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FABRICATION AND THE EFFECTS OF THE EXPERIMENTAL STRUCTURE FACTOR ON BARIUM TITANATE FERROELECTRIC PROPERTIES USING POLYDOMAIN AND LANDAU — KHALATNIKOV MODELS

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Abstract

In this report, powder of barium titanate ($BaTiO_3$) was fabricated using the sol-gel method followed by a sintering process at 900 °C. X-ray diffraction (XRD) was carried out to determine the phase and the crystal structure of the barium titanate. XRD patterns were then refined using the Rietveld analysis method on *Fullproof* software. The results show the barium titanate ($BaTiO_3$) phase has a tetragonal perovskite structure. EDAX and scanning electron microscope (SEM) were examined to analyze the chemical composition and morphology of the material powder respectively. The results showed that the powder barium titanate ($BaTiO_3$) contains Ba, Ti, and O atoms. The crystallite size is 3 μm and the morphology look homogeneous. The structure factor obtained from the experiment is used to calculate the polarization hysteresis using Polydomain and Landau — Khalatnikov models. The results of modeling using the structure factor approach using Landau — Khalatnikov modified model and the Polydomain modified model showed the *R*-factor (R_{wp}) is 8.7 and 11.3 %

Keywords

Barium titanate, ferroelectric, Polydomain model, Landau — Khalatnikov model, structure factor

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Introduction. The phenomenon of ferroelectric has developed rapidly since the discovery of the polarization phenomenon in Rochelle salt more than 100 years ago [1]. Rapid developments occurred in the research and development

of ferroelectric materials based on $PbTiO_3$ (PZT) materials with various applications such as a capacitor, transducers, generators, sensors, actuators, transducers for different purposes and RAM (Random Access Memory) applications [1–7]. Since the restriction regulation of the use of the lead base for electronic application devices in 2002, barium titanate has attracted attention as a substitution for lead titanate (PZT) [8]. Barium titanate is a perovskite oxide material with an ABO_3 structure with the cation Ba^{2+} occupying position A and Ti^{4+} occupying position B at the center of the cell and surrounded by O^{2-} the polarization of the cell forming the material occurs.

Several research conducted by researchers has investigated that barium titanate has several advantages, such as its high dielectric constant, and low dielectric loss [4, 5–7, 9–12]. Buscaglia *et al* [3] reported that the maximum relative dielectric constant of $BaTiO_3$ occurred when the grain size dimension was 100 nm. Dudem *et al* [4] reported that barium titanate micro stone-like architectures ($BaTiO_3$-MSs) composite with polyvinylidene difluoride (PVDF) caused enhancement of a high open-circuit voltage (VOC) and short-circuit current (ISC) properties for piezoelectric nanogenerators (PNG) application. Another research has been reported by Zheng *et al* [5] that the piezoelectric coefficient (d_{33}) of $BaTiO_3$ nanofiber decreased with increasing Ca^{2+} and Mn^{4+} but was still higher than the piezoelectric of native bone. Wang *et al* [6, 7] on their report showed that reducing the leakage current of $BaTiO_3$ doped by strontium (BST) thin film occurred when BST doped by Au or codoping by Mg/La. Butee *et al* [9] reported $BaTiO_3$ doped by Pb caused an enhancement of 1.5 times of saturation polarization and six times of remanent polarization and also increasing of d_{33} properties from 95 pC/N for $BaTiO_3$ to 220 pC/N for $BaTiO_3$ doped by Pb with the composition of $x = 0.15$. Mehta *et al* [10] reported that the increasing of ε value of $BaTiO_3$ with the increasing of La and Fe doping content composition. Jiang *et al* [11] reported that deposition Ag on $BaTiO_3$ nanoparticles improved the catalytic performance properties of $BaTiO_3$. Acosta *et al* [12] reported in their review that $BaTiO_3$ is a promising material for capacitor applications.

Many researchers have developed several methods to fabricate barium titanate; the most popular one is the sol-gel method. This method has the advantage that it can be carried out at room temperature and is low cost [12]. The ferroelectric properties of barium titanate material have attracted much attention from researchers which make researchers develop several modeling to calculate the polarization properties of barium titanate. Several models have been developed such as Landau — Devonshire (LD) theory Landau — Khalat-

nikov dynamic. These models use a material Gibbs free energy approach [13]. Another model is the Polydomain model developed by Wang *et al* [13] uses the free energy and polarization that occurs in the domains. From several models that have been developed, no model uses the polarization approach that occurs in the unit cells.

In this research, the electric polarization of $BaTiO_3$ obtained from the experiment will be compared to the calculated electrical polarization obtained from two model Landau — Khalatnikov theory and Polydomain theory. The calculation of electric polarization needs experimental crystal parameters and structure factor constant (A). $BaTiO_3$ was synthesized using the sol-gel method [1]. By analyzing the X-ray diffractometer pattern of $BaTiO_3$, the crystal parameters and the structure factor constant were obtained. These crystal parameters and structure factor constant were used as input in calculating electrical polarization in the model Landau — Khalatnikov theory and Polydomain theory. The experimental polarization was obtained by means of the Sawyer — Tower circuit. From the polarization experiment, it obtain saturated polarization (P_s), remanent polarization (P_r), and coercive electric fields (E_c).

By comparing the two models with the experimental polarization, it will be seen which model is more suitable for $BaTiO_3$ [15].

Experimental method and simulation. *Experimental method.* As noted, the barium titanate $BaTiO_3$ material was synthesized using the sol-gel method. The sol-gel synthesis was chosen to synthesize the $BaTiO_3$ due to capable obtain high-quality materials with high homogeneity and high purity. It also lows cost and low temperature for synthesizing. The organic compound is usually used in the sol-gel process. The element constituent, barium, and titanium will be bonded in an organic compound by replacing some group of ions that constitute the organic compound. So, the distance between the ion Ba and Ti and oxygen are so close then it is easy to react with each other to form $BaTiO_3$ during heating at a certain temperature.

The barium titanate $BaTiO_3$ material was made using barium acetate [$Ba(C_2H_3O)_2$], acetic acid [$C_2H_4O_2$], titanium isopropoxide [$Ti(OCH(CH_3)_2)_4$], ethylene glycol [$HOCH_2CH_2OH$]. In the initial stage, barium acetate [$Ba(C_2H_3O)_2$] was dissolved using acetic acid [$C_2H_4O_2$] at a temperature of 30 °C on a hotplate and stirred using a magnetic stirrer. Then titanium isopropoxide [$Ti(OCH(CH_3)_2)_4$] and ethylene glycol [$HOCH_2CH_2OH$] 99.5 % were added at the same temperature. The solution was heated using a hotplate

at a temperature of 300 °C until the solution become powder, then sintered at 900 °C for 3 hours to obtain barium titanate powder. In the next step, an X-ray diffraction (XRD) examination was carried out to determine the barium titanate material crystal structure and lattice parameters. The next step is to measure the polarization properties of the hysteresis curve of the barium titanate material using an electronic circuit with the Sawyer — Tower-based circuit. A function generator with a triangular input signal with an input voltage of 20 V and a frequency of 50 Hz was used as a voltage source, and a ceramic capacitor was used as a comparison capacitor [14]. The final step, semiempirical modeling, was carried out using the Polydomain model and Landau — Khalatnikov dynamic model with modification of input parameters calculated from barium titanate crystal structure and polarization factors.

Polydomain model simulation. Landau formulates free energy in a material in the form of Gibbs free energy, which is a combination of several energy components from the constituent domains of the material, such as free energy in the paraelectric phase (G_0), free energy in up polarization (G_{up}), free energy in down polarization (G_{down}), and free energy at the boundary (G_{wall}) between domains shown in equation [14, 21, 22]:

$$G = G_0 + G_{up} + G_{down} + G_{wall}.$$

Then, the condition of the domains that polarization occurs up and down the free energy can be described as in equation [14, 21, 22]:

$$G = G_0 - \frac{\alpha}{2} P_{up}^2 + \alpha P_{up}^2 + \left(1 - 2\alpha_{up}\right) EP,$$

where α is the polarization condition in the volume ratio of the material. The relationship between polarized dipoles up dan down is shown in equation [14, 21, 22]:

$$\alpha_{down} = 1 - \alpha_{up}.$$

Furthermore, the maximum polarization of the material can be calculated by the first derivative of the free energy concerning the polarization equal to zero, as shown in equation:

$$\frac{dG}{dP} = -\alpha P_{up} + \alpha P_{down}^3 + \left(1 - 2\alpha_{up}\right) E = 0.$$

So, the total polarization of the barium titanate ($BaTiO_3$) material from the up and down polarization as:

$$P = \alpha_{up} P_{up} + \alpha_{down} P_{down} = \left(1 - 2\alpha_{up}\right) P_{up}.$$

Then α_{up} can be determined by using equation [14]:

$$\alpha_{up} = \frac{\tan^{-1}\left[A\left(E - E_c\right) + A\left(E - E_c\right)^3 \right]}{\pi} + \frac{\pi}{2}. \quad (1)$$

Then A is assumed as the structure factor constant [15]; E_c is the coercive electric field when saturated polarization was achieved.

Landau — Khalatnikov model simulation. Dynamic Gibbs free energy of Landau —Khalatnikov can be used to explain ferroelectric phenomena. These phenomena can be calculated by using Landau — Devonshire theory as a function of the polynomial equation, as shown in equation [15]:

$$P\left(P, E\right) = F_0 + \frac{A\left(T - T_c\right)}{2} P^2 - \frac{A}{4} P^4 + \frac{1}{6} P^6 - EP,$$

where F_0 is Gibbs free energy of material at paraelectric phase; A is structure factor constant which will be calculated using crystal structure constant from experimental data; T is the temperature of the material; T_c is current temperature; E is the electric field applied to the sample; P is polarization. Hence, Eq. (1) can be rewritten as [15]:

$$F = \frac{A}{2} P^2 - \frac{A}{4} P^4 + \frac{1}{6} P^6 - EP.$$

Landau — Khalatnikov dynamic Gibbs free energy model was used to explain the polarization phenomena on barium titanate, which is a partial differential equation shown in equation [15]

$$\gamma \frac{dP}{dt} = -\frac{\partial F}{\partial P}. \quad (2)$$

Here γ is the switching constant; F is Gibbs free energy of the material. The relation between the free energy as a sine function and polarization can be rewritten using equation

$$\gamma \frac{dP}{dt} = -AP + AP^3 - P^5 + E\sin(\omega t), \quad (3)$$

therefore, Eq. (2) becomes Eq. (3). In Eq. (3) E is the electric field as a function of sine and frequency. Equation (3) was solved by using Runge — Kutta order 4. The Polydomain and Landau — Khalatnikov models were evaluated by calculating R-factor (R_{wp}). Calculation of R-factor (R_{wp}) using equation [15]:

$$R_{wp} = \left[\frac{\sum_{i=1}^{n} \left(P_{i\,exp} - P_{i\,mod} \right)^2}{\sum_{i=1}^{n} P_{i\,exp}^2} \right],$$

where P_{exp}, P_{mod} are the polarization of the experiment and the polarization of the model.

Calculation of structure factor constant. The crystal structure factor constant of $BaTiO_3$ material can be calculated using the total dipole moment approach from the dipole moment as follows:

$$p = \sum_{i=1}^{n} q_i r_i,$$

where the total value of the dipole moment is as followed:

$$\begin{gathered} p = \sum_{i=1}^{15} q_i r_i = 0.25q\left(0i + 0j + 0k\right) + 0.25q\left(i + 0j + 0k\right) + \\ + 0.25q\left(i + j + 0k\right) + 0.25q\left(0i + j + 0k\right) + 0.25q\left(0i + 0j + k\right) + \\ + 0.25q\left(i + 0j + k\right) 0.25q\left(i + j + k\right) + 4q\left(0.5i + 0.5j + 0.5k\right) - \\ - q\left(0.5i + 0.5j + 0.5k\right) - q\left(0.5i + j + 0.5k\right) - q\left(i + 0.5j + 0.5k\right) - \\ - q\left(0i + 0.5j + 0.5k\right) - 0q\left(0.5i + 0.5j + 0k\right) - q\left(0.5i + 0.5j + k\right) = \\ = 0.25qi + 0.25qj + 0.25qk. \end{gathered}$$

Then the length of the polarization vector can be calculated as

$$|p| = \sqrt{\left(0.25aq\right)^2 + \left(0.25aq\right)^2 + \left(-qa\right)^2} = 0.707qa.$$

Then the calculation of the saturated polarization is as followed:

$$P_s = \frac{pxa}{V}.$$

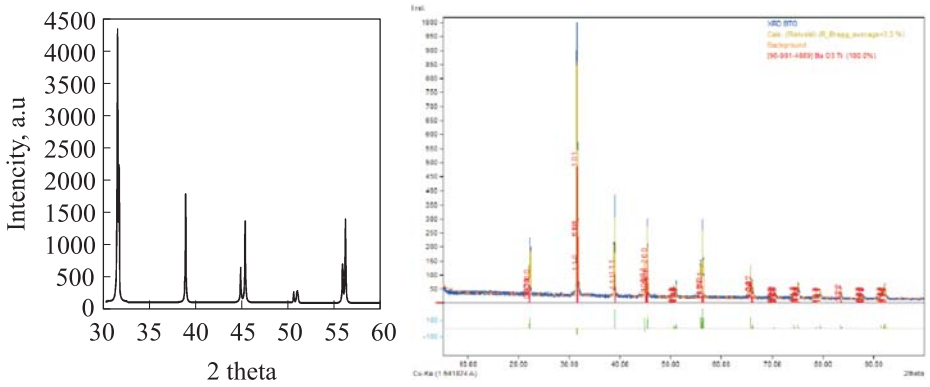
The value from the calculation of spontaneous polarization is then assumed to be the maximum polarization value (P_s) of the $BaTiO_3$ material. Furthermore, at the maximum polarization, the saturated polarization value is obtained using the equation as follows [14, 17]:

$$P_s^2 = \frac{3}{5}\left(1 + \left[1 - \frac{5A}{9}\right]^{1/2}\right). \tag{4}$$

Then, the value of the A constant is the structural factor constant to be calculated.

Results and discussion. XRD pattern of barium titanate ($BaTiO_3$) material made using the sol-gel method is shown in Fig. 1. The result indicates that the barium titanate ($BaTiO_3$) material has been successfully fabricated, as indicated by the appearance of the peaks of the XRD curve, which corresponds to the

database from ICDD, which is displayed using the *Match* program. Furthermore, to determine the crystal structure of $BaTiO_3$, an analysis was carried out by matching the XRD pattern with the ICDD database using the *Match* program, and it was found that several crystal planes had appeared at 110, 111, 200, and 222.



a *b*

Fig. 1. XRD pattern of barium titanate prepared by sol-gel method (*a*, *b*)

Next step, the XRD data was analyzed using Rietveld analysis on *Fullproof* software to determine the crystal structure and lattice parameters of the barium titanate ($BaTiO_3$) material shown in Fig. 2. The Rietveld analysis results then are used to calculate the structural factor constants. The results of the refinement using *Match* software showed the value of χ^2 (chi-squared) is 2.1, which indicates the refinement results are good and are shown in Table 1.

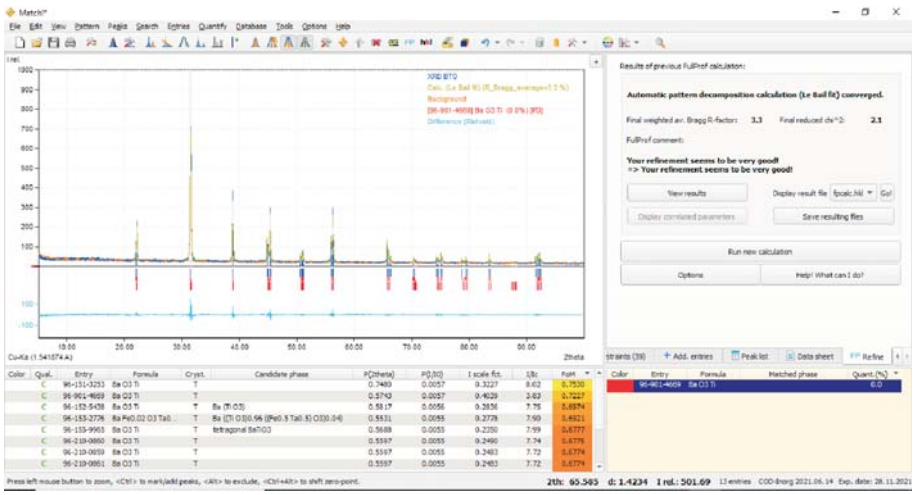


Fig. 2. Refinement of barium titanate X-ray diffraction pattern prepared using sol-gel method

Table 1

Refinement using Rietveld method

Parameter	Before refinement	After refinement
Crystal Structure	Tetragonal	
Lattice parameter, Å:		
a	3,9988	3,9949
c	4,0222	4,0364
Atoms position:		
Ba	0 0 0	0 0 0
Ti	0,5 0,5 0,502	0,5 0,5 0,5
O	0,5 0,5 –0,015	0,5 0,5 –0,015
O	0,5 0 0,513	0,5 0 0,513
χ^2	–	2,1

The refinement result was then visualized using the *Endeavor* software, as shown in Fig. 3. From the result, it was found that the crystal structure of the barium titanate material made using the sol-gel method is tetragonal perovskite, and polarization will occur on the perovskite structure of barium titanate ($BaTiO_3$) [4, 5, 18–20].

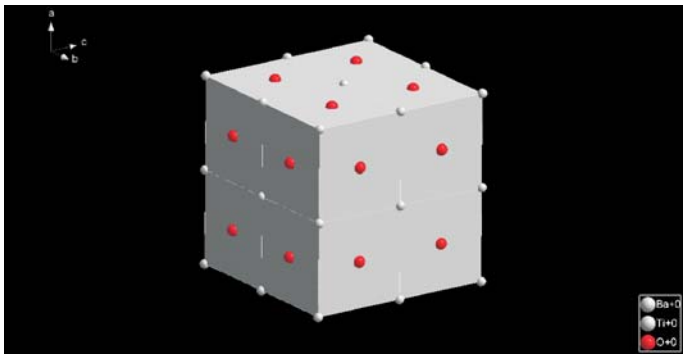
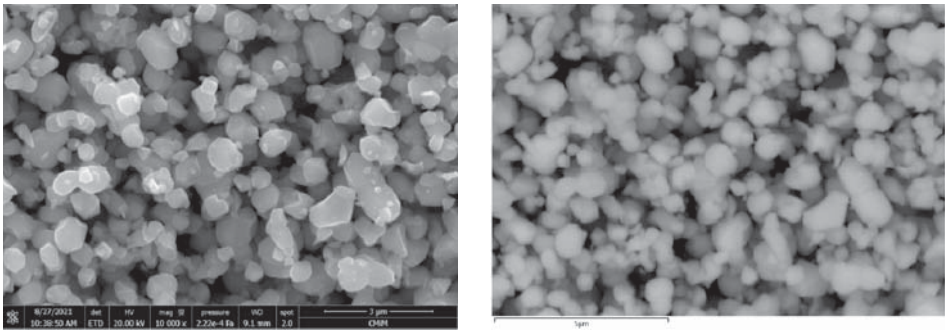


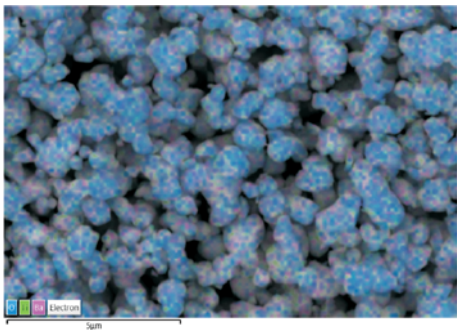
Fig. 3. Crystal structure of barium titanate prepared by sol-gel method

The analysis using EDAX and SEM is shown in Fig. 4. From the SEM results, it can be seen that the crystal material of barium titanate ($BaTiO_3$) has been formed with dimensions of ≤ 3 μm, and it is also seen that the material formed homogeneously. In Fig. 4, *c* it is also seen that the sample has detected three main elements that formed $BaTiO_3$ material, Ba, Ti, and O atoms.

The elements were examined using EDAX of barium titanate ($BaTiO_3$) fabricated using the sol-gel method shown in Fig. 5. The results show that the $BaTiO_3$ material has been formed perfectly, composed of Ba, Ti, and O elements



a *b*



c

Fig. 4. SEM images of barium titanate prepared by sol-gel method

without any impurity elements. The weight composition of barium titanate is shown in Table 2. The composition of each element formed was Ba 52.9 wt %, Ti 22.46 wt %, and O 18.34 wt %.

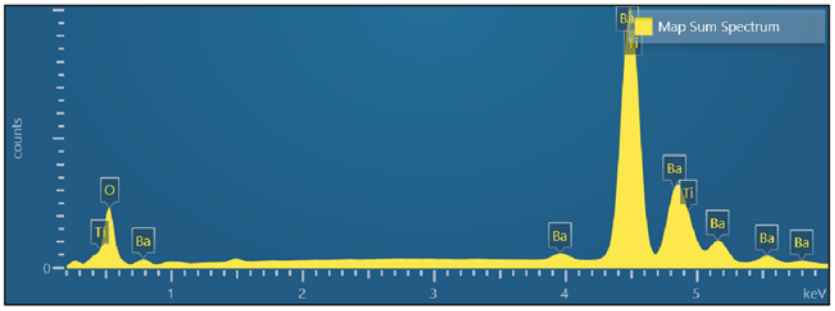


Fig. 5. EDAX result of barium titanate prepared by sol-gel method

Table 2

Composition of barium titanate ($BaTiO_3$) prepared by sol-gel method

Element	Line type	Map Sum Spectrum		
		Weight %	Weight % Sigma	Atomic %
O	K series	18.34	0.26	56.01
Ti		22.46	0.20	22.92
Ba	L series	59.20	0.27	21.07
Total	–	100	–	100

The calculation results of the structure constant A of the barium titanate material, which is calculated using Eq. (4) and then used as input for the hysteresis curve modeling of the barium titanate material using the Landau — Khalatnikov and Polydomain models, shows bellow:

a, Å	c, Å	V, Å^3	P_s,10^1, C/m^2	A
3.9949	4.0364	64.4178	1.5	2.65

The results of the measurement of the ferroelectric properties of the barium titanate ($BaTiO_3$) material using the sol-gel are shown in Fig. 6. From the results it can be seen that the remanent polarization (P_r) is $1.5 \cdot 10^{-1}$ C/m^2, saturated polarization (P_s), coercive electric field (E_c), which are $2.56 \cdot 10^{-1}$ C/m^2 and $4.52 \cdot 10^{1}$ V/mm. Meanwhile, the modeling results using the Landau — Khalatnikov model, the remanent polarization (P_r) is $1.98 \cdot 10^{-1}$ C/m^2, and the value of saturated polarization (P_s), coercive electric field (E_c) are $2.6 \cdot 10^{-1}$ C/m^2 and $7.05 \cdot 10^{1}$ V/mm and also from the results of Polydomain model, showed that remanent polarization $P_r = 3.45 \cdot 10^{-1}$ C/m^2 and the value of saturated polarization $P_s = 3.4 \cdot 10^{-1}$ C/m^2 and coercive electric field $E_c = 6.1 \cdot 10^{-1}$ V/mm. From the results, there are differences in the values of remanent polarization, saturated polarization, and coercive electric field between the experimental and the modeling results. Then, a calculation of the R-factor (R_{wp}) was carried out to determine the agreement between the results

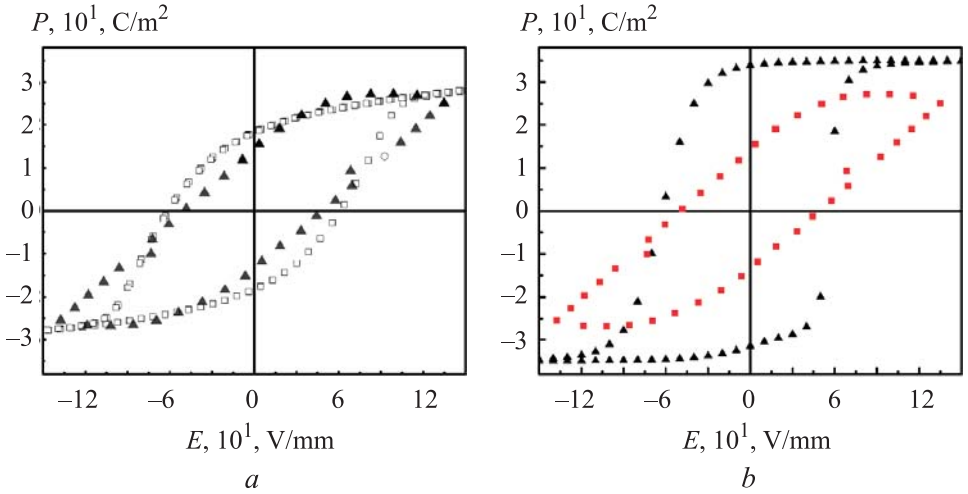


Fig. 6. Hysteresis curve of barium titanate experiment (□) and modified Landau — Khalatnikov modelling (▲) (*a*), experiment (▲) and modified Polydomain modelling (■) (*b*)

of the experiment and modeling using the structural factor as an input; the results obtained were 8.7 % for modeling using modified Landau — Khalatnikov model and 11.3 % for modeling using modified Polydomain model as shown in Table 3. From these results, it was found that the *R*-factor (R_{wp}) value from modeling using the Landau — Khalatnikov model was ≤ 10 % which statistically showed promising results.

Table 3

Remanent polarization, saturated polarization, coercive electric field, and *R*-factor calculation between experiment and modelling results

Result	P_r,10^1, C/m^2	P_s,10^1, C/m^2	E_c,10^1, V/mm	*R*-factor (R_{wp}), %
Experiment	1.50	2.56	4.51	–
Landau — Khalatnikov model	1.98	2.60	7.05	8.7
Polydomain model	3.45	3.40	6.10	11.3

From the results, it can be seen that the Landau — Khalatnikov model with a structural factor approach significantly affects the Gibbs free energy and polarization that occurs in the barium titanate ($BaTiO_3$) crystals [13–15]. While the modeling using the structural factor approach, carried out using the Polydomain model barium titanate ($BaTiO_3$) material, was very dependent on the value of the electric field applied to the material [13]. For further research, we will calculate several additional parameters and factors such as pressure, temperature variations, and doping variations, and also consider some models such as the dipole switching model.

Summary. Barium titanate ($BaTiO_3$) material fabricated using the sol-gel method followed by a sintering process has been formed as shown from the XRD, which has appeared at the peaks of the 110, 111, 200, 222 and Lattice parameters a = 3.9949 Å and c = 4.0364 Å. The crystal structure of the barium titanate material is tetragonal perovskite. It was found that there were no impurities, and the crystal of barium titanate was formed homogeneously with dimensions of 3 μm. The ferroelectric properties of barium titanate ($BaTiO_3$) material are remanent polarization (P_r) with a value of $1.5 \cdot 10^{-1}$ C/m^2, saturated polarization (P_s) is $2{,}56 \cdot 10^{-1}$ C/m^2 and coercive electric field (E_c) is $4.52 \cdot 10^1$ V/mm. The *R*-factor (R_{wp}) between the experimental results and the modeling of the ferroelectric properties of the barium titanate ($BaTiO_3$) material

using the modified Landau — Khalatnikov model and the Polydomain model are 8.7 and 11.3 %. From these results, it can be concluded that the Landau — Khalatnikov model is better for modeling the ferroelectric properties of barium titanate ($BaTiO_3$) material with a structural factor approach.

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