International Journal of
Advances in Scientific Research and Engineering (ijasre)

E-ISSN : 2454-8006

DOI: http://doi.org/10.31695/IJASRE.2018.32927

Volume 4, Issue 10
October - 2018

Preparation and Study of the Structural, Thermal and Spectral Properties of lead Titanate

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ABSTRACT

Compound with the chemical formula (PbTiO3) were prepared using solid-state reaction method. The compound were calcined at 1000 oC for 4 hr. The structural properties of X-ray diffraction were studied and the results showed that the samples are tetragonal perovskite structure, the particle size was homogeneous of the samples, and increase the apparent density on the actual density. The results of the atomic force microscope (AFM) analysis showed a grain size, roughness and root mean square. TGA-DSC analysis shows low weight loss. The results of the infrared spectra for sample showed that the constant force of the bond (k) increased as a result of the stretching vibration Ti-O bond.

Keywords: *Lead Titanate, X-ray Diffraction, Atomic Force Microscope, Thermal analysis, Infrared Spectroscopy.*

1. INTRODUCTION

Lead titanate is a ferroelectric material with a structure similar to $BaTiO_3$ with a high curie temperature (450 ^oC). When the temperature of the substance is reduced below the curie temperature, A phase transition occurs from the cubic to the tetragonal is performed as in Figure.1. It is also difficult to make lead titanate compounds in shapes and sizes because they are affected when cooled under the curie point, as a result of turning the phase from cubic to tetragonal in $PbTiO_3$, resulting in stress greater than 6%, and then pure $PbTiO_3$ cracking and breaking during manufacturing can reduce self-stress during cooling By neutralizing lead titanate with various chemical elements such as Ca, Sr, Ba, Sn [1]. The transformation from the tetragonal to the cubic perovskite depends heavily on the temperature. The critical temperature in which this change occurs is called the curie temperature (T_C). If the material is cool during this temperature. This creates a variation in the structure and allows the distribution of an anisotropic in the lead titanate. This occurs because of the titanium ion change, along with a slight shift of some oxygen ions. Thus, a permanent pulsed electrode is created with more than T_C [2-3].

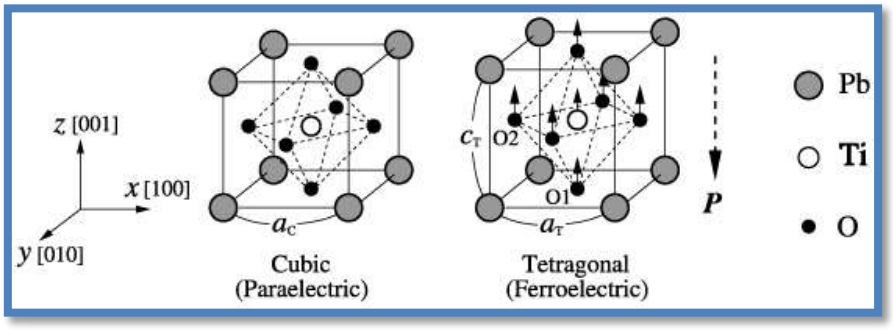

Figure 1: Tetragonal structure and cubic for composite $PbTiO_3$.

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2. EXPERIMENTAL DETAILS

2.1. Samples preparation

The ceramic compound were prepared ($PbTiO_3$) Using PbO , TiO_2 through solid state reactions method. The weight ratios of the samples were calculated using a sensitive balance. The powder were mixed and crushed using a ceramic mortar for 3 hours in order to obtain good homogenous mixture and homogeneous granular size. After that, the powder mixture was heated at 700 ^oC for 4 hours using a thermal oven. After heat The samples were then milled for 1 hour and poly vinyl alcohol (PVA) was added to it. Finally, the powder of calcined materials were pressed into pellets in 10 mm diameter and (2-3) mm thickness. These pellets were sintered at 1000 ^oC for 4 hours.

2.2. Characterization techniques

2.2.1. X-ray diffraction

The x-ray diffraction patterns of the sample were recorded using X-ray device, The lattice parameters (a and c) and Miller indices (hkl) for tetragonal structure are related by [4]:

$$\frac{1}{d^2} = \frac{h^2+k^2}{a^2} + \frac{l^2}{c^2} \quad \ldots\ldots(1)$$

The average particle size (D_p) of the samples was calculated using Scherrer's equation [5]:

$$D_{nm} = \frac{k\lambda}{\beta_{1/2}\cos\theta} \quad \ldots\ldots(2)$$

Where k = 0.9 (assuming the particles are spherical in shape); λ = wavelength of X-ray wavelength; β = full width at half maximum (FWHM) of the diffraction peak; θ = angle of diffraction.
The X-ray density was calculated using the formula [6]:

$$\rho_a = \frac{m}{V} \ldots\ldots(3)$$

Where

m: The sample mass

V: The sample volume, and it can calculated through the following formula:

$$V = \pi r^2 h \quad \ldots\ldots(4)$$

r: The radius of the sample, h: The thickness of the sample.

The theoretical value of the intensity of samples ($\rho_{x\text{-}ray}$) has been calculated through the following relationship [7]:

$$\rho_{x\text{-}ray} = M / N_A a^2 c \quad \ldots\ldots.(5)$$

Where

M is molecular weight expressed in grams, a and c are lattice constants and N_A is Avogadro's number (6.023 x 10^{23} molecule / mole).

It also calculated the percentage of porosity (P) using the following relationship [8]:

$$P = \left(\frac{\rho_{x-ray} - \rho_a}{\rho_{x-ray}}\right) \times 100\% \ldots\ldots (6)$$

2.2.2. Atomic force microscope analysis

The surface topography of the studied models was studied using the AFM technique A two-dimensional and three-dimensional image was taken to identify accurate details of the surface topography and crystalline structures as well as the distribution and size of the granules and the rate of roughness through a computer program.

2.2.4. Thermal analysis

The change in weight due to temperature for the compound under study was measured using the TGA-DSC technique. This technique is based on recording the change in the mass of the sample in terms of temperature or The thermo gravimetric analysis

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(TGA) is based on the differential scanning calorimetry (DSC) technique to analysis compounds by measuring the amount of absorbed and emitted heat and in determining the temperature ratio of these compound [9].

2.2.4. Spectral analysis

The ability of the sample to absorb electromagnetic light within the wavelength (400-4000 cm^{-1}) was determined by FT-IR technique. The following equations were used to calculate the strength of the force and the average length of the bond [10]:
μ =effective mass of Titanium and oxygen, which can be calculated as given below
$\mu = (M_{Ti} \times M_O / M_{Ti} + M_O)$ …….. (7)
Where
M_{Ti}=Atomic mass of titanium (Kg), M_O=Atomic mass of Oxygen (Kg)
$k = 4\pi^2 c^2 \nu^2 \mu$ …… (8)
Where
c = speed of light (cm/sec), ν =Frequency resulting from absorption. The bond force constant (k) is related to average bond length (r) by following equation [11]:
$r^3 = 17/k$ …… (9)

3. RESULTS AND DISCUSSION

3.1. XRD analysis

The results showed that all models were multi crystallization compounds with a crystalline tetragonal structure with differential direction (110), The advantage peaks are observed at the planes shown in the Figures (2). The peaks were indexed by comparing the inter-planer distance with the ICDD (00-006-0452), The (d) values were observed and calculated for $PbTiO_3$ as given in Tables (1) It also showed that no unknown values appeared in the phase, indicating the purity of the compounds that were manufactured.

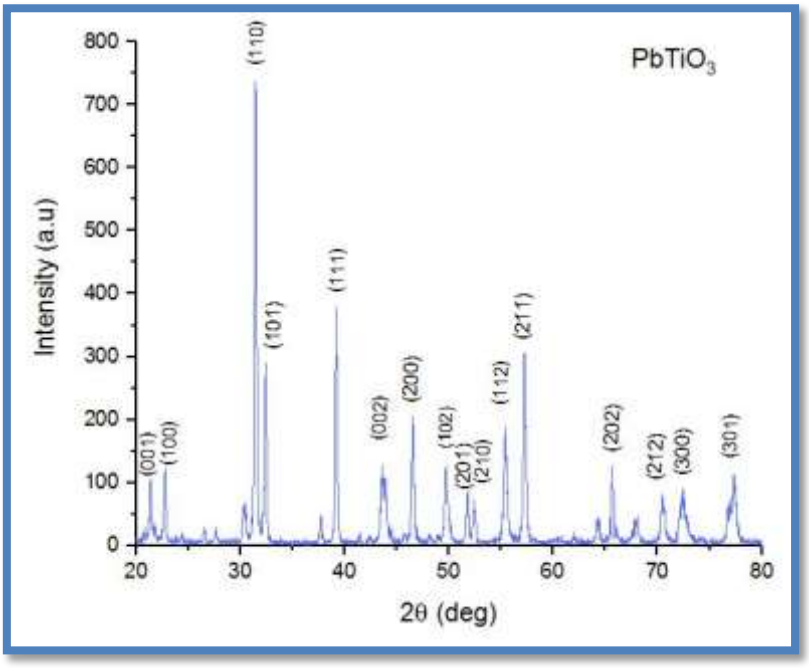

Figure 2: The X-ray pattern of the $PbTiO_3$.

Table. 1 turned out to be a good match with the card (ASTM) to examine the material. The calculations of the apparent density, physical density, particle size, and porosity, as shown in Table (2), It was found that the apparent density increases . The particle size was then calculated using the Scherrer's equation (2).

Table 1: Shows the miller indices (hkl) and inter-planer (d) for ($PbTiO_3$) sample.

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Peak number	Θ (degree)	d (Å)	d_{std}(Å)	(h k l)	I (%)
1	10.72	4.14	4.15	(0 0 1)	12
2	11.41	3.90	3.90	(1 0 0)	15
3	15.74	2.84	2.84	(1 1 0)	100
4	16.23	2.76	2.76	(1 0 1)	38
5	19.61	2.30	2.31	(1 1 1)	53
6	21.81	2.07	2.08	(0 0 2)	16
7	23.27	1.95	1.95	(2 0 0)	29
8	24.87	1.83	1.83	(1 0 2)	16
9	25.89	1.76	1.77	(2 0 1)	11
10	26.21	1.74	1.74	(2 1 0)	9
11	27.69	1.66	1.65	(1 1 2)	24
12	28.63	1.61	1.61	(2 1 1)	50
13	32.83	1.42	1.42	(2 0 2)	18
14	35.24	1.33	1.34	(2 1 2)	11
15	36.23	1.30	1.30	(3 0 0)	10
16	38.67	1.23	1.23	(3 1 0)	15

Table 2: The results of the structure properties to sample $PbTiO_3$.

Sample	a (Å)	c (Å)	c / a	Particle size D (nm)	$\rho_{x\text{-}ray}$ (g/cm^3)	ρ_a (g/cm^3)	Porosity %
$PbTiO_3$	4.015	4.140	1.031	34.65	7.97	6.780	10.08

3.2.The surface topography by AFM

AFM images analysis for ($PbTiO_3$) were performed in Figure. 2, Which show topographic analytical images in two dimensions (2D) and three dimensions (3D), respectively. The bonding between $PbTiO_3$ components is homogeneous and granular bonding with each other, indicating that there are no surface defects, which helps them to reduce the pores in the microstructure, which makes the $PbTiO_3$ complex with characteristic electrical properties. The grain size of the AFM found in Table 3. is larger than the particle size measured by the XRD, because XRD measures the particle size within the sample while AFM measures particle size on the sample surface, The crystalline on the sample surface will be larger.

Table 3: Shows the results obtained from the topography images analysis for the Lead Titanate.

Compound	Roughness average (nm)	Root mean square (nm)	Grain size(µm)
$PbTiO_3$	627.47	881.51	0.71

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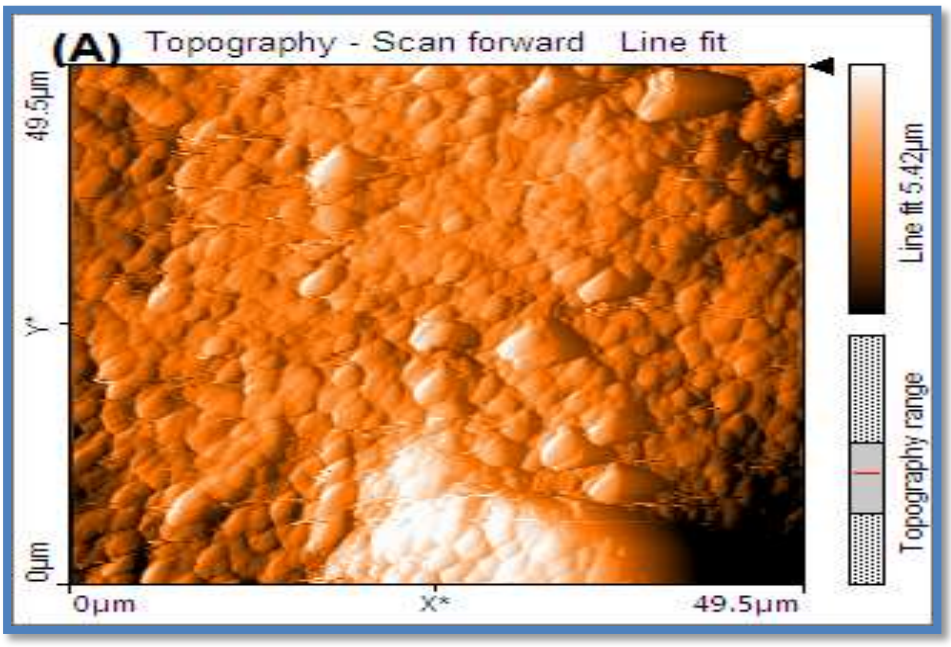
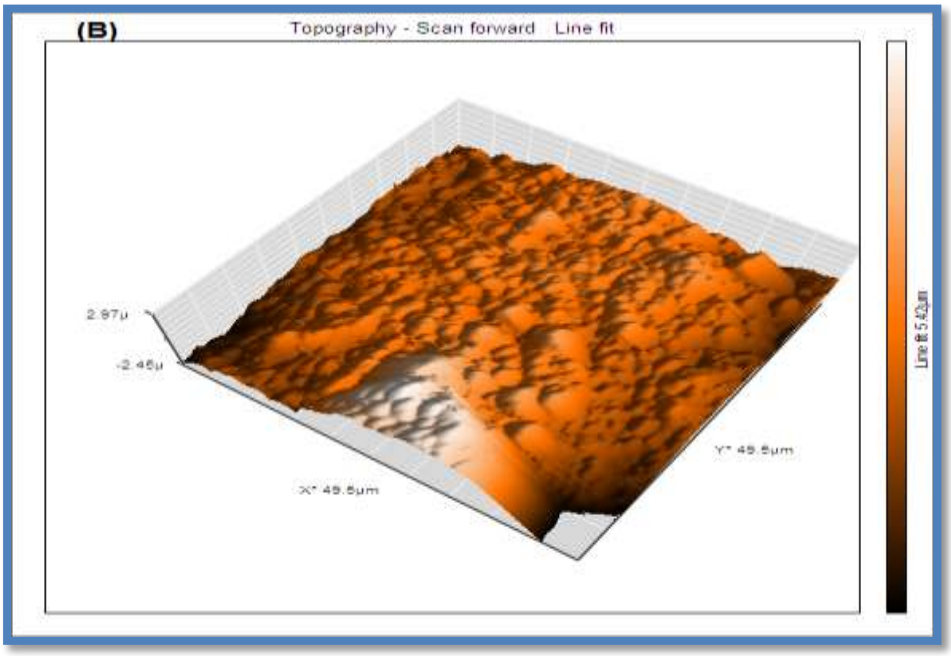


(A) 3D



(B) 3D

Figure 3: AFM microscope images of the sample $PbTiO_3$

3.3. TGA-DSC

Figure 4. shows the results of the TGA-DSC analysis obtained for $PbTiO_3$ powder. The DSC curve shows three regions (peaks) where the heat absorption at 156.4 ^oC is -10 µV and at 297.1 ^oC by -10.7µV and at 346.9 ^oC by -11.5 µV The TGA curve showed a small loss of weight at 590.5^oC by -1mg. It was found that the higher the temperature, the higher the heat absorbent peaks and the higher the weight loss, the higher the temperature of the sintering at or below 1000^oC.

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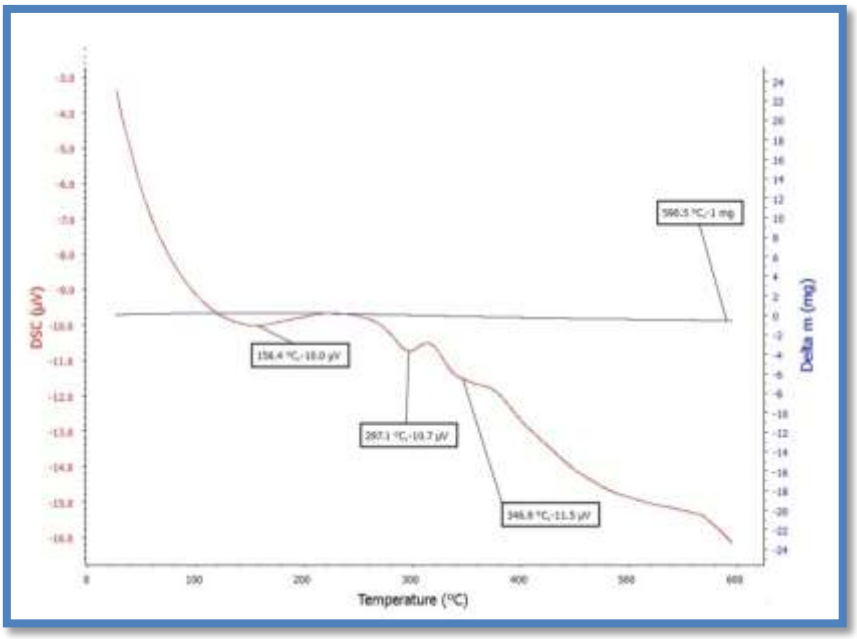

Figure 4: Curve TGA - DSC of the sample $PbTiO_3$

3.4. FTIR analysis

Figures 5. Showed a strong infrared absorption for the sample ($PbTiO_3$) while the strong absorption of infrared radiation at the top 579 cm^{-1} as a result of the stretching vibration Ti-O bond, There was also a strong infrared absorption of the $PbTiO_3$ model at the peak (717 cm^{-1}) and the peak vibration of that family extends to about (1745 cm^{-1}). It shows humidity absorption at (3016 cm^{-1}).

Table 4: Shows the results obtained from FTIR analysis absorption peaks (cm^{-1}) and The bond force constant (k) and average bond length (r).

sample	v (1/cm)	k (dyne/cm)	r (Å)
$PbTiO_3$	579	$2.36*10^7$	1.93

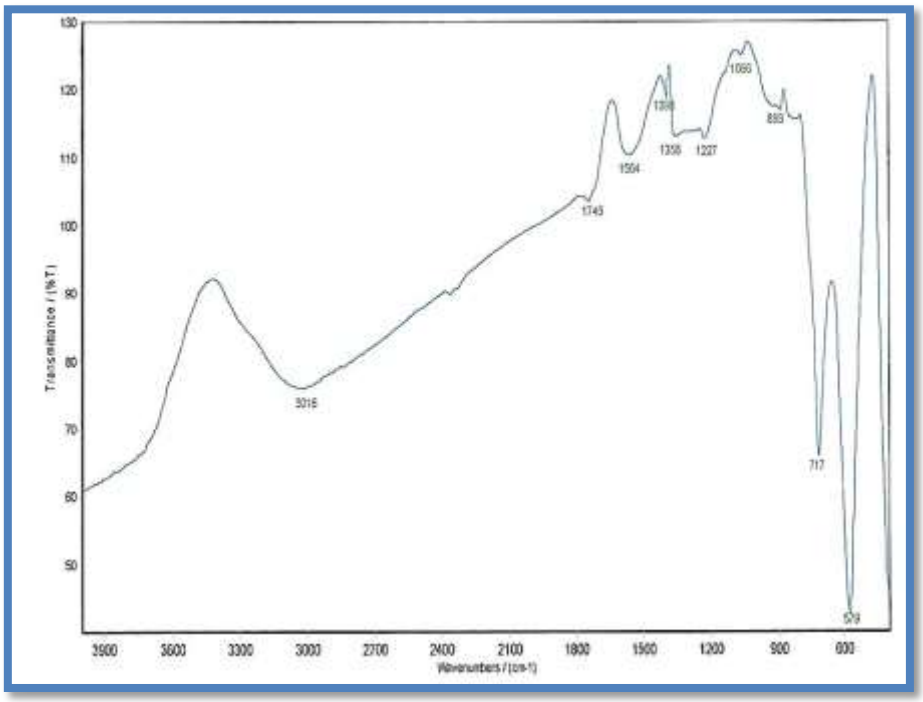

Figure 5: FT-IR spectra of a sample $PbTiO_3$.

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4. CONCLUSION

The Lead Titanate ($PbTiO_3$) materials prepared with good growth and purity in solid-state reaction method by calcination the mixture of (PbO-TiO_2) as a raw materials at1000 ^{0}C for 4 hr. The X-ray diffraction results confirmed the compound of ($PbTiO_3$ that structure was tetragonal. Also the results showed that the particle size of the AFM was greater than the particle size of the XRD.TGA-DSC Show low weight loss. The FT-IR analysis results showed that the constant force of the bond (k) increased as a result of the stretching vibration Ti-O bond.

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