⁻¹¹</td>
</tr>
<tr>
<td>Resistance Rₐ</td>
<td>10.2783·10⁻⁶ Ω</td>
<td>1.03·10⁻⁹ Ω</td>
<td>2.28·10⁻² m</td>
<td>2.35·10⁻¹¹</td>
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<tr>
<td>f ( r )</td>
<td>0.99989</td>
<td>8.85·10⁻⁷</td>
<td>4.60·10⁻⁷ Ωm</td>
<td>4.07·10⁻¹¹</td>
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<tr>
<td>δᵣₑₜ</td>
<td>0</td>
<td>1.14·10⁻⁴</td>
<td>4.60·10⁻⁷ Ωm</td>
<td>5.26·10⁻¹¹</td>
</tr>
<tr>
<td>δᵣₑₜ</td>
<td>0</td>
<td>2.80·10⁻⁴</td>
<td>4.60·10⁻⁷ Ωm</td>
<td>1.29·10⁻¹⁰</td>
</tr>
<tr>
<td>δₑₜ</td>
<td>0</td>
<td>2.89·10⁻⁸</td>
<td>4.60·10⁻⁷ Ωm</td>
<td>1.33·10⁻¹⁴</td>
</tr>
<tr>
<td>Resistivity</td>
<td>4.6047·10⁻⁷ Ωm</td>
<td>Uncertainty, k=1</td>
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<tr>
<td>Conductivity</td>
<td>2.172 MS/m</td>
<td>Uncertainty, k=2</td>
<td>0.002 MS/m</td>
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<tr>
<td>IACS</td>
<td>3.744 %</td>
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</table>
Table 2. The electrical conductivity value and uncertainty of the sample denoted T178.

correction for the measured difference of resistances is insignificant. However, the remaining differences are of the order of few percents (Ti and CuNi). The reason for that is not exactly known but it could be due to material properties (e.g., crystal lattice structure) and/or manufacturing technology (e.g., rolling, pressing).

In addition, the wedge-shaped geometry of a sample can have an effect on the measurement uncertainty. For example, the change in thickness of the sample CuNi was about 30 µm. This causes increase in the uncertainty of average thickness estimate which, in turn, increases the uncertainty in the determination of electrical conductivity. For the sample CuNi the relative uncertainty estimate was 0.7%.

6. CONCLUSION

The electrical conductivity scale at the direct current in the range from 2 MS/m to 14 MS/m has been realized by means of the Van der Pauw technique.

The performed conductivity measurements are traceable to the Estonian national standards for electrical units, for length and for temperature.

The limiting factors of accuracy in the Van der Pauw’ method have been thoroughly investigated. In case of a square sample the highest uncertainty component is due to thickness measurement of the plate. For the investigated samples the relative uncertainties are estimated to be in the range from 0.1% to 0.3% of the measured value.

7. ACKNOWLEDGEMENTS

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8. REFERENCES