Measurement systems analysis for the determination of volume resistivity and polarisation indexes

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***Abstract*— Volume resistivity is one of basic material properties determined for electrical insulating materials. In many cases, the measurement process can affect the accuracy of the measured data. Therefore, this paper analyzes the measurement process while measuring this parameter. A necessary step to perform the analysis is the analysis of the variability that may arise in the whole measurement process. A controlled experiment is then designed to investigate the factors assumed to influence the measurement result based on this analysis. This controlled experiment is followed by experiment evaluation, results analysis, and a suggestion of possible process or organizational changes that can be made to the measurement system to improve the monitoring of material properties using this diagnostic method. The tests performed showed an expected result: the shorting book and the electrostatic bracelet affect the measured value of the volume resistivity but every parameter to a different degree.**

Keywords—volume resistivity, measurement systems analysis, GRR study, stability

I. Introduction

The measurement process is an important activity in determining the properties of materials and equipment conditions. The measurement process analysis is one of the activities that help determine the magnitude of the influence on the measured object. Typically, measurement errors or uncertainties are determined (e.g., according to [1] or industry standards). Detailed analysis of the effects of the measurement on the measured object is only required in the automotive industry ([2], [3] and related customer-specific requirements); elsewhere, it is usually not performed. The procedures and principles set out in this literature can be used for other analyses, as they are general statistical procedures applied to specific analyses requirements.

Given the above, the paper focuses on analyzing the effect of selected influences of the measurement system when measuring volume resistivity and polarization indexes.

II. Volume Resistivity and polarisation indexes Measurement Process

Volume resistivity and polarization indexes belong among basic parameters characterizing the condition of insulating materials. The volume resistivity indicates its ability to prevent the passage of electric current through a sample. The lower the resistivity represents, the higher the conductivity.

The volume resistivity is calculated using the following equation (1):

$$\rho = \frac{22{,}9}{d} \times \frac{U}{I} \tag{1}$$

Where ρ is volume resistivity (Ω·cm), U is applied DC voltage (V), d is the sample thickness (cm), I current at the 10^{th} minute.

Another parameter that helps evaluate the insulation system's quality is the polarization index (PI). PI is determined from the ratio of currents measured during the resistivity measurement (based on absorption current). The standard calculation of PI uses measurement of current taken in 1 minute and after 10 minutes, as shown in (2). A sharp decline in PI is an indication of severe insulation degradation.

$$PI = \frac{I_1}{I_{10}} \tag{2}$$

Where I_1 and I_{10} are current values measured at 1 and 10 minutes of resistivity measurement; similarly, it can be calculated as the ratio of currents in the fifteenth and sixtieth seconds.

The basic arrangement for measuring the volume resistivity consists of a voltage source, a three-electrode system, and a picoammeter. It is recommended that, before the actual measurement, the samples are placed in a shorting book for 24 hours, where the residual electric charge is removed. It is also recommended to use an electrostatic bracelet while handling samples. Again, due to protects against their electrostatic charge during manipulation.

II. Influences Affecting The Measurement Results

Throughout the measurement process, it is possible to identify several influences that could affect the measurement result and thus contribute to the measured value, i.e., cause a systematic measurement error. As factors potentially influencing the results may be considered:

1) the effect of uncertainty in equipment calibration,
2) the effect of the measurement system's bias,
3) the effect of measurement system non-linearity,
4) the effect of measurement system instability,
5) the effect of repeatability on the measurement system,

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6) the effect of reproducibility on the measurement system,
7) the effect of the use of a shorting book,
8) the effect of using an electrostatic bracelet,
9) the effect of the test voltage.

During the preliminary assessment of the individual effects, the following statements and hypotheses were formulated:

- there will be factors that we do not anticipate will affect the measurement system,
- there will be factors that we anticipate will have an impact on the measurement system but will not need to be interact in parallel with the other factors,
- there will be factors that we anticipate will affect the measurement system. At the same time, it should be assumed that the interaction of these factors may also affect the measurement result and, therefore, should be considered together.

This paper only considers factors that affect the measured value, but it is not assumed they may interact with other factors. From the influences mentioned above, three factors were selected, and the experiment was designed as follows:

1) an experiment in which the effect of stability (i.e., time stability) in the measurement system on the measurement result will be evaluated,
2) an experiment to evaluate the effect of the use/non-use of a shorting book on the measurement result,
3) an experiment to evaluate the effect of the use/non-use of the electrostatic bracelet on the measurement result.

Thus, the objective is to analyze the measurement system for volume resistivity and polarization indexes with factors determined by preliminary analysis and divided into three experiments. The analysis of the remaining factors is beyond the scope of this paper.

III. Experimental Design

The output variables of the experiment (response) were chosen to be a minute polarization index, a ten-minute polarization index, and volume resistivity. Due to the laboratory measurement conditions, disturbing factors were not considered, and other unexamined effects were considered constant.

In the first experiment investigating the effect of time stability, repeated measurement on a single material sample was proposed. This measurement was carried out at intervals of at least one day for 20 days. The constant parameters are using a shorting book and an electrostatic bracelet, where both the shorting book and the electrostatic bracelet are constant. The resulting data set is then statistically evaluated using appropriate time series analysis methods.

For the other experiment, focusing on the effect of the shorting book, an experiment is designed again involving measurement on a single sample, identical to the previous paragraph. The data from the previous experiment are used for this measurement, and ten additional values are also measured when the shorting book is not used. Not using the shorting book allows measurement to be made at short time intervals. The data set contains data from measurement with or without the shorting book, and it is possible to determine the influence of this factor. Methods for evaluating single-factor experiments are used to evaluate this effect.

The last experiment investigating the effect of using a bracelet follows a similar procedure. Again, the data from the first sub-experiment, where an electrostatic bracelet is used for the measurement, is used. Subsequently, a set of data (10 values) is measured on the same sample without using the electrostatic bracelet over a short period. The effect of using an electrostatic bracelet can be compared using the same evaluation methods for the measured data.

IV. Measuring Process

A flat sample made of polyethylene (PE) with the size of 100 × 100 mm and the average thickness of 0.3 mm (see Fig. 1) were chosen for the abovementioned experiments.

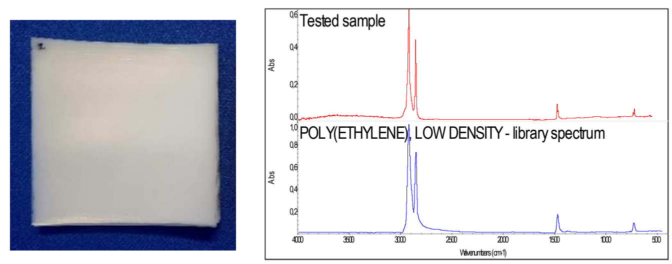

Fig. 1. Tested material and its identification using Fourier Transform Infrared Spectroscopy (FT-IR)

The one-minute and ten-minute polarization indexes and volume resistivity were measured and evaluated for this material. All quantities were obtained from the time dependence of the current flowing through the material during the measurement (see Fig. 2) as follows:

1) the minute polarization index (dimensionless) - calculated as the ratio of the current flowing through the material at the fifteenth second after the voltage was applied to the value of the current flowing after the first minute of the measurement,
2) the ten-minute polarisation index (dimensionless) - calculated as the ratio of the current flowing through the material at the first minute after the voltage was applied to the value of the current flowing after the ten minutes of the measurement,
3) the volume resistivity (Ω·m) - was calculated according to equation (1), using the average of the last ten measured values of current at the tenth minute of the measurement.

An example of the time dependence of the current for one series of measurement is shown in the following Fig. 2. For the experiment, the most critical parameter is the leakage current, representing the current's steady-state value. Due to the nature of the samples (insulation material) and thus the minimal values of the measured currents (E-16 A), it is complicated to achieve a genuinely constant (steady) value in a short time period. For this reason, it is usually the case that a certain period is chosen after applying the voltage (5, 10, and 30 min, etc.), after which the current is taken as constant. However, it is always advisable to view the time dependence of the current to confirm this assumption. In our example, a period of 10 minutes was chosen.

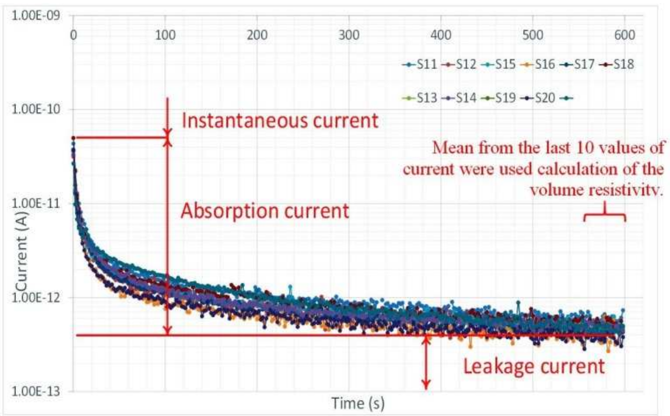

Fig. 2. Time dependences of current for individual measurement

The current from which the above parameters are subsequently calculated was measured with a Keithley Instruments electrometer with an internal source of DC voltage (model 6517) and a three-electrode system (Keithley Instruments, model 8009). The measurement was carried out by an experienced person who had been adequately trained in this type of measurement. Measurement proceeded following the [5] and under a DC voltage. The test voltage was set to 1000 V, and the actual measurement was performed using a programmed procedure in the Keysight VEE graphical dataflow programming software. When a shorting book was used, the sample was, before measurement, placed in this short-circuit book for at least 24 h.

The measurement procedure started with a five-minute sample discharge in the electrode system, followed by connecting the test voltage and measuring the absorption current. The steady-state current was recorded for 10 minutes of polarization. The data produced in a dataset were further processed, and the analyzed parameters (resistivity and polarization indexes) were calculated using MS Excel. TIBCO Statistica software was used for statistical analysis of the results.

V. Experimental Results

A control chart for individual values and moving ranges were used to evaluate the first experiment examining the effect of temporal constancy (Figure 3). The chosen type of control chart helps analyze the measured data's behaviour. When evaluating the control charts for the individual values and the sliding ranges of the variables under evaluation, it is evident that the measured values do not show any trends, non-random groupings or values outside the control limits. This graphical representation shows that the measurement system does not exhibit significant variations over time and can be considered temporally stable for measuring the physical quantities listed.

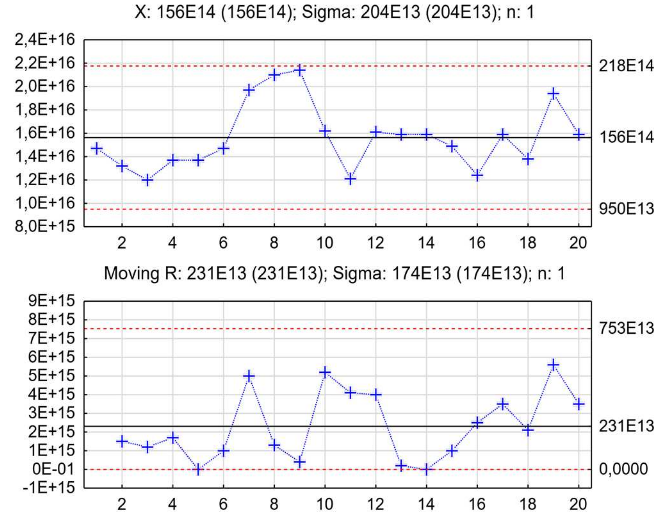

Fig. 3. Control diagram for individual values and sliding ranges

The other experiment investigated the effect of the shorting book on the result of the measurement. One-factor analysis as a statistical tool was used for the evaluation. The essential tool here represents the analysis of variance, which helps decide the significance/insignificance of each factor at the chosen level of significance α. After running the tests, it could be concluded at the significance level $\alpha = 0.05$ that the use of the shorting book does not affect the value of the one-minute or ten-minute polarization index. Although the measured values of the current waveforms differ (see Fig. 2), their ratio, i.e., the polarisation index, is not affected.

However, the situation is different for volume resistivity. Here, it could be concluded at the significance level $\alpha = 0.05$ that the effect of the shorting book is proven; the *p*-value of the analysis of variance test is significantly less than 0.05. This fact is then confirmed by using a method for multiple comparisons, e.g., using Tukey's or Fisher's method. An unexpected result of the analysis is that when the shorting book is not used, the volume resistivity value is about by 30 % higher, see Figure 4.

The same statistical tools were used to evaluate the last experiment, investigating the effect of using a shorting bracelet. The main result here is that at the chosen level of significance $\alpha = 0.05$ the use of the electrostatic bracelet does not affect the minute polarization index. In the case of the ten-minute polarization index, the null hypothesis of no effect due to the use of the electrostatic bracelet was rejected at the same significance level (α = 0.05); the proper significance level represented by the p-value is $p = 0.042$. Practically, therefore, the electrostatic bracelet should affect the ten-minute polarization index. Since the rejection of the hypothesis occurs, based on a non-significant difference between α and p, it is a decision in the marginal band. The result is classified in the range of uncertainty in decision making rather than as an unambiguous impact decision of the electrostatic bracelet.

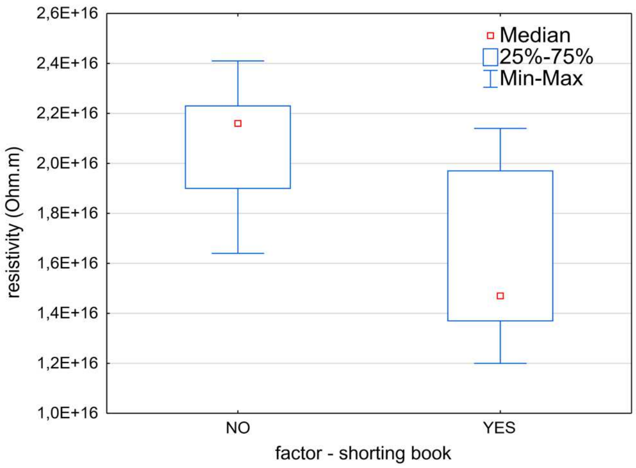

Fig. 4. Comparison of the change in volume resistivities using a shorting book

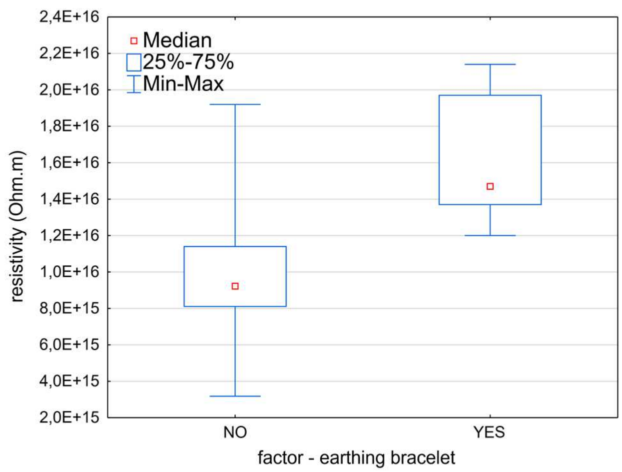

Fig. 5. Comparison of the change in volume resistivities using the bracelet

In the case of the evaluation of the effect of the use of an electrostatic bracelet on volume resistivity, the conclusion is more explicit. After the tests were performed, it can be concluded at a significance level $\alpha = 0.05$ that the use of an electrostatic bracelet affects the volume resistivity of the material. Again, the *p*-value of the analysis of the variance test is significantly less than 0.05. The volume resistivity is approximately by 60 % higher after using the bracelet than in the condition where the electrostatic bracelet is not used (Figure 5). In addition, the variance of the measured values is approximately by 1/3 smaller when the bracelet is used than in case the bracelet is not used. The higher resistivity is related to the lower value of leakage currents for the measurement obtained using an electrostatic bracelet, reducing sample charging during handling.

VI. Conclusions

Based on the experiments performed, the following conclusions can be stated. The measuring system shows the characteristics of a time stable device, which is proved by the first experiment. This fact is based on examining actual data over 20 days. However, to confirm this conclusion, it would be advisable to repeat this experiment after some time or carry out an experiment covering a longer time interval. Continuous validation at regular intervals need not be considered here.

The shorting book and the electrostatic bracelet do not affect the measured material's minute and ten-minute polarization index. Although we found a statistically significant effect on the measured value when using the electrostatic bracelet on the ten-minute polarization index, we understand this significance to be borderline. We cannot exclude the possibility that repeated measurement would reach a similar borderline conclusion but in the realm of acceptance of the hypothesis.

However, the tests performed showed that the shorting book and the electrostatic bracelet affect the measured value of the volume resistivity. Using the shorting book reduces the volume resistivity values, while using the electrostatic bracelet increases the volume resistivity. Using the electrostatic bracelet is approximately twice as great as the effect of using the shorting book. This can be explained by the fact that the measurement of the absorption current in the electrode system is first preceded by a five-minute sample discharge (defined by standard), which can partially simulate the location of the sample in the shorting book. In contrast, the actual handling of the sample without the use of the shorting bracelet may result in a more significant charge that this process can no longer sufficiently eliminate.

After a comprehensive look at the analyzed factors, it is recommended to use the shorting book and the electrostatic bracelet when measuring volume resistance. These measures will then help eliminate the systematic errors demonstrated by this analysis.

The future work will focus on analyzing other remaining unanalyzed possible influences mentioned at the beginning of the paper. However, it will probably be necessary to conduct an experiment that includes investigating the interactions among different factors.

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