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## The determination of measurement errors in impedance spectroscopy

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# The determination of measurement errors in impedance spectroscopy

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**Abstract.** The paper deals with the methodology, quantification and comparison of measurement deviations for two types of the ceramic shard. The ceramic material has better polarization properties than concrete material. For impedance measurement the ceramic shard is more appropriate. The IS method diagnostics based on processing of measured data which aims evaluating the absolute error and the relative error (deviation) of the loss factor values. Results show the spectrum parts which are useful in the lower frequency range only. When Agilent 33220A and Agilent 54645A are used to carry out the IS, the most important factor from the viewpoint of the  $\text{tg}\delta(f)$  determination accuracy is the accuracy of the phase shift between the excitation signal and that at the measured system output. Other factors are the measurement accuracy of the electric capacity and resistance of the oscilloscope input channel.

## 1. Introduction

Impedance measurements are feasible with specialized devices (such as Agilent 4294A, 4284A), which are in most cases acquisition price exacting. Fortunately, there are cases where the impedance measurements can be carried out with alternative device blocks consisting of an exciting signal generator, a recording device and a data processing device. An impedance measurement apparatus consisting of a sine-wave voltage, a double-channel oscilloscope and a PC has been implemented at the Faculty of Civil Engineering of the University of Technology of Brno. Until now, experimental data in the form of the loss factor spectra versus frequency plots have been acquired and other frequency and impedance plots have been derived [1, 2].

The measurement reproducibility has been verified on various materials. The absolute errors of thus acquired physical quantity of the frequency spectrum have been assessed on the basis of mutual deviations of the spectrum individual points. The reliability of frequency interval readings has been established based on the observed deviations. So far, the measurement absolute error has not been determined from the input quantity accuracy.

The loss factor values [3–5] have been calculated from the formula:

$$\text{tg}\delta = \frac{1}{\text{tg}\varphi_1} \quad (1)$$

where:

$$\varphi_1 = \varphi_2 + \arctg \frac{\sin \varphi}{\frac{U_2}{U_1} - \cos \varphi} \quad (2)$$



$\varphi$  is the phase difference between the voltages  $U_1$  and  $U_2$  of the respective exciting and the attenuated signal at the specimen output,  $\varphi_1$  is the phase shift between the voltage and current vectors in the impedance consisting of the electric resistance  $R$  and the capacitance  $C$  of the oscilloscope input channel at the exciting signal frequency  $f$ .

$\varphi_2$  is determined from:

$$\varphi_2 = \arctg \frac{1}{2 \cdot \pi \cdot f \cdot R \cdot C} \quad (3)$$

The values of  $f$ ,  $\varphi$ ,  $U_1$  and  $U_2$  are calculated as arithmetic means of the quantity measured. The measurement repetition number can be set in the automated measurement software, which controls Agilent 33220A generator, Agilent 54645A oscilloscope and the PC. In compliance with prof. Horák's Practical Physics textbook (basic principle of physical measuring in Czech language), the absolute error of  $\text{tg}\delta$  of each point of the frequency spectrum is determined as follows [6]:

$$\delta_A(\text{tg}\delta) = \sqrt{\left(\frac{\partial \text{tg}\delta}{\partial f} \cdot \delta_A(f)\right)^2 + \left(\frac{\partial \text{tg}\delta}{\partial R} \cdot \delta_A(R)\right)^2 + \left(\frac{\partial \text{tg}\delta}{\partial C} \cdot \delta_A(C)\right)^2 + \left(\frac{\partial \text{tg}\delta}{\partial \varphi} \cdot \delta_A(\varphi)\right)^2 + \left(\frac{\partial \text{tg}\delta}{\partial U_2} \cdot \delta_A(U_2)\right)^2 + \left(\frac{\partial \text{tg}\delta}{\partial U_1} \cdot \delta_A(U_1)\right)^2} \quad (4)$$

where  $\delta_A(f)$  is the absolute error of the frequency which is used in the calculation. Absolute errors of the quantities  $f$ ,  $\varphi$ ,  $U_1$  and  $U_2$ ,  $R$ ,  $C$  are defined as follows:

$\delta_A(f)$  = arithmetic mean of the measured frequency / 10 000.....Hz

$\delta_A(R)$  =  $R/100$  (corresponds to 1% accuracy)..... $\Omega$

$\delta_A(C)$  =  $C/100$  ((corresponds to 1% accuracy).....F

$\delta_A(\varphi)$  = 1.....degree

$\delta_A(U_2)$  =  $0.0035/\sqrt{5}$  (corresponds to the amplitude measurement accuracy for 5 repeated measurements).....V

$\delta_A(U_1)$  =  $0.0035/\sqrt{5}$  (corresponds to the amplitude measurement accuracy for 5 repeated measurements).....V

Let the loss factor absolute error be denoted ERR and let the second powers of the terms in the parentheses under the second root sign be related to the variables under question and be denoted  $\text{FREQ}$ ,  $\text{CAPAC}$ ,  $\text{REZIST}$ ,  $\text{PHAS}$ ,  $U_2$ ,  $U_1$ , respectively. The values of the terms will be expressed in numbers and their squared roots will be shown in diagrams for the sake of discussion. Furthermore, we will compare the incidence of the deviation with the loss factor values for two different specimens of the ceramic shard. The partial derivatives in the rem 4 have been calculated by Mathcad system.

## 2. Materials and methods

The ceramic specimens have been manufactured using Chvaletice fly ash, their firing temperature being 1 050°C. The firing losses amounted to 11.2%. The bulk density was about 2,005 kg/m<sup>3</sup>. The water absorption capacity: 7.5%. The bending strength: 24 MPa. The granulometry: about 5% on a 63 mm sieve. The ceramic shard specimens differ from each other by the admixture:

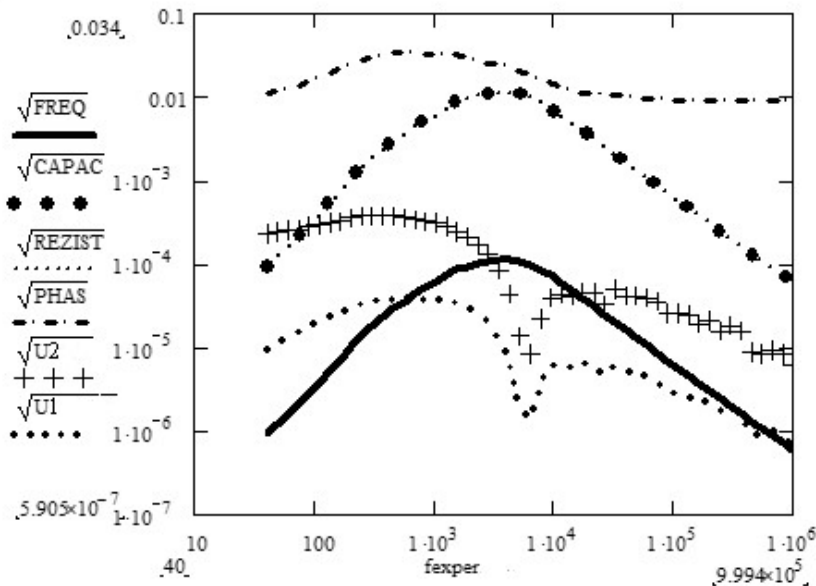
Bentonite ( $\text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2 \cdot 9\text{H}_2\text{O}$ ) - clay. It is a white to light-yellow powder material featuring a specific chemical composition. It is used as ceramics plasticizing agent in the ceramic industry. In this research, a non-activated bentonite, in which the calcium and magnesium ions have not been replaced by activation by sodium ions, has been used. The apparent volume density was 936 kg/m<sup>3</sup>, pH = 8.7. Bentonite admixture: 5%.

Water glass – sodium water glass  $\text{Na}_2\text{SiO}_3$  of a silicate module of 1.6 and a density of 1560 kg/m<sup>3</sup> (KOMA a.s.) has been used to prepare the specimens.

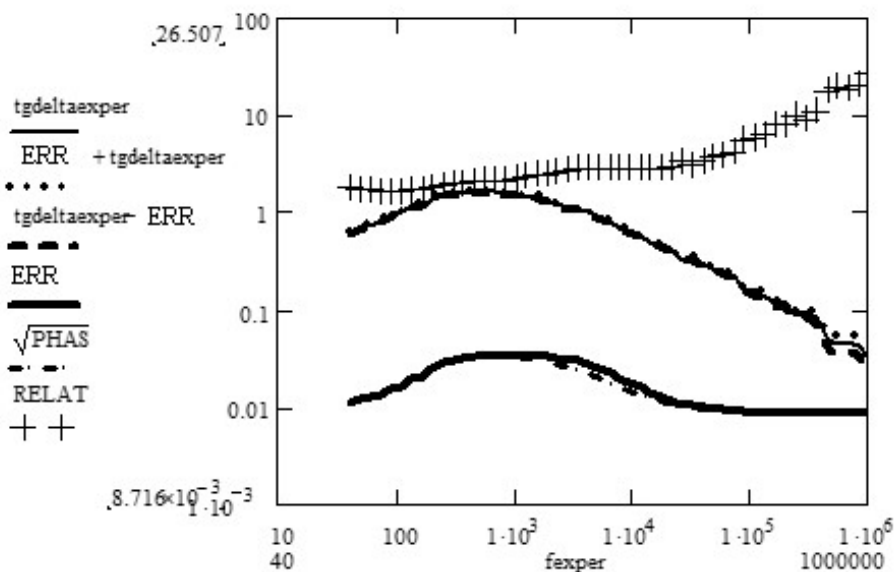
The specimens were inserted between brass plate electrodes to be IS-tested. Agilent 33220A and Agilent 54645A were used to carry out the IS [7–10].

### 3. Results and discussion

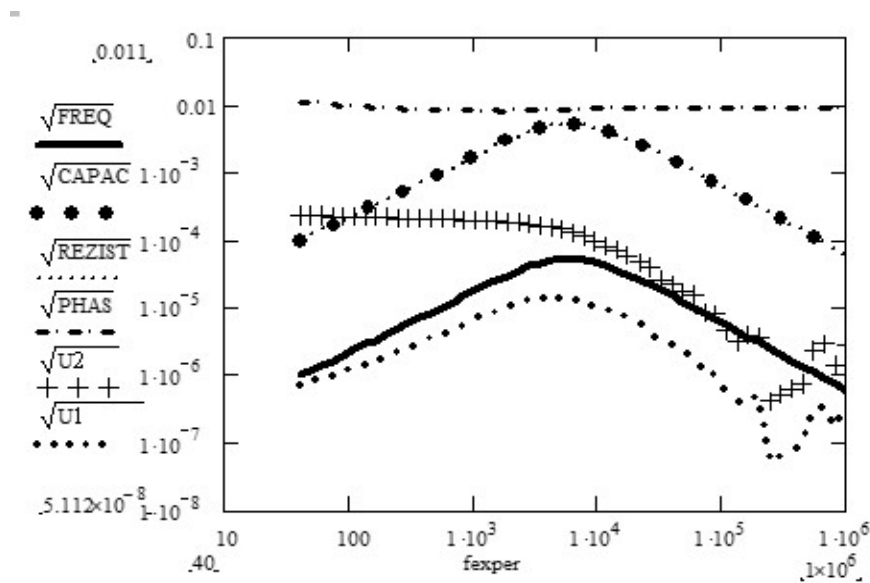
The diagrams in figure 1 and figure 3 show the square roots of FREQ, CAPAC, etc. For the ceramic shard with 5% of bentonite (figure 1), the highest values are acquired by the terms representing the effect of the determination accuracy of the phase difference between  $U_1$  and  $U_2$  on the total absolute error of the loss factor. In the frequency range from  $10^3$  to  $10^5$  Hz an increased accuracy contribution is manifested by the determination of the electric capacitance and resistance, the oscilloscope input channel capacitance plus the electrode and coaxial cable capacitance. The remaining terms acquire negligible values.



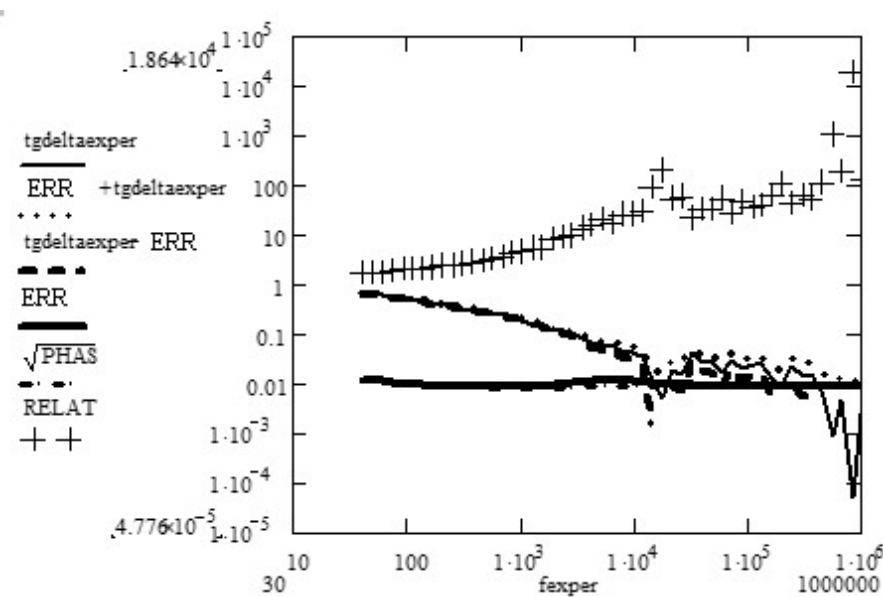
**Figure 1.** Square roots of FREQ, CAPAC, REZIST, PHAS, U2, U1 for 5% - bentonite ceramic specimens. They are expressed in relative frequency units, related to the experiment frequency of electric field in Hz.



**Figure 2.** Loss factor and its absolute and relative errors versus frequency plots for the frequency spectrum and 5% - bentonite ceramics. They are expressed in relative frequency units, related to the experiment frequency of electric field in Hz.



**Figure 3.** Square roots of FREQ, CAPAC, REZIST, PHAS, U2, U1 for sodium-water-glass ceramic specimens. They are expressed in relative frequency units, related to the experiment frequency of electric field in Hz.



**Figure 4.** Loss factor and its absolute and relative errors versus frequency plots for the frequency spectrum and sodium-water-glass ceramic specimens. They are expressed in relative frequency units, related to the experiment frequency of electric field in Hz.

In the case of sodium-water-glass ceramic shard, a similar situation takes place, however, the values of all terms are several orders of magnitude lower.

The loss factor values and their absolute errors can be compared in the diagrams of figure 2 and figure 4. The bentonite ceramic shard shows the loss factor to exceed unity and the absolute error (ERR) of the loss factor determination ranges from 0.01 to 0.05. The loss factor values do not fall into this interval throughout the entire frequency spectrum. The absolute error (ERR) is growing slowly, exceeding 10% for  $\text{tg}\delta$  below 0.1. The envelope of the  $\text{tg}\delta(f)$  curve, which arose by adding and subtracting the absolute error to/from the loss factor values, forms a narrow band which broadens in the highest frequency range only.

For the sodium-water-glass ceramic specimens, the  $\text{tg}\delta(f)$  spectrum shows a decreasing tendency, the respective values being below 0.7. For this material, the values of ERR do not fall below 0.01, however, they are significantly lower than those for bentonite ceramics. In figure 4, the loss factor gets very low values close to the absolute error value. In consequence, the relative error is growing up. Based on the relative deviation values, the loss factor value reliability and the experimental data from various parts of the frequency range can be assessed.

The values of accuracy are better for low frequency than for right side of frequency spectra from  $10^4$  Hz to  $10^6$  Hz. This is only for our measured data specific. Other scientific equipment as impedance analysers are, have typical accuracy 0.02% for precision bridge, but only up to  $10^5$  kHz or  $2 \cdot 10^5$  kHz.

#### 4. Conclusion

The IS method diagnostics based processing of intermediate data which aims at evaluating the absolute error and the relative error (deviation) of the loss factor values shows the spectrum parts to be applicable in the lower frequency range only. When the above mentioned instruments are used to carry out the IS, the most important factor from the viewpoint of the  $\text{tg}\delta(f)$  determination accuracy is the accuracy of the phase shift between the excitation signal and that at the measured system output. Other factors are the measurement accuracy of the electric capacity and resistance of the oscilloscope input channel.

The paper compares two distinct spectra of  $\text{tg}\delta(f)$  and its determination accuracy for two ceramic shard specimens. Different values of  $\text{tg}\delta(f)$  have also been accompanied by different values of the standard deviation, which in this case have been directly proportional to the loss factor values.

In conclusion, it can be stated that the IS method measurement by means of Agilent 33220A generator, Agilent 54645A oscilloscope and plate electrodes can be considered satisfactory unless the loss factor value falls below 0.1.

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