

FAST IMPEDANCE SPECTROSCOPY METHOD USING SQUARE PULSE EXCITATION

Jerzy Hoja¹, Grzegorz Lentka²

Gdansk University of Technology, Gdansk, Poland, ¹ hoja@eti.pg.gda.pl, ² lentka@eti.pg.gda.pl

Abstract: The paper presents a fast impedance spectroscopy method for objects with very high impedance $|Z_x| \geq 1 \text{ G}\Omega$ modeled by multi-element two-terminal RC networks. The method is based on analysis of the object response in the time domain after square pulse excitation. The object impedance spectrum was obtained using continuous Fourier transform. Simulation tests were performed for different excitation pulse widths. Measurements in the realized system proved the usefulness of the method for parametric identification of equivalent circuits of the objects.

Keywords: impedance spectroscopy, voltage pulse, time domain response

1. INTRODUCTION

Impedance spectroscopy (IS) is an universal and effective tool used for testing of electrical properties of technical and biological objects. It allows finding an object's equivalent circuit in the form of multi-element two-terminal networks. IS is used in many areas, e.g. in research of corrosion, protective coatings, fuel cells and impedance measurement of membranes, isolation and biomedical materials.

In order to perform IS, measurement systems generating the excitation signal with a specified shape and simultaneously analyzing the object response signal are used. Instruments available nowadays use an impedance measurement technique based on object excitation with a SST (single sine technique) harmonic signal and vector measurement of two signals: voltage across and current through the object under measurement [1, 2]. By repeating measurements at different frequencies, the impedance or admittance spectrum can be obtained as a function of frequency in a range of several decades. The spectrum allows the analysis of measured object's properties using the Complex Nonlinear Least Square Fitting (CNLS) algorithm [3] which makes parametric identification of object equivalent circuit components possible.

The main disadvantage of the SST technique is the very long measurement time, especially for very low measurement frequencies (mHz, μHz). The use of low frequencies for IS is necessary in the case of component identification of objects with a very high impedance

modulus $|Z_x| \geq 1 \text{ G}\Omega$. Such a case appears when performing IS of e.g. high-thickness anticorrosion coatings, dielectric materials and it causes that the impedance spectrum measurement lasts even a few hours and is possible mainly in the laboratory. In the above case, even the use of MST (multi sine technique) accelerating IS [4] does not allow to shorten meaningfully the spectroscopy time.

The authors, searching for a method making fast impedance spectroscopy possible (lasting not more than a few minutes) for an object directly in the field, like anticorrosion coatings on bridges, pipelines etc., have used a method based on an analysis of the response signal in the time domain when exciting the object with a square pulse signal. The proposed method can be counted to a new class of IS techniques, which use non-harmonic excitation signals like a voltage or current [5] unity step signal or pseudo-random white noise [6]. Unfortunately, the above-mentioned methods were not used in commercially available IS instrumentation. So, after simulation tests and verification in a laboratory system, the authors plan to implement the developed method into a portable IS analyzer, which will be put into production.

The paper presents the theoretical background of the method and an analysis of simulated impedance spectra of the tested object. The method was evaluated for different pulse times and different response sampling parameters (sampling frequency, acquisition time and AD converter resolution were taken into account).

2. IMPEDANCE MEASUREMENT METHOD

In order to meaningfully shorten the time of classical IS, realized by sequential impedance measurement at selected frequencies of a harmonic signal, the single square pulse excitation was used. The measurement of tested impedance Z_x response amounts to sampling and quantizing signals proportional to the voltage across and current through Z_x , using AD converters. The measurement system to verify the presented conception is presented in Fig. 1. The system consists of a personal computer with an installed DAQ card and input circuitry connecting the measured object (Z_x).

The excitation signal for the measured impedance Z_x is prepared with the aid of a DA converter placed on the DAQ card. In order to limit the maximum value of current flowing

through the measured object, the programmed resistor R_0 has been used at the output of a buffer supplying the excitation signal $u_0(t)$. Current $i_x(t)$ is converted to voltage $u_1(t)$ in a current-to-voltage converter realized using amplifier A_1 . A change of the current range is realized with the aid of the programmable resistor R_R . This allows to match the signal $u_1(t)$ to the input range of the ADC1 converter existing on the DAQ card. The generated excitation signal $u_0(t)$ is not an ideal square pulse, additionally it is deformed by parasitic capacitance. Due to these facts, a measurement amplifier was used in the system for real voltage $u_x(t)$ appearing across Z_x . The amplifier output signal $u_2(t)$ is quantized by the ADC2 converter.

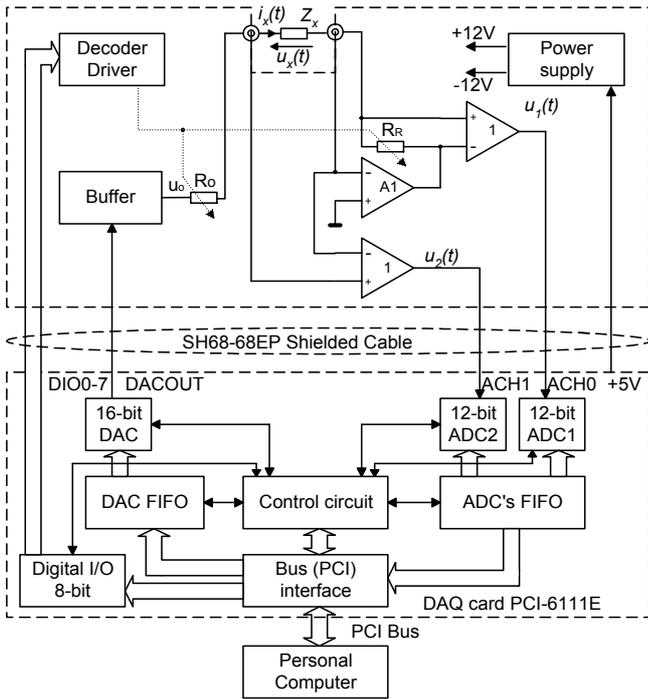


Fig. 1. Block diagram of a measurement system realizing the method of fast IS

Current $u_1(t)$ and voltage $u_2(t)$ responses contain information about the impedance of the tested object as a function of frequency. This information can be extracted by using Fourier transformation, after approximating time responses using linear functions and calculation of transform using formulas:

$$U_i(j\omega) = \int_0^{\infty} u_i(t) \exp(-j\omega t) dt, \quad \text{where: } i = 1, 2 \quad (1)$$

To calculate the classical discrete Fourier transform it is necessary to sample voltages $u_1(t)$ and $u_2(t)$ with a constant time step. Assuming a maximum measurement time equal to 100 s and a sampling frequency equal to 10 kHz, the required number of samples of each signal should be equal to 1000000. Transform calculation using such a number of samples would last even dozens of minutes. Therefore, in the presented solution, the acquisition time was divided into time segments in which sampling is performed with a different frequency. A maximum number of 6 time segments was assumed with high limits as follows: 0.01 s, 0.1 s, 1 s, ...,

1000 s, and a constant number of 1000 samples in each segment. For the assumed condition, the maximum number of samples collected during the acquisition time is equal to $N = 6000$. In this case the transform can be calculated using the definition, it means that (1) can be written:

$$U_i(j\omega) \approx \sum_{n=1}^{N-1} \int_{t_n}^{t_{n+1}} \tilde{u}_i(t) \exp(-j\omega t) dt, \quad (2)$$

where $\tilde{u}_1(t)$ and $\tilde{u}_2(t)$ are linear approximations of each response signal (current and voltage) between samples.

After approximation and integral calculation we obtain:

$$\text{Re } U_i(\omega) \approx \sum_{n=1}^{N-1} \left[\frac{1}{\omega} (u_i(t_{n+1}) \sin \omega t_{n+1} - u_i(t_n) \sin \omega t_n) - \frac{u_i(t_{n+1}) - u_i(t_n)}{t_{n+1} - t_n} \cdot \frac{\cos \omega t_{n+1} - \cos \omega t_n}{\omega^2} \right] \quad (3)$$

$$\text{Im } U_i(\omega) \approx \sum_{n=1}^{N-1} \left[-\frac{1}{\omega} (u_i(t_{n+1}) \cos \omega t_{n+1} - u_i(t_n) \cos \omega t_n) + \frac{u_i(t_{n+1}) - u_i(t_n)}{t_{n+1} - t_n} \cdot \frac{\sin \omega t_{n+1} - \sin \omega t_n}{\omega^2} \right] \quad (4)$$

Determining from (3) and (4) the spectra of the signal proportional to the voltage across and current through the measured object, on the basis of impedance definition, the impedance spectrum can be calculated using formula (5):

$$Z(\omega) = \frac{\text{Re } U_2(\omega) + j \text{Im } U_2(\omega)}{\text{Re } U_1(\omega) + j \text{Im } U_1(\omega)} R_R, \quad (5)$$

3. ANTICORROSION COATINGS SPECTROSCOPY

The developed method for the fast spectroscopy method is aimed at high-impedance objects, as mentioned at the beginning. The method is particularly dedicated to impedance measurement of anticorrosion coatings, which allows to evaluate the condition of the anticorrosion protection. Due to safety and economical reasons it is necessary to determine the quality of a protective coating in order to estimate the renovation time. This leads to the need of impedance measurement of anticorrosion coatings and as the result to diagnose their condition, also performed in the field, directly on the protected object.

Figure 2a presents a photograph of a typical cell for impedance measurement of the anticorrosion coating on a high-voltage power line pylon, and Fig. 2b presents a cross-section of the coating [7]. When the coating is new and the protection relies on the barrier mechanism and there is no electrolyte penetration in the coating, the equivalent circuit contains only two elements: capacitance C_c (of an order of tens – hundreds pF) and resistance R_p (several– hundreds GΩ) modeling the properties of the coating material. After the first period of exploitation, the coating loses its protection barrier and the electrolyte penetrates the coating, but adhesive properties are still active and there is no undercoating corrosion. At this stage, the resistance of the electrolyte in the coating pores influences R_p , whose value decreases the more the electrolyte penetrates the coating.

Additionally, the electrolyte penetration causes an increase of the dielectric constant and as a result the increase of the capacitance C_c . In the next stage, the continuity of the coating is broken, undercoating corrosion appears, and the equivalent circuit contains new elements; double-layer capacitance C_{dl} and charge-transfer resistance R_{ct} . As the corrosion expands, the value of R_p still decreases, until finally the coating is destroyed and the equivalent circuit contains only R_{ct} and C_{dl} .

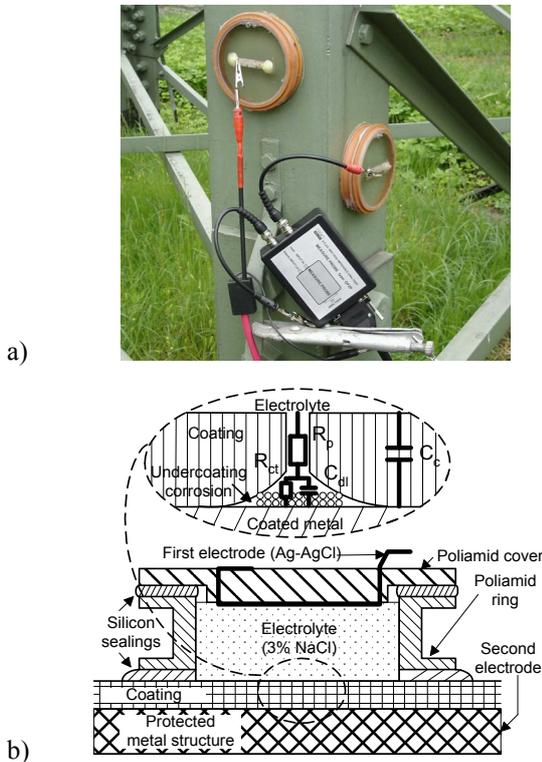


Fig. 2. a) Photograph of the cell for impedance measurement of anticorrosion coating on the high-voltage power line pylon b) Cross-section of the coating

For simulation tests and practical verification of the method, the four-element two-terminal RC network shown in Fig. 3 was used. The configuration and the values of the components are a typical example of the equivalent circuit of high-thickness anticorrosion coating in the early stage of undercoating corrosion induction. This is a very important moment in coating diagnostics, as the fast renovation of the coating can prevent the expansion of corrosion.

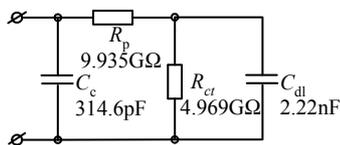


Fig. 3. Schematic diagram of the object under test

4. EVALUATION OF THE METHOD BY SIMULATION

To compare the calculated and real impedance spectra for different parameters of the excitation signal and response sampling, the algorithm of the method presented in Section 2 was evaluated by simulation. The simulation was performed using Matlab for the object presented in Fig. 3.

Figure 4 presents timings of signals $u_1(t)$ and $u_2(t)$ when exciting a two-terminal RC network with a square pulse with 1 V amplitude and pulse widths 0.5 s and 2 s (the assumed pulse width results from the estimation of the object's time constant on the level of 1 s). For a shorter pulse, the voltage $u_2(t)$ on the measured two-terminal network Z_x does not reach the maximum value resulting from the voltage divider created by resistor $R_0 = 1 \text{ G}\Omega$ and the maximum value of the impedance modulus $|Z_x| \cong 15 \text{ G}\Omega$. Resistor R_0 has limited the current flowing through Z_x to 1 nA, so a range resistor $R_R = 1 \text{ G}\Omega$ was used in the current-to-voltage converter, matching voltage $u_1(t)$ to the AD converter operating range.

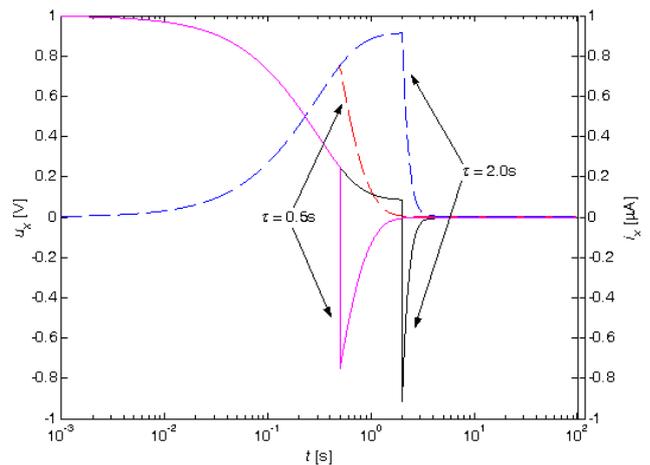


Fig. 4. Current (continuous line) and voltage (dashed line) signals on the tested two-terminal RC network

For the values of resistors R_0 and R_R determined above, using formulas (3-5), the shape of the impedance spectrum of the two-terminal RC network was analyzed depending on a changing square pulse width τ and sampling frequency, assuming a total acquisition time equal to 100 s. The simulation results are presented in Fig. 5, where impedance spectra were shown in the Nyquist plot, for frequencies in range of 0.01 Hz – 1kHz. The continuous line in the graph presents the impedance spectrum calculated theoretically using nominal values of RC components (Fig. 3).

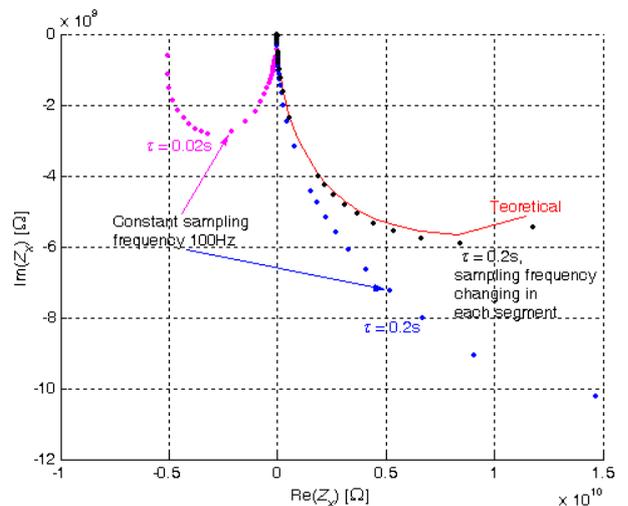


Fig. 5. Impedance spectra in the Nyquist plot

When analyzing curves it can be noted that the spectra obtained from measurement with constant sampling frequency (100 Hz) differ meaningfully from the theoretical one. Much better results were obtained (the spectrum is closest to ideal) in case of sampling with a variable frequency. Due to this fact, in the system presented in Section 2 and in following simulations, the sampling frequency changing in decades in each segment is used.

The presented spectra, due to the wide range of changes of real and imaginary parts of impedance, do not allow to evaluate precisely the quality of their fitting to the theoretical characteristic. Because of this, it was decided to perform the evaluation on the basis of an error of identification of components of the equivalent circuit of the tested object. For parametric identification of components the computer program LEVM [3] was used. LEVM uses the iterative CNLS method to find the function describing a two-terminal RC network with a known structure well fitted to the impedance spectra calculated from (3-5). Relative errors of four identified elements were calculated in relation to square pulse width when sampling with an ADC converter with 16-bit resolution (Fig. 6).

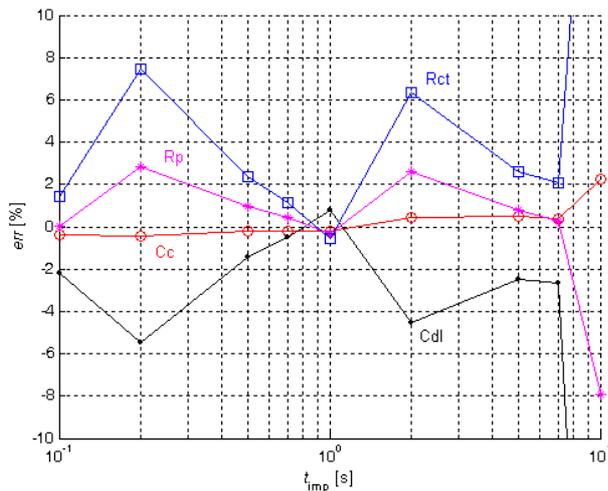


Fig. 6. Error of identification of elements of a two-terminal RC network when sampling with a 16-bit AD converter

The graphs shown the significant influence of the excitation pulse width on determined impedance spectra which were the basis to identify components of the equivalent circuit of the two-terminal network. Relative errors for all identified components reach a clear minimum for a square pulse of $\tau = 1$ s width. This is a time comparable to the time constant of the tested two-terminal RC network. The resolution of the AD converter assumed in the simulation is not always possible to assure. The verification of the method is performed in the system with a DAQ card equipped with 12-bit AD converters (Fig. 1), so the simulation was repeated for a 12-bit converter.

The graphs of errors presented in Fig. 7 show again that the optimal excitation pulse width is equal to 1 s. The accuracy of component determination is worse, especially for C_{dl} and R_{ct} , which are placed deeper in relation to terminals of the two-terminal RC network. A simulation performed with a longer acquisition time (1000 s) has not shown a significant accuracy improvement.

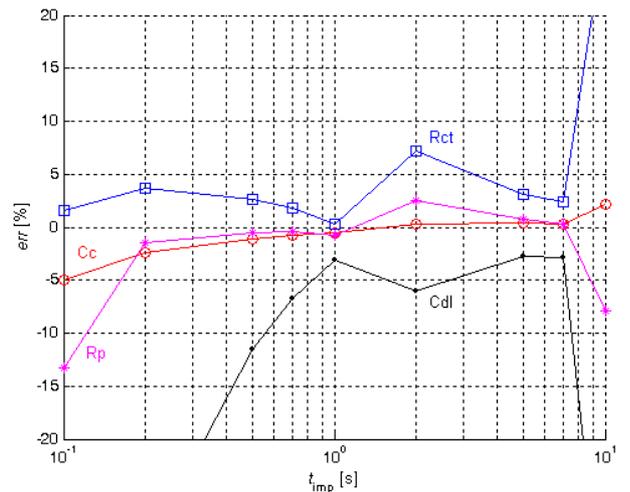


Fig. 7. Error of identification of elements of a two-terminal RC network when sampling with a 12-bit AD converter

The significant increase of identification errors of components C_{dl} and R_{ct} for pulse duration times above 7 s is surprising. This effect can be caused by a too low sampling frequency in the time segment above 10 s. Due to this, the signals $u_1(t)$ i $u_2(t)$ sampling time segments selection was modified. The three time segments during pulse duration were assumed, and the same segments were repeated immediately after the pulse, then two segments were added with the duration time equal to $10 t_{imp}$ and $100 t_{imp}$. Additional sampling segments were entered in order to extend the measured impedance spectrum in the range of low frequencies (from 0.01Hz). The total number of samples collected in modified measurement cycle is equal to 8000. The modifications caused the change of relative error curves (Fig. 8) for pulse duration time greater than 1s. The graphs show that the optimal pulse duration should not be shorter than the time constant of the measured object.

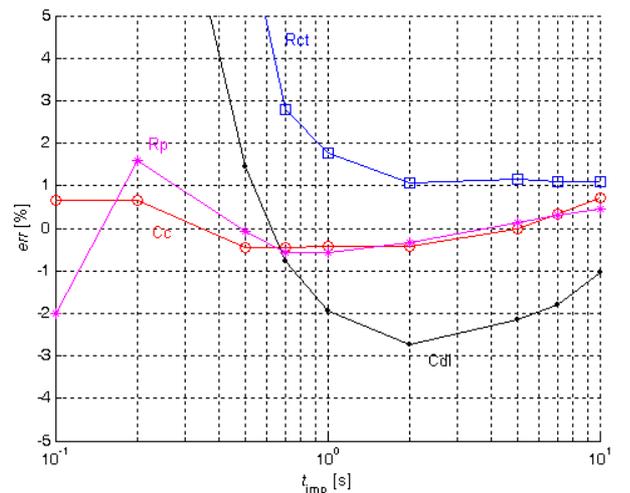


Fig. 8. Relative error of component identification of a two-terminal RC network for a modified measurement cycle

To compare the obtained identification results, optimal results are shown in Table 1 for both resolutions and different methods of time segment selection. For each component value, the standard uncertainty was given as a standard deviation calculated by the LEVM program.

Table 1. Identification results for simulation tests of the object.

		C_c [pF]	R_p [GΩ]	C_{dl} [nF]	R_{ct} [GΩ]
ADC 16bit Basic cycle	Value	314.4	9.968	2.238	4.972
	Err [%]	-0.19	-0.32	0.81	-0.56
	StDev	0.2	0.007	0.008	0.010
ADC 12bit Basic cycle	Value	313.4	9.922	2.151	5.012
	Err[%]	-0.51	-0.78	-3.11	0.24
	StDev	0.5	0.021	0.022	0.027
ADC 12bit Modified cycle	Value	313.6	9.965	2.177	5.088
	Err[%]	-0.44	-0.57	-1.94	1.76
	StDev	0.4	0.016	0.016	0.022

The analysis of Table 1 shows that the proposed method of fast impedance spectroscopy using square pulse excitation allows the identification of components of the tested two-terminal RC network in a time of approx. 100 s, with an uncertainty level comparable to SST spectroscopy. But the measurement time for an impedance spectrum using the SST method performed for frequencies in the range 0.01 Hz – 1 kHz, with 3 points per decade (1-2-5 steps) is equal to 1900 s. The measurement time was calculated assuming that at each frequency the measurement lasts for 10 periods of the signal [8]. A significant (19 times) shortening of the measurement time was obtained, which is extremely useful especially in case of measurements performed directly in the field.

5. EXPERIMENTAL VERIFICATION

Taking into account the conclusions arising from performed simulations, the algorithm of measurement process was developed and implemented in the realized measurement system (Fig. 1). The algorithm contains automatic range selection, excitation pulse generation with optimal time and selection of an appropriate sampling frequency in particular time segments of the measurement cycle. Figure 9 presents a block diagram of the algorithm which is realized in three steps. In the first step, the one of 8 range resistors R_R is chosen (also concurrently R_0) starting from the highest value $R_R = 1 \text{ G}\Omega$ (next R_R : 100 MΩ, 10 MΩ, ... 100 Ω). In order to do this, a test pulse with minimal duration time ($t_{\min} = 0.1 \text{ s}$) is generated and the voltage across the measured object u_2 is sampled. If the value of the voltage u_2 , at the end of the test pulse is greater

than $0.1U_0$ (U_0 – amplitude of test pulse u_0), the range resistor is accepted.

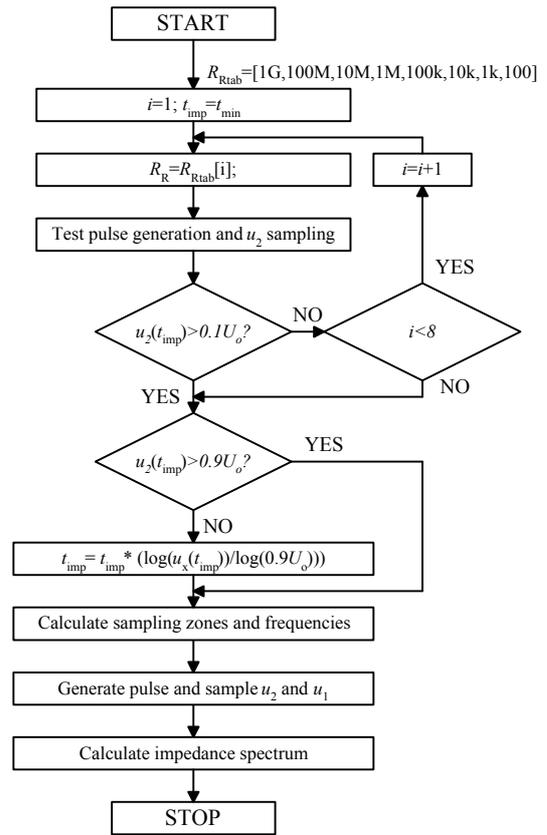


Fig. 9. Algorithm of the measurement process implemented in the system

In the second step, the optimal measurement pulse duration time is selected. The pulse duration is increased by a coefficient arising from an exponential curve to increase the voltage across the object above $0.9 U_0$. In the third step, on the basis of the pulse duration obtained in the second step, time segment limits are calculated as shown in Fig. 10 (the time axis values are given for an exemplary pulse duration of 1 s). Assuming 1000 samples in each time segment, sampling frequencies are then determined for each segment (for the presented example in the first segment the sampling frequency is 100 kHz). Then the pulse is generated and the voltage u_x and current i_x response of the object is acquired (total number of samples is equal to 8000). Finally, the impedance spectrum is calculated at required frequency points using (3-5).

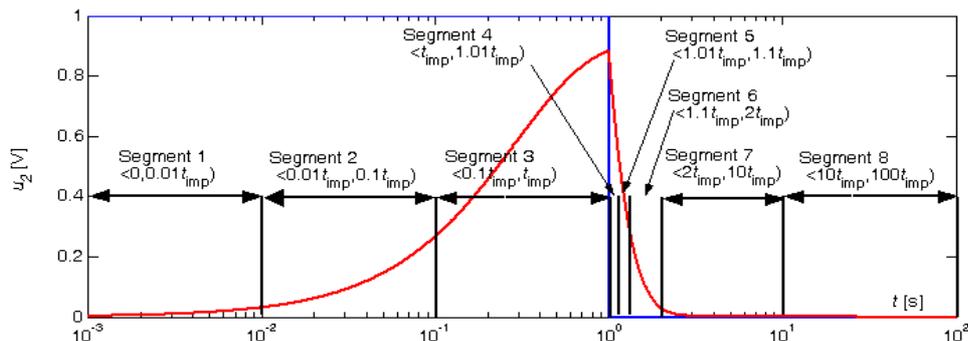


Fig. 10. Time segments of the sampling process in the proposed modified method and the curve of the voltage across the object

The presented algorithm implemented in the measurement system allowed experimental verification of the method. The measurements were performed for the two-terminal RC network (Fig. 3). Signals proportional to voltage (u_2) across and current (u_1) through the measured impedance Z_x , at the output of the input circuitry are shown in Fig. 11. A series of ten measurements of u_1 and u_2 was performed for square pulse excitation with an amplitude of $U_0 = 1$ V and duration time $t_{imp} = 2$ s. The impedance spectra were calculated allowing parametric identification of the object using LEVM software. Table 2 presents mean values of the determined parameters, their relative errors and standard deviation for the measurement series.

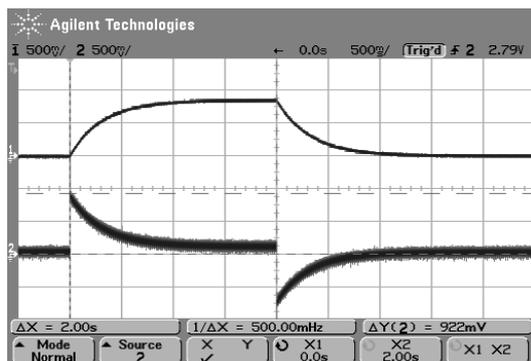


Fig. 11. Oscillogram of u_2 (Ch1) and u_1 (Ch2) in the realized system

Table 2. Identification results from measurement of the object.

		C_c [pF]	R_p [G Ω]	C_{dl} [nF]	R_{ct} [G Ω]
ADC 12bit Modified cycle	Meas.	313,19	9,84	2,09	4,84
	Err [%]	-0,45	-1,00	-5,64	-2,55
	StDev	0,35	0,01	0,01	0,02

The identification errors calculated from measurements are 2-3 times greater (depending on the location of the component in the structure of the two-terminal network) than those obtained from simulations. The error increase is caused by non-ideal parameters of the input circuitry which were not taken into account during simulation. The error sources are difficult to eliminate (e.g. parasitic capacitances) in case of very high impedance measurements. The second cause of the increase of the identification error is the power-line-induced noise with a frequency of 50 Hz which is added to sampled signals u_1 (Fig. 11) extracted in the input circuitry, in spite of careful shielding of the tested object.

The obtained accuracy fulfils requirements for measurements carried out directly in the field. This can be also useful in some cases in the laboratory, when the most important is shortening of the measurement time. So the developed method can be an alternative to classical IS.

6. CONCLUSIONS

A method and the measurement system for fast impedance spectroscopy of objects modeled by multi-element two-terminal networks was developed. The method is based on analysis of the time domain response of the object Z_x after square pulse excitation. The impedance

spectrum of Z_x was determined by transformation of the time domain response of the tested object to the frequency domain using continuous Fourier transformation. The proposed algorithm of transform calculation using a selected sampling frequency for each segment of the acquisition time has shortened the calculation time as well as made possible much better fitting of the obtained spectrum to the theoretical one.

The performed simulation test for a 4-element two-terminal RC network has proved the usefulness of the method for parametric identification of the object's equivalent circuit. The relative error of component determination does not exceed 0.2%-0.8% depending on the placement of the element in the structure of the two-terminal RC network in case of the use of a 16-bit AD converter and has increased to a maximum value of ca. 2% in case of a 12-bit ADC when using a modified selection of time segments in which the response signals are sampled.

In the practical verification of the method in the realized measurement system, a 2-3 times increase of the identification errors was obtained. This is caused by non-ideal parameters of the input circuitry connected to the measured object with very high impedance, and the noise induced in the object mainly by power lines. The obtained accuracy is acceptable for the measurements performed directly in the field. The main advantage of the proposed method is meaningful shortening of the measurement time in relation to classical IS. For the tested object, the measurement time was shortened from approx. 31 min. in the case of the Solartron set (Impedance Interface 1294 and Frequency Response Analyzer 1255) to approx. 1 min. 40 s.

The good results of simulation and verification directed the authors to further improvements of the method (e.g. increase of sampling frequency to average samples to eliminate noise) and to realize a system using 16-bit ADCs.

REFERENCES

- [1] E. Barsoukov, J. R. Macdonald, "Impedance Spectroscopy: Theory, Experiment and Applications", J.Wiley&Sons, 2005.
- [2] J. Hoja, G. Lentka, "Virtual instrument using bilinear transformation for parameter identification of high impedance objects", Meas. Sci. Tech., Vol. 14, No. 5, pp. 643, May 2003
- [3] J. R. Macdonald: LEVM Manual ver.7.11. CNLS Immittance Fitting Program. Solartron Group Limited 1999.
- [4] R. Bragos, R. Blanco-Enrich, O. Casas, J. Rosell, "Characterisation of Dynamic Biologic Systems Using Multisine Based Impedance Spectroscopy", Proceedings of the IEEE IMTC, pp. 44-47, Budapest, Hungary, 2001.
- [5] E. Barsoukov, S. H. Ryu, H. Lee, "A novel impedance spectrometer based on carrier function Laplace transform of the response to arbitrary excitation", J. Electroanal. Chem. Vol. 536, pp. 109-122, 2002
- [6] J. Smulko, K. Darowicki, A. Zielinski, "On electrochemical noise analysis for monitoring of uniform corrosion rate", IEEE TIM., Vol. 56, No 5, pp. 2018-2023, 2007.
- [7] J. Bordzilowski, K. Darowicki, S. Krakowiak, A. Krolikowska, "Impedance measurements of coating properties on bridge structures", Progress in Organic Coatings, Vol. 46 pp. 216-219, 2003.
- [8] Solartron: Frequency Responce Analyser 1255 Operating Manual, Dec. 2000. High Impedance Interface 1294 Operating Manual, Dec. 2001