Application of Impedance Spectroscopy for Electrical Characterization of Ceramics Materials

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***Abstract*—The choice of the most suitable equivalent electrical circuit to model the impedance response of lanthanum doped $BaTiO_3$ ceramics was emphasized in this paper. The method for the determination of the starting values for the basic equivalent circuit was also presented. These starting values were used in the fitting procedure of experimental impedance data with simulated data in order to select the most correct and appropriate equivalent circuit**.

***Index Terms*—$BaTiO_3$ ceramics, Impedance spectroscopy, Equivalent circuit.**

I. Introduction

IMPEDANCE spectroscopy (IS) offers an effective method for the characterization of electrical properties of electroceramics especially of ferroelectric materials such as $BaTiO_3$ based ceramics [1-4]. Having in mind that these materials are used in polycrystalline form, which is characterized primarily by the grain boundaries and grains (bulk), it is necessary to undesrtand the role and the effects of different microstructural features on the overall electrical properties of materials [3-5]. The resistance and capacitance values of bulk and grain boundary, which are frequency and temperature dependent, can be evaluated from IS spectra. Experimental data can be analysed through four possible complex formalisms that represent the same information yet expressed in various ways. Data can be represented through electrical impedance Z^*, electric modulus M^*, admittance Y^*, and dielectric permittivity ε^*. The relationship between them is given as

$$M^* = j\omega C_0 Z^* = j\omega C_0 \left(1/Y^*\right) = 1/\varepsilon^* \qquad (1)$$

where: $\omega = 2\pi f$ is angular frequency, $C_0 = \varepsilon_0 S/d$ vacuum capacitance of the cell without sample, ε_0 is the permittivity of free space, $8.85\text{x}10^{-12}$ F/m, and S and d are area and thickness of the sample, respectively.

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To extract the informations about the resistive and capacitive values of grain and grain boundary regions it is necessary to adopt such electrical model that most precisely represents electrical characteristics of the considered material at different frequencies and at different temperatures. The choice of the adequate model subsumes that the acquired simulation results have to be consistent with experimental data, and is largely based on designer's experience. The acquired values of the model elements have to be real, logical and expected.

The main objective of this study is the strategy of choosing the correct model for the interpretation of IS data. The comparison between models, represented through equivalent electrical circuits, regarding their precision and possibilities of models for optimal fitting of experimental data, is also emphaised. For impedance spectroscopy measurements $BaTiO_3$ doped with lanthanum (La-BT) was used as an example. La-doped $BaTiO_3$ was sintered at1300°C for 2 hours. Impedance measurements were made after the samples had been placed in a furnace at selected temperature for 30 min. IS measurements were perfomed at room temperature, 200, 300 and 350°C in a frequency range from 20 Hz to 1.0 MHz using LCR-meter Agilent 4284A [6].

The multidimensional Simplex identification algorithm and in-house made software were used for fitting and simulation process of IS data [6,7].

II. Basic Equivalent Circuit to Model IS Data of La-$BaTiO_3$ Ceramics

In modeling of IS response for doped and undoped ceramic it is possible to chose more than one equivalent circuit that contains at least the bulk and grain boundary elements [1,3,4]. Therefore, at the very beginning of the analysis the solution is not straightforward. Numerically it is possible for simulated curves to coincide with experimental data by applying different models. However, only one of those solutions represents a real model of electrical behavior of ceramic materials. Concerning $BaTiO_3$ ceramics, the total ceramic impedance is mainly influenced by grain impedance and grain boundary impedance. Hence, it is assumed that impedance data can be presented with the most simple, yet often satisfying basic equivalent circuit consisting of two parallel resistorcapacitor (*RC*) elements connected in series, as shown in Fig.1, [1,3]. One of these impedances relates to the grain-boundary (R_1,C_1) and the other to the grain (bulk) (R_2,C_2).

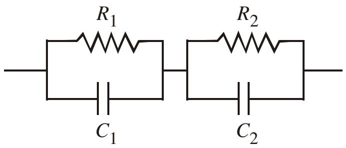


Fig. 1. Basic equivalent circuit to model IS of La-BT ceramics

In the case where the contact resistance of the sample-electrode interface can not be ignored, it is necessary to take into consideration another (*RC)* element connected in series with two other mentioned elements. Since the contribution of sample-electrode interface impendance to the overall IS response is usually very small, in this analysis it is not taken into account.

Complex impedance for circuit given in Fig.1 is:

$$Z^{*} = Z' - jZ'' = \frac{1}{1/R_1 + j\omega C_1} + \frac{1}{1/R_2 + j\omega C_2} \quad (2)$$

where:

$$Z' = \frac{R_1}{1+(\omega R_1 C_1)^2} + \frac{R_2}{1+(\omega R_2 C_2)^2} \quad (3)$$

$$Z'' = R_1 \frac{\omega R_1 C_1}{1+(\omega R_1 C_1)^2} + R_2 \frac{\omega R_2 C_2}{1+(\omega R_2 C_2)^2} \quad (4)$$

On the basis of expression $M^{*} = j\omega C_0 Z^{*} = M' + jM''$ the components of electrical moduls are easily obtained:

$$M' = \frac{C_0}{C_1}\frac{(\omega R_1 C_1)^2}{1+(\omega R_1 C_1)^2} + \frac{C_0}{C_2}\frac{(\omega R_2 C_2)^2}{1+(\omega R_2 C_2)^2} \quad (5)$$

$$M'' = \frac{C_0}{C_1}\frac{\omega R_1 C_1}{1+(\omega R_1 C_1)^2} + \frac{C_0}{C_2}\frac{\omega R_2 C_2}{1+(\omega R_2 C_2)^2} \quad (6)$$

In order to estimate the *RC* values for each element it is necessary to observed concurrently the impedance Z^* and electrical modulus M^* of the samples. This is because each parallel *RC* element contributes to the shape of impedance spectra in a different way, most often in the shape of semicircular arcs in $Z''(Z')$ and $M''(M')$ complex plane or in the shape of peaks in spectroscopic plots of the imaginary components $Z''(\log f)$ and $M''(\log f)$ which will be discussed later. In addition, to get a straightforward model, valuated both at different frequencies and different temperatures, the influence of individual *R* and *C* parameters onto the impedance and electrical modulus of the discussed ceramics, have to be considered. The influence of *RC* parameters on the impendance shape is illustrated in Figs. 2 and 3.

Fig. 2 shows the imaginary and real parts of impedance $Z''(Z')$ in complex plane for the case where the same capacitance values (C_1=C_2) are chosen, while the value of resistance R_2 remains constant and resistance R_1 changes. The analysed characteristic at such conditions contains only one set of semicircles whose shape and dimensions are determined by dominant resistance (in this case R_1). In the case of different *C* values, C_1,<C_2 of semicircles lines appear on impedance complex plane, as shown in Fig. 3. The shape of curves is determined by the dominant value of resistance, here that is also resistance R_1.

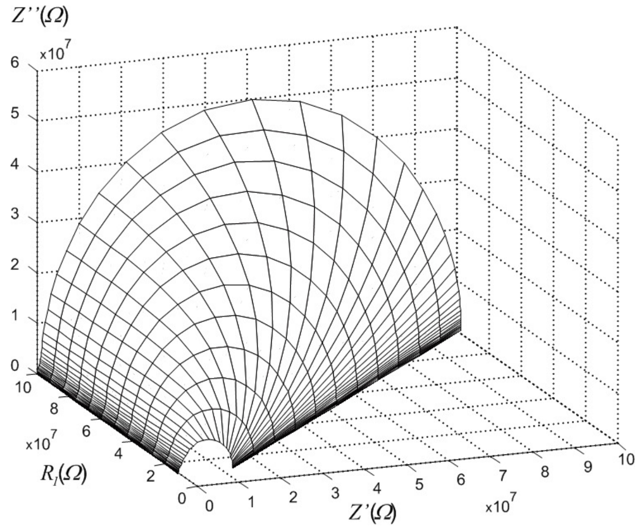


Fig. 2. Impedance of equivalent circuit from Fig.1 shown in complex plane Z"(Z'), where: C_1=C_2=10^{-12}F, R_2=10^6Ω, and R_1 changes in the range from $5x10^6$Ω to 10^8Ω

By the analysis of electrical modulus of impedance $M''(M')$, in function of relative relation of equivalent circuit elements, the similar results were obtained, as given in Figs. 4 and 5.

Fig. 4 presents the analysis of electrical impedance modulus in the case of equal resistance values R_1=R_2, while capacitance value C_2 changes at fixed capacitance value C_1. In this case only one set of semicircle lines is obtained whose shape and size are primarily determined with that *RC* element which has lower capacitance value (now that is C_2 capacitance). In other words, if one capacitance is much higher than another, semicircle arc attached to it in complex plane *M''(M')* is lost. Fig. 5 shows an example of electrical modulus in the case of mutually different fixed resistance values R_1 and R_2, at the change of C_2 capacitance (C_1 is also fixed). In this case the considered charachteristic contains two sets of semicircle arcs. Here too, the dominant effect onto the shape and appereance of secondary arc has the *RC* element with smaller capacitance value (that is C_2).

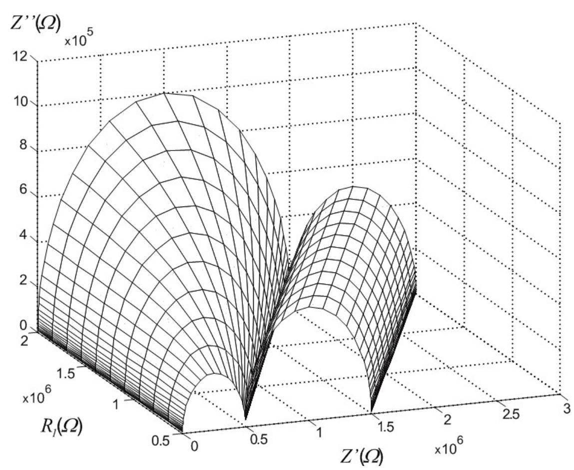


Fig. 3. Impedance of equivalent circuit from Fig.1 shown in complex plane Z"(Z'), where: C_1=10^{-12}F, C_2=10^{-9}F, R_2=10^6Ω, and R_1 changes in the range from $0.5x10^6$Ω do $2x10^6$Ω

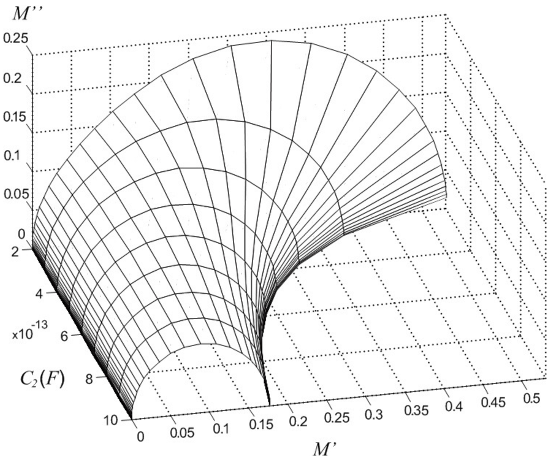


Fig. 4. Electrical modulus of equivalent circuit from figure 1 shown in complex plane M''(M'), where: $R_1= R_2=10^6\Omega$, $C_1=10^{-12}$F, and C_2 changes in the range from $0.2x10^{-12}$F to 10^{-12}F

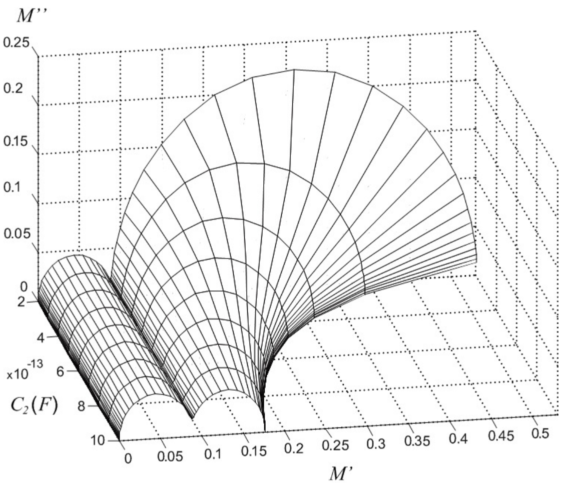


Fig. 5. Electrical modulus of equivalent circuit from figure 1 shown in complex plane M''(M'), where: $R_1=10^8\Omega$, $R_2=10^6\Omega$, $C_1=10^{-12}$F, and C_2 changes over the range $0.2x10^{-12}$F to 10^{-12}F

The previous analysis demonstrates the usefulness of concurrent visual inspection of spectroscopic plots of $Z''(Z')$ and $M''(M')$. It has to be said that this comparison is not always simple, because with the increase of frequency the dots on curves in $Z''(Z')$ complex plane, move towards coordinate center (Figs. 2,3), while with the increase of frequency, the dots on curves in $M''(M')$ complex plane, move away from coordinate center (Figs. 4,5).

III. DETERMINING THE VALUES OF THE BASIC EQUIVALENT CIRCUIT ELEMENTS

During determining of equivalent circuit elements, based on experimental impedance spectra, various cases can occur. It should be noted that most often only modulus and the angle of their impedance are measured for ceramic materials. On the basis of these measurements, it is very demanding to distinguish values of certain elements of equivalent circuit. Which of *RC* elements, of the basic equivalent circuit from Fig. 1, will be detected depends on the characteristic being considered, as well as on relative relation of the equivalent circuit elements values, as analysed already in Figs. 2-5. While impedance characteristic is more appropriate for determining *RC* element with higher resistance, electric modulus is more appropriate for determining *RC* element with lower capacitance.

Here follows an example of determining the model's parameters for basic equivalent circuit, using experimental IS data obtained for La-doped $BaTiO_3$ ceramics. Because of the limited frequency range of equipment, the IS spectra obtained at lower temperature are incomplete, whereas spectra obtained at higher temperatures showed satisfactory quality. Measurments perfomed at 350^oC over the frequency range from 20 Hz to 1.0 MHz are used in further analysis.

Three-dimensional (3D) image of IS spectrum, obtained at 350^oC, together with two-dimensional (2D) projections of imaginary $Z''(\log f)$ and real parts $Z'(\log f)$) of impedance in a function of frequency, and also 2D $Z''(Z')$ impedance in a complex plane, are given in Fig. 6.

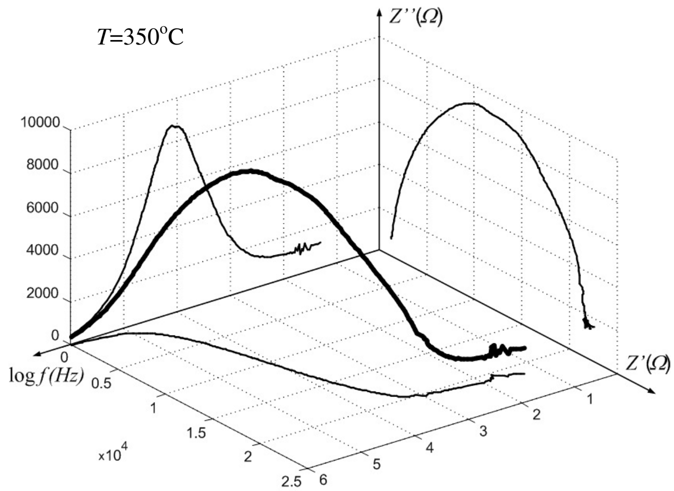


Fig. 6. 3D image of experimental impedance characteristic of the measured sample and 2D images of its projections

For the sake of clarity, measured impedance spectrum $Z''(Z')$ in a complex plane, for the same La-BT sample, is again presented in Fig. 7 (the curve is denoted with open circles). On the basis of visual inspection of IS spectrum shown in Fig.7 with those shown in Figs. 2 and 3, it can obviously be concluded that this is the case when $R_1 » R_2$, as the measured IS spectrum contains only one semicircle arc. Applying the basic equivalent circuit, given in Fig. 1, the simulated spectrum is obtained and presented with curve (a) in Fig. 7. The total resistance of the circuit can easily be read from the curve, and it is $R_u=R_1+R_2 \approx R_1=21$ kΩ. The presented spectrum allows for the determination of only total resistance of sample R_u.

Determination of other circuit parameters includes a consideration of the imaginary components of impedance and electric modulus $Z''(\log f)$ and $M''(\log f)$ as a function of frequency (Fig. 8). By direct comparison of spectroscopic plots, obtained on 350^oC, it can be seen that electric modulus $M''(\log f)$ has two peaks over the investigated frequency range, and that lower frequency $M''(\log f)$ peak coincides to that of $Z''(\log f)$ peak. The peaks in the $Z''(\log f)$ and $M''(\log f)$ plots are known as Debye peaks. It can also be noted that the advantage of the combined analysis of both dependences is in the fact that the peaks of these characteristics, that correspond to each of *RC* elements, can overlap on frequency scale, as is the case here with $M''(\log f)$ and $Z''(\log f)$ peaks (Fig.8). In this particular case both *RC* elements determine the electric modulus peaks, while the impedance peak is determined by the element with higher resistance. The frequency at the Debye peak maxima for each *RC* element is given by the expression $2\pi f_{\max} RC = 1$.

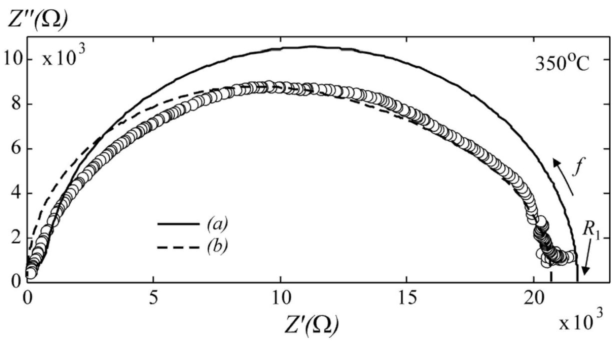


Fig. 7. Experimental AC impedance spectrum in complex plane (presented with open circles) for La-BT at 350°C, modelled impedance spectrum using the calculated values of circuit elements (a) and for fitted values (b)

Capacitance values in equivalent circuit can more precisely be determined by using the spectroscopic plot of electric modulus in a complex plane, shown in Fig. 9. That spectrum presents the connection between two incomplete semicircles arcs and corresponds to the case given in Fig. 5, where $R_1 » R_2$, and C_2 is somewhat higher than C_1. Therefore, in this way, according to graphs from Figs. 7 and 8, and their comparison with Figs. 2 to 5, relative ratios of equivalent circuit elements are determined. That is, it is assumed that the equivalent circuit from figure 1 is satisfactory for application in this case too.

Capacitance values can be estimated from the intercept of idealized M'' curve (presented by broken line in Fig. 9) with M' axis: In the concrete case, according to Fig. 8, $M''(\log f)$ maximum correspond to $\log f_{1max}$=4.16, and:

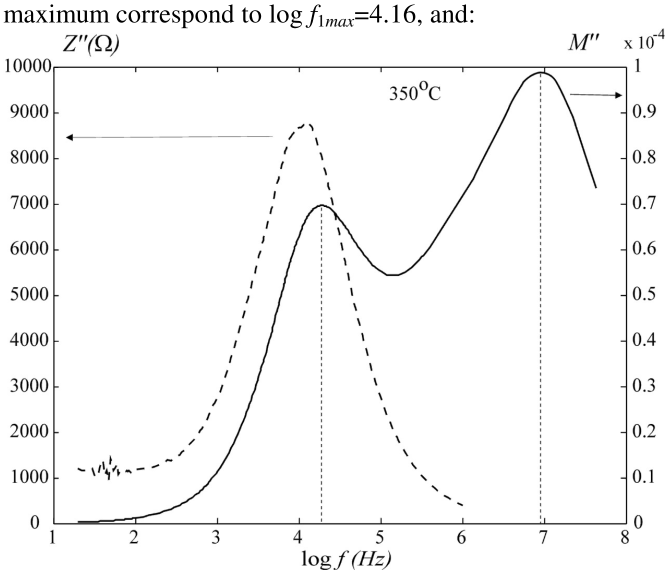


Fig. 8. Impedance Z'' and electric modulus M'' of the measured sample in funcion of frequency

$$C_1 = \frac{1}{2\pi f_{1\max} R_1} = 524.33 \text{ pF} \quad (7)$$

$$C_1 = 8.85 \cdot 10^{-14} / 1.7 \cdot 10^{-4} = 520.59 \text{ pF} \quad (8)$$

which is close to the value calculated by equation 7.
From Fig. 9, it also follows that:

$$8.85 \cdot 10^{-14} \left(\frac{1}{C_1} + \frac{1}{C_2} \right) = 5.7 \cdot 10^{-4} \quad (9)$$

so the capacitance C_2=220.58 pF.

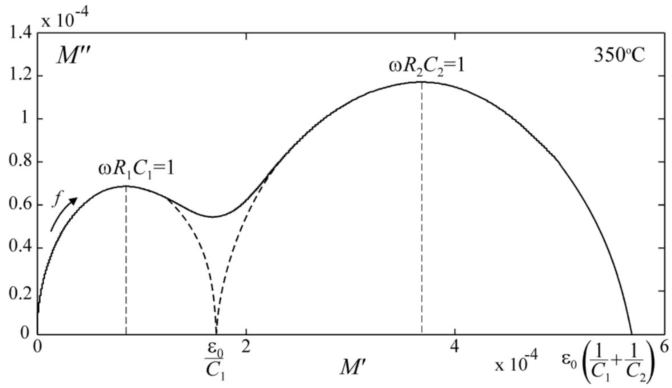


Fig. 9. Experimental characteristic of impedance Z'' and electric modulus M'' of the measured sample as a function of frequency

To determine the resistance (R_2) there is a need again for the combined analysis of dependence $M''(\log f)$ from Fig. 8 and $M''(M')$ from Fig. 9. The resistance R_1, already calculated from Fig. 7, is approximately equal to R_u=21Ω. The R_2 value can be calculated using expression $2\pi f_{2\max} R_2 C_2 = 1$. As from Fig. 8: $\log f_{2max}$=6.9, and C_2=220.58 pF, the required resistance value for R_2 is R_2=90.83Ω.

The presented algorithm of calculating the basic circuit elements can be used for IS spectra obtained at any temperature. The frequency dependence of M'' values for La-$BaTiO_3$ ceramic sample at selected temperatures is given in Fig. 10.

At lower temperatures only one peak can be noted on a M'' plots, however, at higher temperatures this peak has a tendency to split into two peaks whose positions move towards higher frequences with temperature increase. The lower frequency peak has approximately the same amplitude which indicates that capacitance that determines it does not change with the increase of temperature, while higher frequency peak rises with temperature increase, which means that capacitance that determines it decreases with the increase of temperature. These results show that at higher temperatures both parallel *RC* elements significantly contribute to equivalent circuit impedance, as then differences between *RC* elements are greater. At lower temperatures the differences between *RC* elements are smaller, and only one frequency peak characterizes Z' and M' plots, as noted earlier in Figs. 2 to 5.

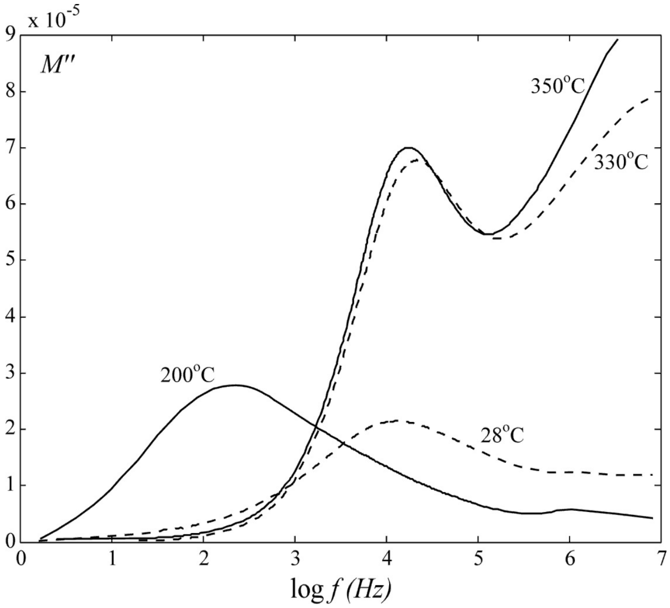


Fig. 10. Electric modulus M" of sample as a function of frequency for selected temperatures

By applying the previously described procedure, the approximate values of equivalent circuit elements can be gained for any of selected temperature. In accordance with previously presented analysis, it can be assumed that the equivalent circuit from Fig. 1 is highly real. It can also be assumed that elements R_1C_1 and R_2C_2 represent two different part in microstructure of ceramics. It is very well known that grain boundary resistance is higher compare to grain/bulk, so it is for believe that R_1C_1 and R_2C_2 elements of basic equivalent circuit, (Fig.1), correspond to grain boundary and grain region respectively. To illustrate the extent of accuracy of this way of modelling, the simulated spectrum $Z''(Z')$, using already calculated parameters R and C, is shown as a curve (*a*) in Fig. 7. It is obvious that there is a significant deviation of modelled and experimental IS spectrum, because with the previous procedure only approximate values of equivalent circuit elements could be obtained.

One of the method to increase the accuracy and precision in determination of *RC* values is to use a method of models parameters identification by means of fitting experimental and theoretical data using multidimensional identification algorithm, such as multidimensional Simplex method [6,7]. The $Z''(Z')$ plot, presented as a curve (*b*) in Fig. 7, was obtained by applying this fitting method, and the corresponding R and C values were as follows: R_1 =14.80 kΩ, C_1 = 714.63 pF, R_2 =5.93 kΩ, C_2 = 7.98 nF.

The discrepeance between experimental and simulated IS spectra (Fig.7) leads to the conclusion that the basic equivalent circuit needs adjusting, or that there needs to be applied a more complex model than the usual one given in Fig. 1. On the other hand, it can be concluded that impedance $Z''(Z')$ in reality does not have the shape of ideal semicircle arc. Due to that, here follows the consideration on the possibility of applying different equivalent circuit models capable for better simulation of impedance spectra of doped $BaTiO_3$ ceramics.

IV. ANALYSIS OF MORE COMPLEX CIRCUIT MODELS

Impedance response of ceramic materials could be rarely presented by an ideal semicircle arc and treated of simple parallel *RC* elements. As already indicated, for doped $BaTiO_3$ taken as an example, the arc in complex impedance Z'' vs. Z' dependence is not ideal (Fig. 7). For a low frequency range a semicircle arc is depressed with its centre below the real axis. To model the IS spectra that contain the semicircle arc and distorted part of this arc, a modified equivalent circuit is proposed and corresponding simulated impedance spectra are given in Fig. 11. It has to be noted that a basic equivalent circuit, that can be presented with two parallel *RC* elements serially connected refers to the ideal "Debye" like ceramic behavior. In the real case the ceramic materials do not manifest the ideal "Debye" like behavior, thus to illustrate more fully the deviation from an ideal capacitor, an additional element with constant phase (*CPE*) is introduced into equivalent circuit [3,4]. *CPE* element is used to explain phenomena on the grain boundary areas on one side, and phenomena relating to unhomogeneity, diffusion processes or stress that occurs in the sample, on the other side. For the non ideal "Debye" like behavior in ceramic materials, *CPE*-element is introduced mainly instead of ideal capacitor or as an addition to the parallel *RC* connection. Impedance of *CPE*-elements can be determined as:

$$Z^*_{CPE} = [A(j\omega)^n]^{-1} \tag{10}$$

Where A is a constant that is independent of frequency, and n is an exponential index that is the measure of distortion of $Z''(Z')$ characteristics. For an ideal "Debye" like behavior, it stands that n=1, and *CPE* represents an ideal capacitor with the value $C=A$. The value of n bellow 1 indicates that the capacitor is frequency dependent. For n=0, *CPE* behaves like pure restistance with the value $R=1/A$.

The relation between parameters of equivalent circuit (R, n, A) and curve parameters (x, y, ω_{max}) are shown in Fig. 11.

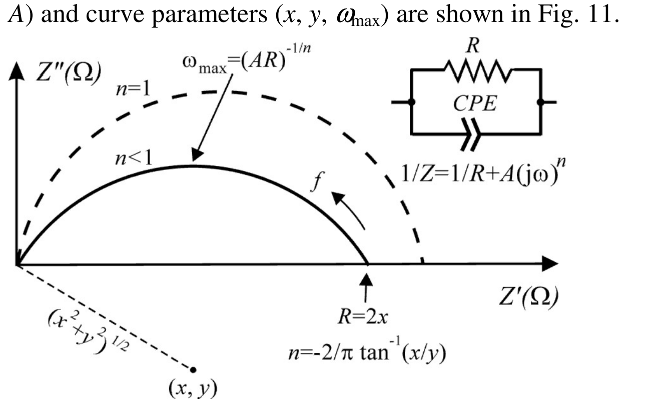
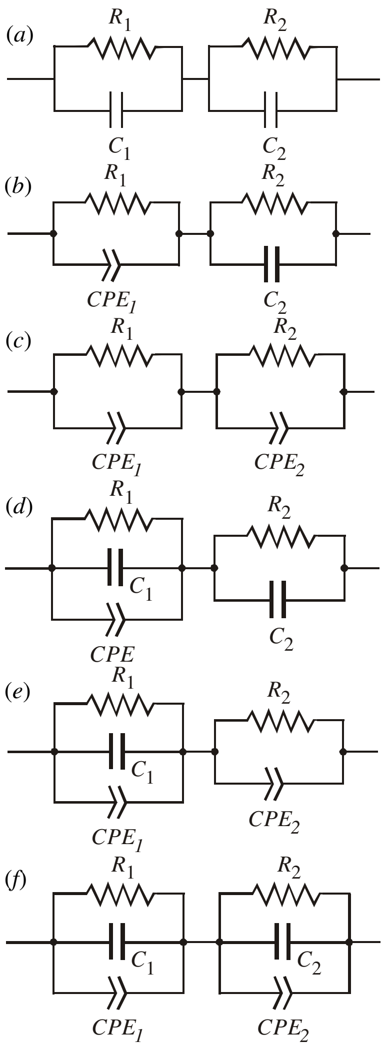


Fig. 11. Modelled complex impedance characteristic for equivalent R-CPE circuit, shown as inset in the image

The calculated values of all impedance formalisms of an equivalent circuit, given in expression (1) have to be identical with the values of impedance formalisms as measured on the sample. Several different equivalent circuits can be suggested for the same sample and the choise of the satisfactory equivalent circuit has to be realised on the basis of additional information.

Besides the basic circuit, with two parallel *RC* elements, the five more equivalent circuits with *CPE* elements have been chosen for the purpose of modelling impedance characteristics for La-BT ceramics. The proposed electrical circuits together with the expression for total impedance Z^* are given in Fig. 12. Multidimensional Simplex identification algorithm, realized with Matlab tools, [6,7] was used for fitting procedure and determination of model' s parameters.



(a) $$Z^* = 1/\left(1/R_1 + j\omega C_1\right) + 1/\left(1/R_2 + j\omega C_2\right)$$
R_1=14.80kΩ, R_2=5.93kΩ, C_1=714.63pF, C_2=7.98nF

(b) $$Z^* = 1/\left(1/R_1 + A_1\left(j\omega\right)^{n1}\right) + 1/\left(1/R_2 + j\omega C_2\right)$$
R_1=12.88kΩ, R_2=9.22kΩ, A_1=22.52nF, C_2=1.31nF, n_1=0.75

(c) $$Z^* = 1/\left(1/R_1 + A_1\left(j\omega\right)^{n1}\right) + 1/\left(1/R_2 + A_2\left(j\omega\right)^{n2}\right)$$
R_1=13.55kΩ, R_2=8.57kΩ, A_1=1.63nF, A_2=63.16nF, n_1=0.95, n_2=0.71

(d) $$Z^* = 1/(1/R_1 + j\omega C_1 + A_1\left(j\omega\right)^{n1}) + 1/(1/R_2 + j\omega C_2)$$
R_1=13.22kΩ, R_2=9.11kΩ, C_1=394.11pF, A_1=56.56nF, C_2=1.41nF, n_1=0.64

(e) $$Z^* = 1/\left(1/R_1 + j\omega C_1 + A_1\left(j\omega\right)^{n1}\right) + 1/\left(1/R_2 + A_2\left(j\omega\right)^{n2}\right)$$
R_1=22.78kΩ, R_2=1.88kΩ, C_1=698.33pF, A_1=894.12nF, A_2=23.68nF, n_1=0.27, n_2=0.79

(f) $$Z^* = 1/\left(1/R_1 + j\omega C_1 + A_1\left(j\omega\right)^{n1}\right) + 1/\left(1/R_2 + j\omega C_2 + A_2\left(j\omega\right)^{n2}\right)$$
R_1=21.32kΩ, R_2=365.14Ω, C_1=424.46pF, C_2=223.16pF, A_1=11.75nF, A_2=219.68nF, n_1=0.67, n_2=0.63

Fig. 12. Various equivalent circuits to model La-BT ceramic

The impedance formalisms for all analysed circuits are shown in Fig. 13. As can be seen from Fig. 13, a fairly good agreement between experimental and modelled results, for all impedance formalisms, is obtained by applying an equivalent circuit with two parallel *R-CPE* elements that are serially connected, i.e., the circiut denoted as circuit (c) in Fig. 12. To determine model's parametes of all circuits given in Fig. 12, the initial values of each *RC* elements or *R-CPE* parallel connection are established according to the procedure from chapter 3. The initial values for *A* and *n,* in circuits 12*b* to 12*f,* are established on the basis of measured data from the real part of admittance *Y'* [3]. The obtained initial parameters values, for all applied circuits, have been refined to get the best possible fitting of experimental data.

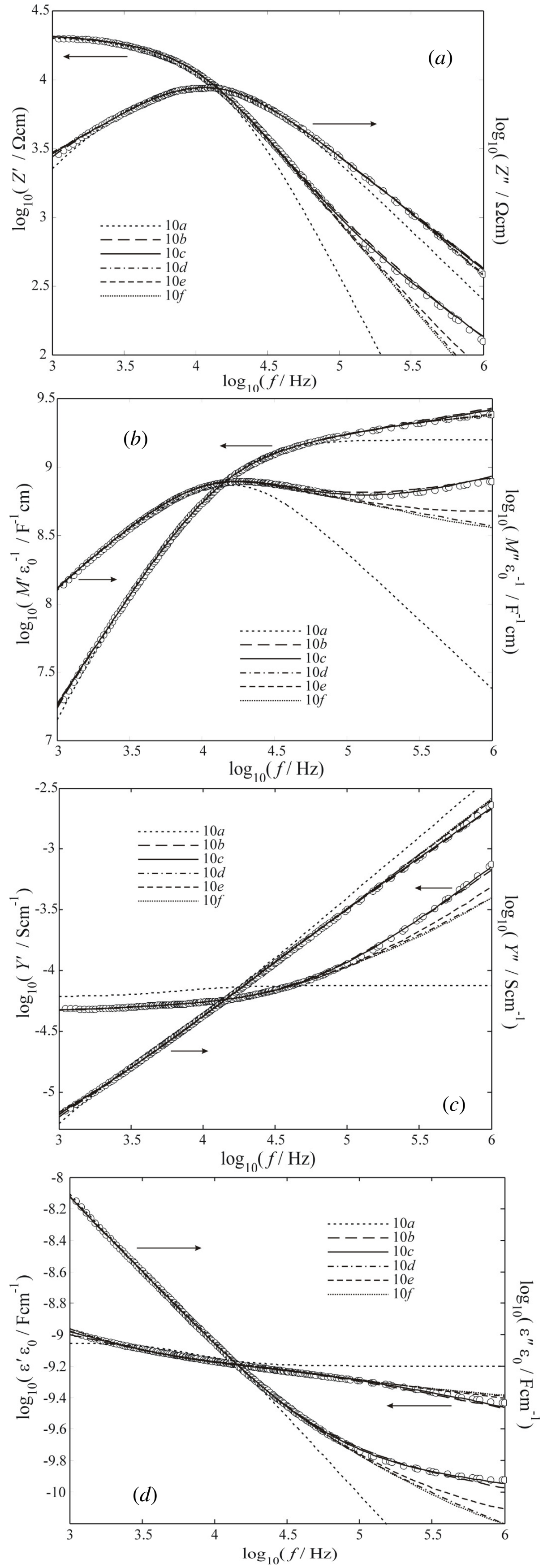


Fig. 13. Normalised frequency dependences of real and imaginary impedance parts for (a) Z^*, (b) M^*, (c) Y^* and (d) ε^*, in log-log ratio, measured at 350°C (circles), and modeled results obtained through best fitting for equivalent circuits presented in figure 12 (colored lines)

Typical results obtained through fitting procedure of equivalent circuits parameters are shown with a full line, while experimental data are marked with open circles. The significant deviation, at lower and higher frequencies, between experimental and simulated values, was obtained with equivalent circuits (*a*), (*d*) and (*e*), in comparison with results gained with other circuits, especially as related to the circuit (*c*) given in Fig. 12.

III. CONCLUSION

The impedance responce of La-doped $BaTiO_3$ ceramics were interpreted in terms of basic equivalent circuit with two *RC* elements connected in series and another five proposed equivalent circuits that contain resistors, capacitor and *CPE* elements. By the analysis of equivalent circuit impedance, combined with the analysis of experimental data of electrical modulus, it was possible to extract the resistance and capacitance contribution of grain and grain boundary regions to the overall electrical properties of doped $BaTiO_3$ ceramics. The method of determining initial values of basic eqivalent circuit elements of ceramic materials was presented. For the basic equivalent circle these values presented the initial parameters during the process of fitting the experimental and simulated impedance in order to get a more precise model.

Final adjustment of model parameters was performed by identification of unknown parameters applying multidimensional Simplex algorithm realised in Matlab. Satisfactory agreement between theoretical and experimental results was gained by applying an equivalent circuit that has two parallel *R-CPE* elements serially connected. The circuit paramer n, being lower than one, $0.95 \leq n \geq 0.63$, highlights the capacitive nature of grain and grain boundary. The grain boundary resistance is higher than the same one of grains.

There is also a discussion on the applicaton and justification of application of several more complex models of this ceramic. Better investigation results would be obtained with an analysis in the wider frequency range than the available one in this case.

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