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## Effect of Synthesis Method on the Structural Behavior of CaFeO<sub>2.5</sub> Compound

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CaFeO<sub>2.5</sub> samples were synthesized by solid solution, mirror furnace and Sol-gel methods. The effect of the synthesis method on the behavior structure was investigated. Phase structures are comparatively characterized and studied by means of X-ray powder diffraction. Experimental results have revealed that the synthesis method has a strong influence on the structure of the studied compounds. All samples obtained by the three methods are crystallized in the *Pnma* orthorhombic system. We obtained the best results in the case of the Sol-gel technique. In the Sol-gel method, the lattice parameters obtained are  $a = 5.41631 \text{ \AA}$ ,  $b = 14.73899 \text{ \AA}$  and  $c = 5.58790 \text{ \AA}$ . Also, the value of the average crystallite size  $D = 52.03 \text{ nm}$  and the dislocation density  $\delta = 3.69 \times 10^{10} / \text{cm}^2$ . Since the values of the lattice parameters in this method were the smallest of the three methods, which exhibiting a weak shrink of the volume compared to the solid solution and mirror furnace one. This shrinking is a natural result of the decrease in the value of the average crystallite size and the increase of dislocation density with a reason for the inverse relationship between them. This allows us to conclude the importance of the Sol-gel method for obtaining CaFeO<sub>2.5</sub> nanoscale compound.

**Keywords:** Crystallite size; Dislocation density; Nanoparticle; Sol-gel; X-ray powder diffraction.

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### Introduction

Recently, ABO<sub>3- $\delta$</sub>  perovskite oxides have been receiving much attention. These non-stoichiometric oxides have become a very important fields of research with different technological applications, such as ceramic membranes for oxygen separation and electrodes of solid oxide fuel cells (SOFCs), electrocatalysis, gas sensor and wastewater treatment [1-7]. Among these ABO<sub>3- $\delta$</sub>  perovskite oxides, the CaFeO<sub>2.5</sub> compound ( $\delta = 0.5$ , have a brownmillerite structure) considered as a candidate material for energy and environmental applications [8-11]. CaFeO<sub>2.5</sub> compound exhibit two crystal structures as a function of the temperature, which crystallizes in the orthorhombic structure with space group *Pnma* (S.G. N° 62) up to 500 °C, then change its structure to the orthorhombic space group *I2mb* (S.G. N° 46) [12-16].

Today, we know that the X-ray diffraction (XRD) is a powerful non-destructive technique for characterizing crystalline materials. It provides information on structures, phases, average crystallite size and other structural parameters, such as crystallinity, strain, and crystal defects [17]. Consequently, the average crystallite size has a great influence on the microstructure, and therefore on the physical properties of the resulting ceramics [18-21].

The aim of the present study is to compare the lattice parameters, crystallite size and the dislocation density determined by X-ray powder diffraction analysis in all brownmillerite CaFeO<sub>2.5</sub> powders obtained by three synthesis methods. The first sample was synthesized by conventional solid solution, the second one was made by the mirror furnace based on the melting zone technique [22-23], while the third was the sol-gel method. We focus our study to the analysis and determination of structural

properties, the crystallite size and the density dislocation, which occur during the samples formation. Especially, we will estimate the effect of preparation process in different synthesized method on structural behavior of these samples.

## I. Materials and Methods

### 1.1. Synthesis methods

The  $\text{CaFeO}_{2.5}$  samples have been prepared by the conventional solid solution, mirror furnace using melting zone technique and sol-gel synthesis methods.

#### 1.1.1. Solid solution method

$\text{CaFeO}_{2.5}$  sample was prepared in air by solid-state reaction. Stoichiometric amounts of commercial  $\text{CaCO}_3$ ,  $\text{SrCO}_3$  and  $\text{Fe}_2\text{O}_3$  oxides were well mixed with acetone in agate mortar for few minutes. The mixture was annealed at  $1000^\circ\text{C}$  for 12 hours, and then the powder was compacted in pellets, each of 1 g and 13 mm in diameter. The pellets were heated in air in conventional furnace at  $1200^\circ\text{C}$  for 24 hours. Then, the samples were quenched in liquid nitrogen. This operation is repeated two times. The pellets were ground to fine powder for phase characterization. A portion of the obtained powder was used as a starting point of the second method so-called "mirror furnace method".

#### 1.1.2. Mirror furnace method

The amount of powder of  $\text{CaFeO}_{2.5}$  sample as-prepared by solid solution method was put in a latex tube in order to prepare the feed rod. A hydraulic pressure of 10 bars was applied to obtain a solid bar, and it was calcined in air for 12 h at  $950^\circ\text{C}$ . Afterwards, a high temperature around  $1600^\circ\text{C}$  is applied using the mirror furnace concentrated on a relatively small spot size at the bottom of the rod until melting. The bottom falls down as a molten drop directly in liquid nitrogen. Each part requires between 4 to 5 minutes in order to melt and form a molten drop and then fall.

#### 1.1.3. Sol-gel method

$\text{CaFeO}_{2.5}$  powder was prepared using a Sol-gel synthesis method. Stoichiometric amounts of  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  and  $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  were mixed in deionized water and heated at  $70^\circ\text{C}$ . Citric acid was then added as the metal ions complexing agents. The molar ratio of total metal ions and citric acid in the solution was 1:2. To ensure complete complexation, solution pH was adjusted to 6 by adding  $\text{NH}_3$  aqueous solution. The final powders were obtained from the solution after water evaporation at  $120^\circ\text{C}$  to form a brown gel followed by calcination of this gel at  $600^\circ\text{C}$  for 12 hours in air. The powder was heated in air in conventional furnace at  $800^\circ\text{C}$  for 24 hours. This operation is repeated two times in order to obtain the phase.

## 1.2. Characterization

### 1.2.1. X-ray powder diffraction measurements

Phase identification and unit cell parameter determination are checked using powder X-ray diffraction (XRD) at room temperature. Powder X-ray diffraction data were recorded using a Bruker D8 Advance diffractometer (Bragg-Brentano configuration,  $\text{CuK}\alpha_1$ ) in the  $2\theta$  range of  $10 - 80^\circ$ .

### 1.2.2. EDX spectroscopy

The energy dispersive X-ray spectroscopy (EDX) analysis was carried out for  $\text{CaFeO}_{2.5}$  sample using a phenom (JSM-6400) scanning electron microscope equipped with an EDX micro-analytical system, intended for the observation of dry and conducting samples.

### 1.2.3. Crystallite size and dislocation density

The dislocation density ( $\delta$ ), which represents the amount of defects in the sample is defined as the length of dislocation lines per unit volume of the crystal, and is evaluated from Williamson and Smallman's formula [24, 25]:

$$\delta = \frac{1}{D^2}, \quad (1)$$

where  $D$  is the average crystallite size.

The crystallite sizes were determined from the full-width at half-maximum (FWHM) in radians using the Scherrer's formula. Its mathematical equation is given as follow [26]:

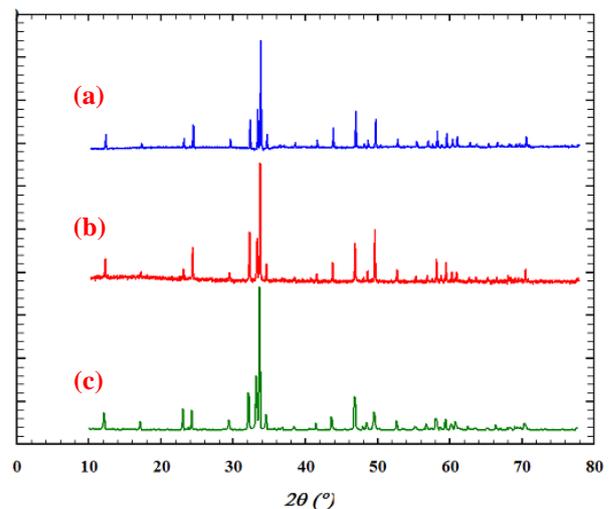
$$D = \frac{k \cdot \lambda}{\beta_{hkl} \cdot \cos\theta}, \quad (2)$$

where  $k$  is the shape function for which a value of 0.9 is used,  $\lambda$  is the wavelength of the incident X-Ray ( $\text{Cu-K}\alpha_1 = 1.54056 \text{ \AA}$ ),  $\theta$  is the diffraction angle and  $\beta_{hkl}$  is full-width at half-maximum (FWHM) of the XRD peak.  $\beta_{hkl}$  is appreciated as the estimated correct broadening of the sample.

## II. Results and Discussion

The results of X-ray powder diffraction at room temperature of  $\text{CaFeO}_{2.5}$  samples prepared by Solid Solution (SS), Mirror Furnace (MF) and Sol-Gel (SG) methods are presented in Fig. 1.

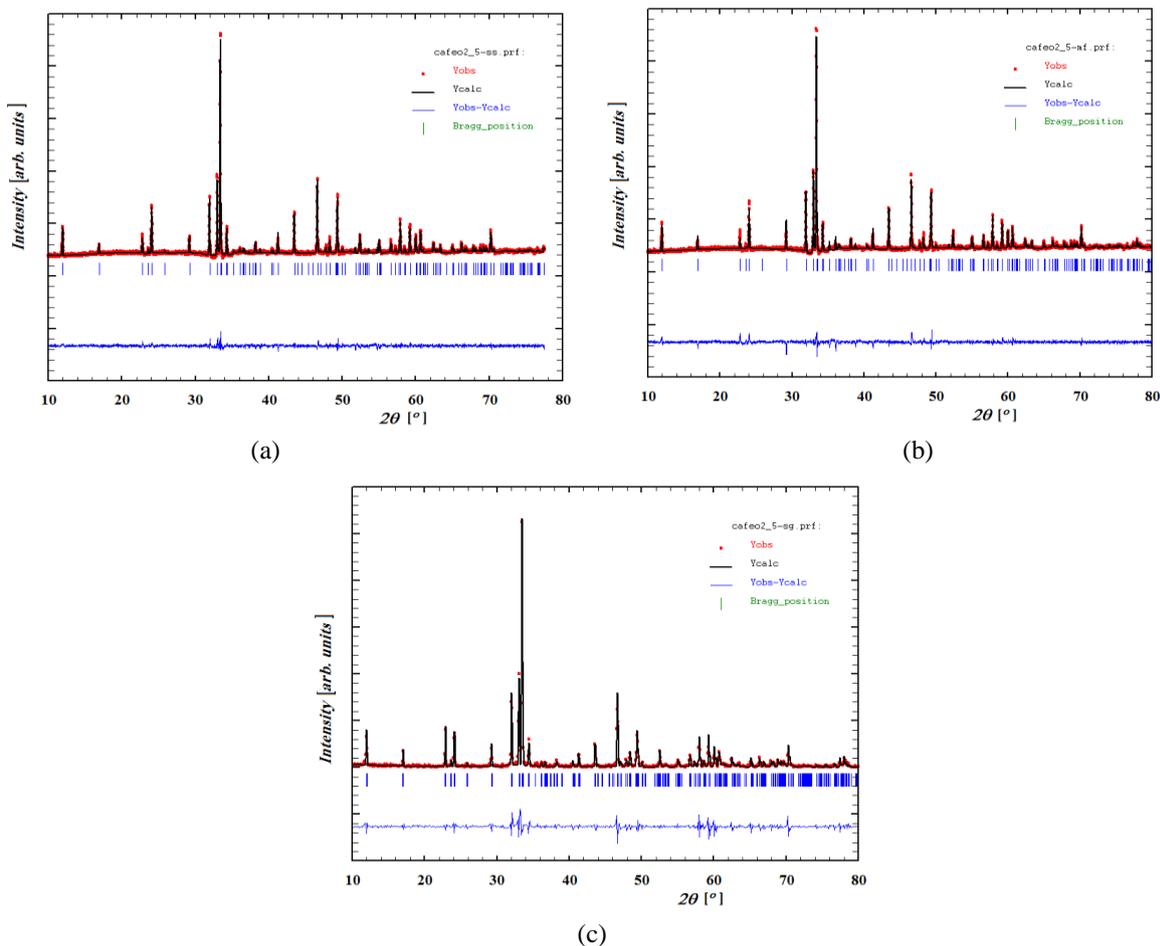
As can be seen, no differences are observed in the pattern for the samples prepared by the three routes. The crystal structures of the three  $\text{CaFeO}_{2.5}$  samples were identified as an orthorhombic brownmillerite (space group  $\text{Pnma}$ , No. 62), in agreement with the literature data [14].



**Fig. 1.** X-Ray powder diffraction pattern of  $\text{CaFeO}_{2.5}$  samples obtained by (a) SS, (b) MF and (c) SG methods.

**Table 1**Summary of lattice parameters of CaFeO<sub>2.5</sub> samples synthesized by SS, MF and SG methods.

Synthesized method	Space group	Lattice parameters (Å)			Volume (Å <sup>3</sup> )
		<i>a</i>	<i>b</i>	<i>C</i>	
SS	<i>P nma</i>	5.42193 (5)	14.75619 (12)	5.59481 (5)	447.624 (3)
MF	<i>P nma</i>	5.42434 (11)	14.76254 (24)	5.59675 (10)	448.171 (26)
SG	<i>P nma</i>	5.41631 (7)	14.73899 (15)	5.58790 (8)	446.087 (8)
[14]	<i>P nma</i>	5.42580 (10)	14.76580 (30)	5.59740 (10)	448.443 (30)

**Fig. 2.** Le Bail refinement of experimental XRD pattern of CaFeO<sub>2.5</sub> (a) SS, (b) MF and (c) SG samples respectively, refined at room temperature in *Pnma* space group.

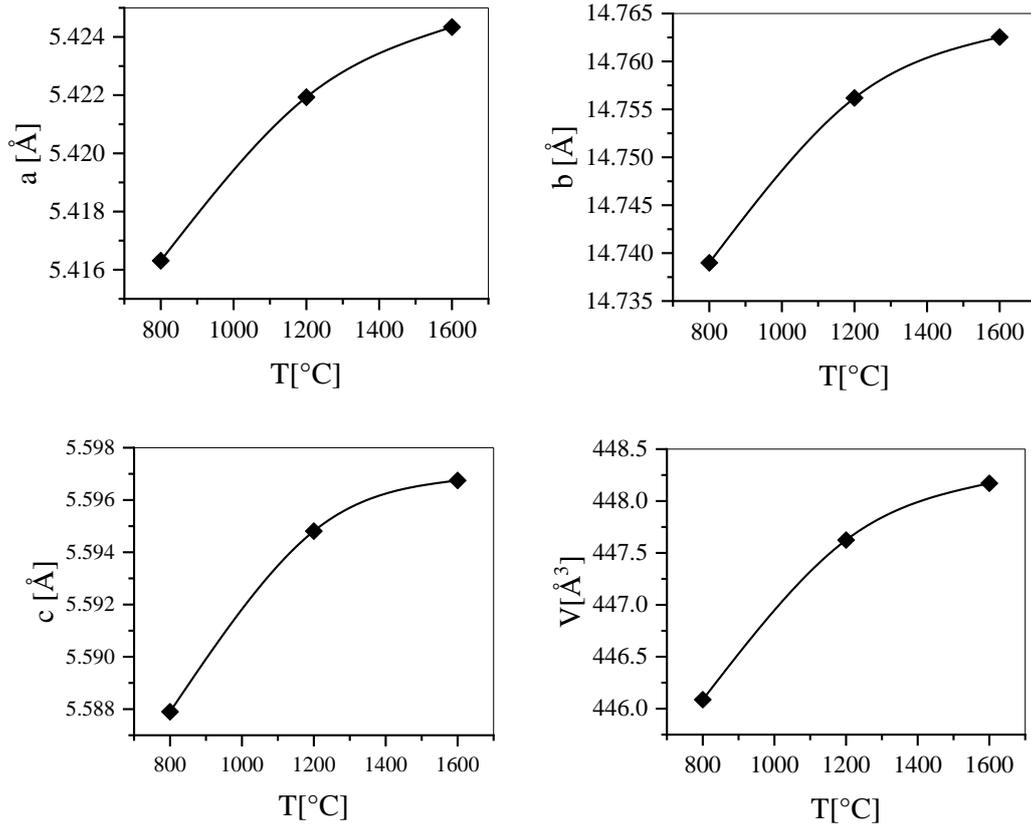
Therefore, the lattice parameters of all samples were refined by means of the Fullprof software [27] using Le Bail method. All peaks of different CaFeO<sub>2.5</sub> samples can be refined on the brownmillerite structure *Pnma*. The refinement results are summarized in Table 1, and the final Le Bail plot is presented in Fig. 2a, 2b and 2c.

Comparison of the values obtained from the indexing of X-ray powder diffraction patterns, showed that the results match in case of samples obtained by three methods gave slightly different results. Lattice parameters and the unit cell volume of CaFeO<sub>2.5</sub> obtained by the three methods (Table 1) are smaller than from those listed by Shaula et al. [14], and are monotonically decreasing with the decrease of synthesis temperature and absence of quenching in liquid N<sub>2</sub> step (Fig. 3). Also, we have noticed that the results of SS and MF methods are very close because the CaFeO<sub>2.5</sub> sample obtained by MF method is prepared firstly by solid solution method. While, in the XRD pattern collected from the all CaFeO<sub>2.5</sub> samples, the

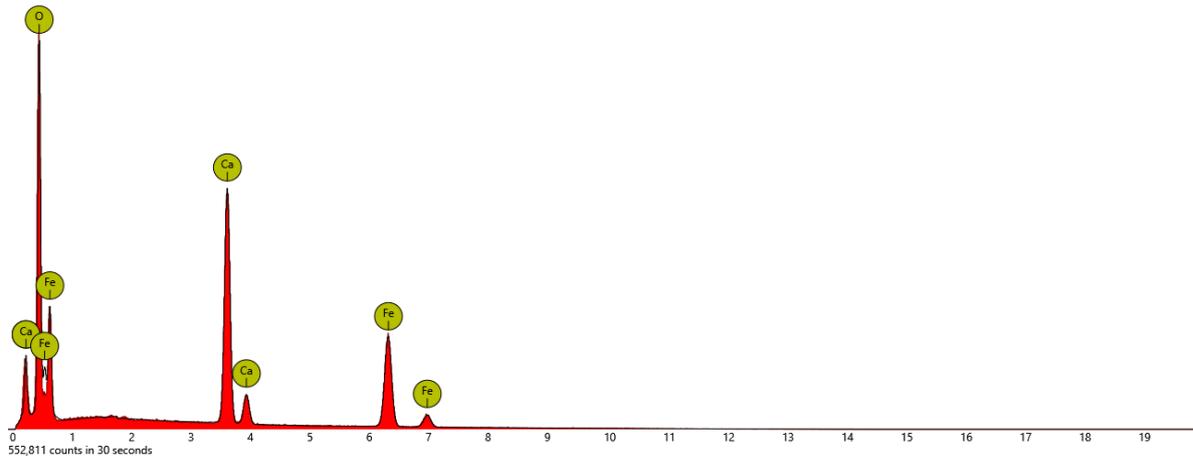
(111), (131) and (151) reflections and all representative *P* lattice ( $h+k+l=2n+1$ ) are present, indicating a primitive lattice.

A quantitative analysis of the precursor CaFeO<sub>2.5</sub> by the energy dispersive X-ray spectroscopy (EDX) analysis was used to check the chemical composition. Small amount of the sample's size is observed on a holey carbon film supported by a copper grid. EDX analysis indicates a Ca : Fe ratio of 1:1 in CaFeO<sub>2.5</sub>, which is consistent with the expected stoichiometry, within the limit of experimental error (Table 2). The EDX spectrum confirms the desired compounds (Fig. 4).

On the other hand, the increase of peak intensities in the XRPD pattern of the second method which is due to the enhancement of the crystallinity during the synthesis process. In the general case, the growth of the grains



**Fig. 3.** Variation of lattice parameters and volume lattice extracted from Le Bail refinement of X-Ray diffraction pattern as a function of the synthesis temperature in  $\text{CaFeO}_{2.5}$  samples.



**Fig.4.** Representative EDX spectrum of the sample  $\text{CaFeO}_{2.5}$ .

**Table 2**  
Compositions of  $\text{CaFeO}_{2.5}$  extracted by theoretical calculation and EDX analysis.

Element	Ca	Fe	O
Theoretical composition (% wt.)	25.10	34.97	39.93
Experimental sample composition (% wt.)	24.01	34.63	41.36

supported by the thermal energy contribution is associated with the increasing of the temperature, which allows the interpretation of the increasing of crystallite sizes.

We know that the breadth of the Bragg peak is a combination of both instrument and sample dependent effects [28]. So, we are used  $\text{LaB}_6$  standard for instrumental broadening correction. The corrected broadening corresponding to the diffraction peak of  $\text{CaFeO}_{2.5}$  was estimated by using the relation [29, 30]:

$$\beta_{hkl} = \left( \beta_{measured}^2 - \beta_{instrument}^2 \right)^{\frac{1}{2}}, \quad (3)$$

The crystallite size of characteristic peaks, the average

**Table 3**

Average crystallites sizes D in some XRD peaks and dislocation density  $\delta$  for CaFeO<sub>2.5</sub> compound prepared by SS, MF and SG methods.

	(hkl) peak	2 theta (degree)	FWHM= $\beta$ (degree)	crystallites size (nm)	D average (nm)	Dislocation density $\delta$ ( $\times 10^{10} / \text{cm}^2$ )
SS	(020)	12.0532	0.1114	71.70	98.18	1.04
	(101)	22.8920	0.0862	94.03		
	(040)	24.1605	0.0969	83.84		
	(200)	32.0429	0.0825	100.19		
	(002)	33.0904	0.0773	107.16		
	(141)	33.5071	0.0749	110.80		
	(161)	43.5614	0.0735	116.42		
	(202)	46.6920	0.0816	106.00		
	(080)	49.4425	0.0936	93.46		
MF	(020)	12.0001	0.1417	56.36	78.01	1.64
	(101)	22.8362	0.1131	71.66		
	(040)	24.0997	0.1212	67.01		
	(200)	31.9828	0.1018	81.16		
	(002)	33.0231	0.1023	81.00		
	(141)	33.4455	0.1029	80.63		
	(161)	43.4941	0.0942	90.82		
	(202)	46.6236	0.1073	80.64		
	(080)	49.3685	0.0942	92.84		
SG	(020)	12.0010	0.1689	47.29	52.03	3.69
	(101)	22.8853	0.1181	68.65		
	(040)	24.1467	0.1667	48.74		
	(200)	32.0170	0.1256	65.82		
	(002)	33.0694	0.1515	54.71		
	(141)	33.5133	0.1409	58.88		
	(161)	43.5620	0.1853	46.16		
	(202)	46.7104	0.1980	43.70		
	(080)	49.4270	0.2547	34.34		

crystallite sizes and the dislocation density for the three samples obtained from X-ray analysis are presented in Table 3.

We can see that the average crystallite sizes of CaFeO<sub>2.5</sub> samples extracted from some reflections are varied from 71.70 nm to 116.42 nm for the sample obtained by *SS* method, from 56.36 to 92.84 nm for *MF* method and varied from 34.34 nm to 68.65 nm for the one obtained by *SG* method. Also, the average crystallite sizes calculated by the Scherrer's formula change from 98.18 nm for *SS* method, 78.01 nm for *MF* method to 52.03 nm for *SG* method. It is worth mentioning that CaFeO<sub>2.5</sub> sample prepared with the *SG* method can have a greater value of full-width at half-maximum (FWHM) than the samples prepared with both *SS* and *MF* methods indicated the decrease of the particle sizes.

We can explain this situation to the decrease of the dislocation density. Dislocations are considered as a category from the most important crystal defects. This type of defect depends upon the morphological characteristics of the material and its crystallite size and preparation process [31]. The dislocation densities were found to be between  $1.04 \times 10^{10}$  and  $3.69 \times 10^{10}/\text{cm}^2$ . It is evident that the decrease in crystallite size causes an increase in the dislocation density.

Through the above-mentioned, we can say that the Sol-gel technique is one of the well-established synthetic

approaches that have potential control over the textural and surface properties of the materials [32]. This confirms that Sol-gel is a good method of preparation at a relatively low temperature compared to other methods.

## Conclusions

In summary, the influences of synthesis method on the structural behaviors of CaFeO<sub>2.5</sub> compounds were investigated. The three synthesis methods studied lead to slight differences in the cell parameters of compounds, which is a result of the different temperature during preparation. As the temperature increases from 1200 °C in solid solution and to 1600 °C in mirror furnace methods, both CaFeO<sub>2.5</sub> samples yield the same lattice parameters in the range of the error standard deviation (e.s.d.) with average crystallite sizes of 98.18 and 78.01 nm respectively. This increase of the average crystallite sizes is in agreement with that the growth of the grains supported by the thermal energy contribution is generally associated with the increasing of the temperature and the time of preparation. So, sol-gel process with synthesis temperature at 800 °C gave us smaller lattice parameters, also smaller average crystallite size of 52.03 nm, and greater dislocation density of  $3.69 \times 10^{10}/\text{cm}^2$ . So, decreasing the synthesis temperature applied in the 3<sup>rd</sup> sample leads to raising the dislocation density. We can

conclude that the preparation method for CaFeO<sub>2.5</sub> influences on the average crystallite sizes in different samples and thereby in its lattice volume. For these reasons, the Sol-gel process is an excellent method for obtaining CaFeO<sub>2.5</sub> nano-particle.

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## Вплив методів синтезу на поведінку структури сполуки CaFeO<sub>2.5</sub>

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Зразки CaFeO<sub>2.5</sub> синтезували твердорозчинним, дзеркальним та золь-гель методами. Досліджено вплив методів синтезу на поведінку структури. Методом порошкової рентгенівської дифракції порівняльно охарактеризовано та досліджено фазові структури. Результати експерименту показали, що метод синтезу має суттєвий вплив на структуру досліджуваних сполук. Усі зразки, отримані трьома методами, кристалізують в ромбічній системі Pnma. Найкращі результати отримано для випадку золь-гель методу. У золь-гель методі отримано параметри ґратки  $a = 5,41631 \text{ \AA}$ ,  $b = 14,73899 \text{ \AA}$  та  $c = 5,58790 \text{ \AA}$ . Також, отримано значення середнього розміру кристалітів  $D = 52,03 \text{ нм}$  і густини дислокацій  $\delta = 3,69 \cdot 10^{10} / \text{см}^2$ . Значення параметрів ґратки цьому методі були найменшими з трьох методів тавони демонструють слабку усадку об'єму порівняно із твердорозчинним і дзеркальним синтезом. Це усадка є закономірним результатом зменшення значення середнього розміру кристалітів і збільшення густини дислокацій, щоспричинене зворотною залежністю між ними. Це дозволяє зробити висновок про важливість золь-гель методу для отримання нанорозмірної сполуки CaFeO<sub>2.5</sub>.

**Ключові слова:** Розміри кристалітів; Щільність дислокацій; наночастинка; золь-гель; Порошкова рентгенівська дифракція.