CHARACTERIZATION OF STRUCTURAL AND MORPHOLOGICAL PROPERTIES OF $PbTiO_3$ POWDER

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Abstract

Lead titanate ($PbTiO_3$) powders were prepared by using solid state reaction method at different temperatures. The starting chemicals used in this work are lead oxide (PbO) and titanium dioxide (TiO_2). Phase identification and structural properties of $PbTiO_3$ powders were examined by X-ray diffraction XRD. From XRD result, $PbTiO_3$ powders were significantly formed with tetragonal symmetry. Surface morphology of $PbTiO_3$ powders was observed by Scanning Electron Microscopy (SEM). The lead titanate ($PbTiO_3$) powders were quite suitable for cost - effective and uncomplicated trends of ferroelectric materials such as random access memory (RAM).

Key words: lead oxide (PbO), titanium dioxide (TiO_2), lead titanate ($PbTiO_3$), X-ray diffraction (XRD), Scanning Electron Microscopy (SEM)

Introduction

The science and technology of nanostructured materials is advancing at a very rapid pace. Nowadays, the preparation and functionalization of one-dimensional nanostructured materials have become one of the most important roles of the nanotechnology. A perovskite is any material with the same type of crystal structure known as the perovskite structure with the oxygen in the face centers. Most of the perovskites contain oxygen as the anion. Hence, perovskite oxide can be presented by their general formula as ABO_3. Lanthanum manganite represents a class of perovskite oxide materials drew a lot of attention due to their intriguing and interesting properties like colossal magnetoresistance, ferroelectricity, superconductivity; charge ordering spin transport, thermo powder and the variation in these aforementioned properties according to change in their crystalline structure. Lanthanum manganite ($LaMnO_3$) and Barium Titanate ($BaTiO_3$) and Lead Titanate ($PbTiO_3$), etc. and these are based on the perovskite ABO_3 structure several ferroelectric materials have structure that are closely related to perovskite. Perovskite type oxide materials are important for electronic applications since they exhibit diverse physical properties such as super conductivity, dielectricity, ferroelectricity and magnetism.

Materials and Methods

Lead Oxide (PbO)

Lead oxide, also called lead (II) oxide or lead monoxide, is the inorganic compound with the molecular formula PbO. PbO occurs in two polymorphs: litharge having a tetragonal crystal structure and massicot having an orthorhombic crystal structure. Modern applications for PbO are mostly in lead-based industrial glass and industrial ceramics, including computer components. It is an amphoteric oxide.

Titanium Dioxide (TiO_2)

Titanium dioxide, also known as titanium (IV) oxide or titania is the naturally occurring oxide of titanium, chemical formula TiO_2. When used as a pigment, it is called titanium white,

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Pigment White 6, or Cl 77891. It is noteworthy for its wide range of applications from paint sunscreen to food colouring.

Titanium dioxide occurrences in nature are never pure; it is found with contaminant metals such as iron. The metal can also be mined from other minerals such as ilmenite or leucoxene ores, or one of the purest forms, rutile beach sand. Figure 1 shows the stating materials used in this research work.

Lead Oxide (PbO)

Titanium Dixide (TiO_2)

Figure 1 Starting materials

Solid State Reaction Method

The solid state reaction method is the most widely used method for the preparation of polycrystalline solids from a mixture of solid starting materials. Solids do not react together at room temperature over normal time scales and it is necessary to heat them to much higher temperatures, often to 1000 °C to 1500 °C in order for the reaction to occur at an appreciable rate. The factors on which the feasibility and rate of a solid state reaction depend include reaction conditions, structural properties of the reactants, surface area of the solids, their reactivity and the thermodynamic free energy change associated with the reaction.

Preparation of $PbTiO_3$ powders by Solid State Reaction Method

Lead titanate $PbTiO_3$ nanoparticles were successfully prepared by solid state reaction method. Lead oxide (PbO) and titanium dioxide (TiO_2) were weighed with digital balance and mixed with desired stoichiometric composition of $PbTiO_3$ nanoparticles. The mixture was ground by agate mortar to become a homogeneous mixture. The mixed $PbTiO_3$ powders were calcined at 500 °C, 600 °C and 700 °C for 1.5 h in furnace and reground with agate mortar. The reground $PbTiO_3$ powders passed through 100, 250 and 400 mesh sieves. Finally, homogeneous $PbTiO_3$ powders were obtained. In this section, the structural properties of $PbTiO_3$ powders were characterized by X-ray diffraction method (XRD). Figure 3 shows the block diagram of preparation of $PbTiO_3$ powders with different temperatures.

Mesh Sieving

Sieving is a simple technique for separating particles of different sizes. Many natural and manufactured materials consist of differently shaped and sized particles. Sieve analysis is used to divide the particulate material into size fractions and then to determine the weight of these fractions. In this way a relatively broad particle size spectrum can be analyzed quickly and reliably. Most sieving processes are carried out on dry materials. The particle size is characterized by mesh, the size of wire used in the screen. Mesh size is not a precise measurement of particle size. Screens can be made with different thicknesses of wires and the thicker the wires, the smaller the particles passing through that screen, and vice versa. The mesh size is the number of openings (little squares) in one inch of screen, so a 100 mesh screen means there are 100 openings across one linear inch of screen, a 400 mesh has 400 openings and so on. Therefore, as the mesh number increases, the particle size decreases (higher mesh number = finer powder). In this work, three-stage mesh sieves (100, 250, 400 mesh) shown in Figure 2 were used for sieving process.

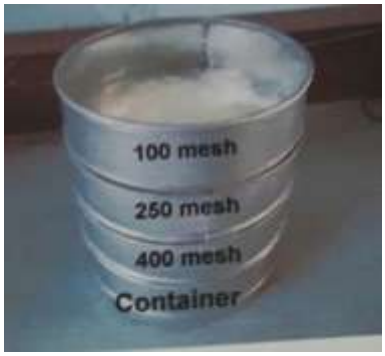


Figure 2 Photograph of Mesh Sieving

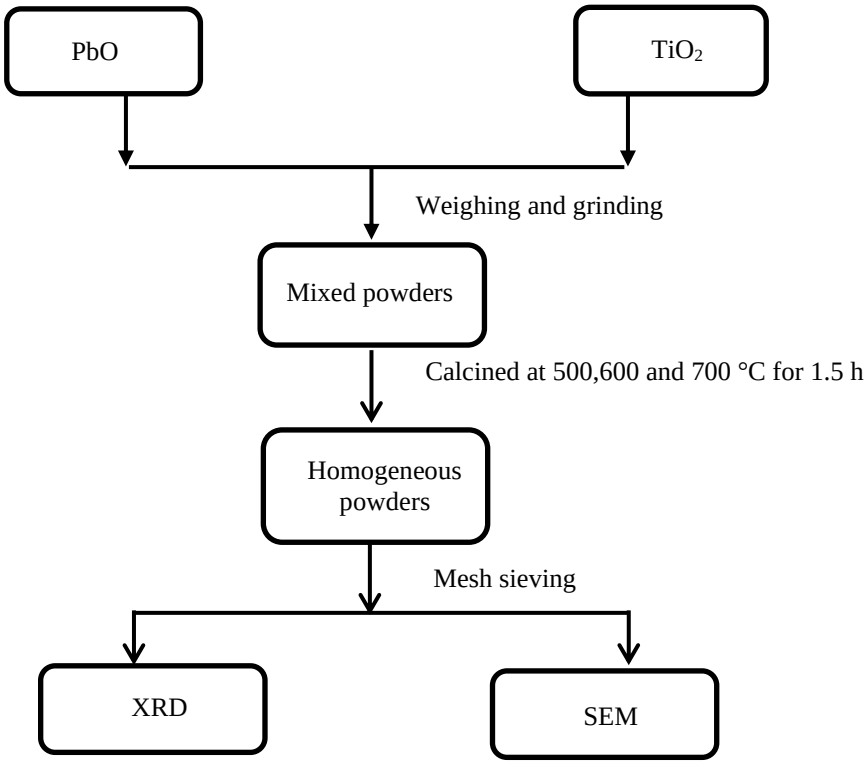


Figure 3 The block diagram of preparation of $PbTiO_3$ powders with different temperatures

Methods of Characterization

$PbTiO_3$ powders was characterized to determine their structural and morphological properties. The characterization techniques include X-ray diffraction technique (XRD) and Scanning Electron Microscopy (SEM).

Results and Discussion

XRD Analysis of $PbTiO_3$ Powders

The great deal of information about the crystallographic information of crystalline $PbTiO_3$ powder has been studied by crystallite size. The XRD patterns of $PbTiO_3$ specimens were perovskite type with tetragonal structure as shown in Figure 4 (a-c). There are several diffractions of the standard peaks were scanned within the diffraction angles range from 10° to 70°. $PbTiO_3$ powders were obtained and examined its phase formation by X-ray diffractometer using Cu-K_α radiation with wavelength of 1.54056 Å. All the peak heights and peak positions were in good agreement with the JCPDS (Join Committee on Powder Diffraction Standards) in 75-1605 > $PbTiO_3$ library file.

The average crystallite-sizes were 23.992, 21.613, 27.245 nm for different temperatures respectively. For the powder, 2θ, FWHM values and crystallite sizes are given in Table 2-3. The lattice parameter (a and c) and lattice distortion (lattice strain) c/a of dominant peaks are described in Table 1. The crystallite size was calculated using a well-known Debye Scherrer's formula. Thus, the $PbTiO_3$ powders were successfully obtained by solid state reaction method with tetragonal structure.

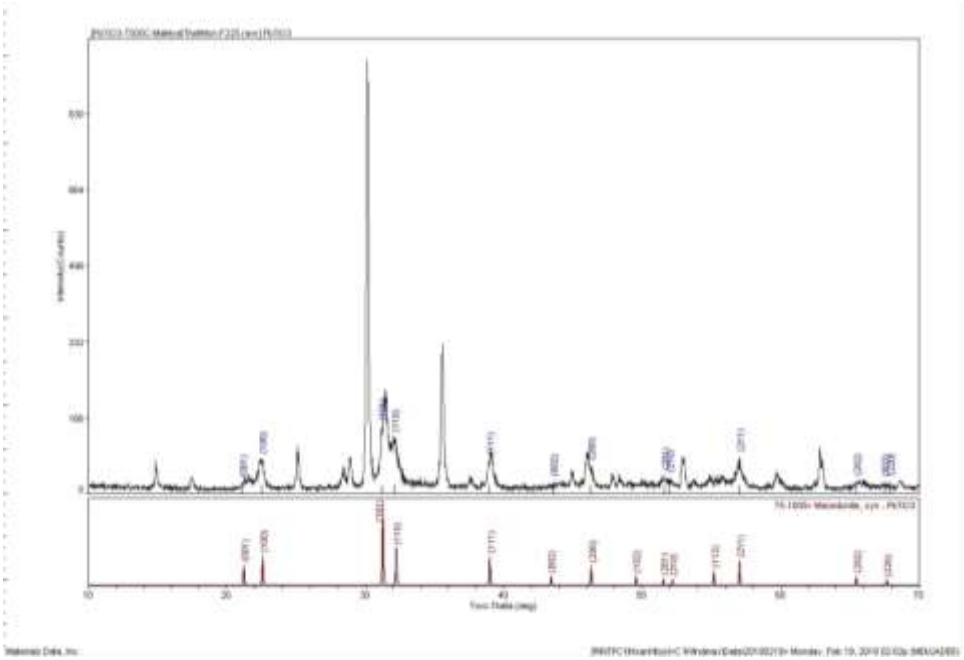


Figure 4 (a) The XRD patterns of $PbTiO_3$ powders at 500 °C

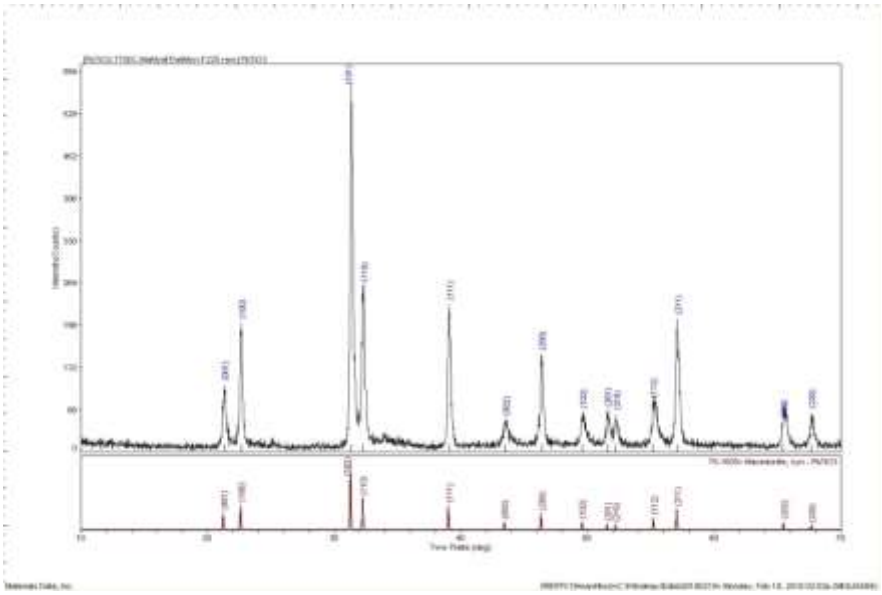


Figure 4 (b) The XRD patterns of $PbTiO_3$ powders at 600 °C

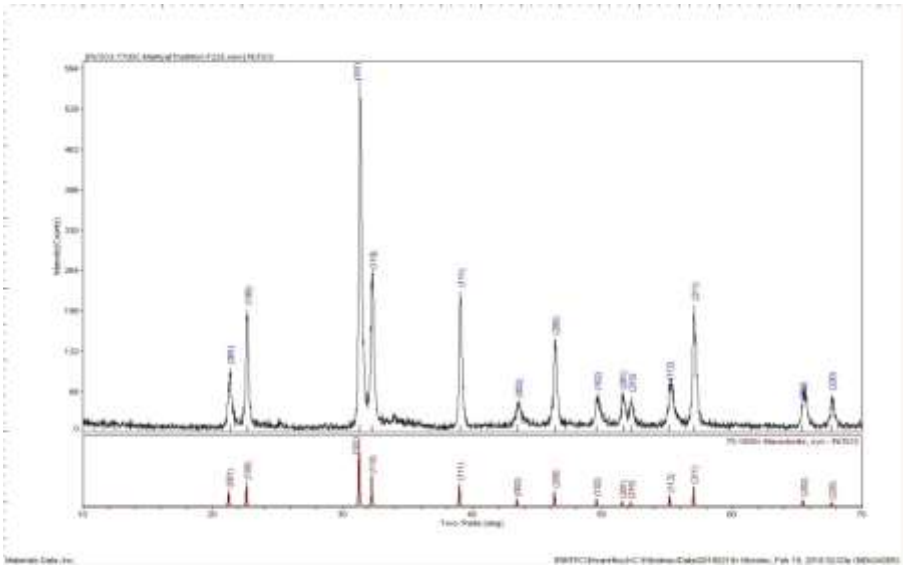


Figure 4 (c) The XRD patterns of $PbTiO_3$ powders at 700 °C

Table 1 Lattice Parameters and c/a ratios of different Temperatures for $PbTiO_3$ Powders

Temperatures	Lattice Parameter (Å)		Tetragonality c/a
	a	C	
500 °C	3.9263	4.2016	1.0701
600 °C	3.9227	4.1768	1.0648
700 °C	3.9167	4.1598	1.0621

Table 2 Structural Properties of $PbTiO_3$ Powders at 700 °C

Sr No.	(h k l)	2 θ (deg)	FWHM (deg)	Observed Crystallite Size (nm)
1	(001)	21.338	0.242	33.190
2	(100)	22.653	0.223	36.002
3	(101)	31.347	0.261	30.710
4	(110)	32.288	0.273	29.420
5	(111)	39.071	0.256	31.330
6	(002)	43.501	0.502	15.990
7	(200)	46.361	0.285	28.180
8	(102)	49.584	0.380	21.120
9	(201)	51.563	0.251	31.980
10	(210)	52.245	0.354	22.660
11	(112)	55.120	0.484	16.570
12	(211)	57.071	0.305	26.330
13	(202)	65.385	0.264	30.380
14	(220)	67.640	0.291	27.570
Average crystallite size				27.245

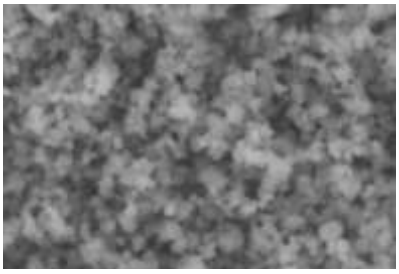
Table 3 Structural Properties of $PbTiO_3$ Powders at 500 °C and 600 °C

Sr No.	(h k l)	2 θ (deg)	FWHM (deg)	Observed Crystallite Size (nm)
1	(001)	21.128	0.375	21.380
2	(100)	22.536	0.474	16.930
3	(101)	31.195	0.322	24.920
4	(110)	32.114	0.718	11.180
5	(111)	38.940	0.527	15.230
6	(002)	43.571	0.100	80.260
7	(200)	46.264	0.549	14.620
8	(201)	51.577	0.521	15.400
9	(210)	51.947	0.561	14.310
10	(211)	57.028	0.410	19.560
11	(202)	65.451	0.515	15.580
12	(003)	67.491	0.256	31.330
13	(220)	67.910	0.167	48.130
Average crystallite size				23.992

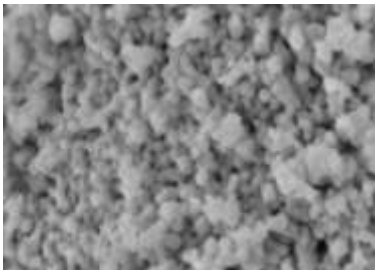
Sr No.	(h k l)	2 θ (deg)	FWHM (deg)	Observed Crystallite Size (nm)
1	(001)	21.239	0.303	26.470
2	(100)	22.552	0.267	30.050
3	(101)	31.282	0.320	25.050
4	(110)	32.235	0.277	28.990
5	(111)	39.005	0.254	31.610
6	(002)	43.388	0.483	16.610
7	(200)	46.373	0.351	23.230
8	(102)	49.463	0.721	11.130
9	(201)	51.519	0.318	25.230
10	(210)	52.132	0.587	13.660
11	(112)	55.223	0.549	14.620
12	(211)	57.036	0.354	22.660
13	(202)	65.468	0.380	21.120
14	(003)	67.566	0.604	13.290
15	(220)	67.719	0.392	20.480
Average crystallite size				21.613

SEM Analysis of $PbTiO_3$ Powders

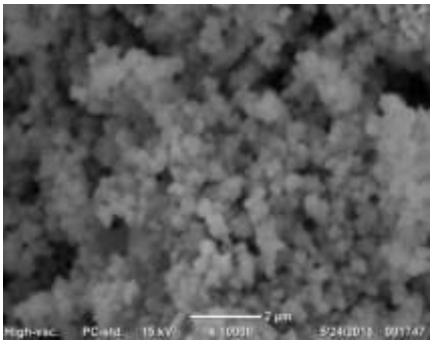
Figure 5 (a-c) shows the SEM analysis of $PbTiO_3$ powders annealed at 500 °C, 600 °C and 700 °C, exhibit grained microstructure and nanostructure with small grain size. The grain sizes were calculated by using well known bar code system. The average grain sizes of $PbTiO_3$ powder were found to be about 260 nm at 500 °C, 275 nm at 600 °C and 320 nm at 700 °C respectively. These figures indicates that most of the grain size was regular structure and a few number of large grain size were found. It looks fairly dense and smooth. From the images, they were clearly found that the little amount of pores and grain growth were examined. The orientation of grain was towards left for all images. The grain size of $PbTiO_3$ powders seen to be uniform but some of the grain size were slightly large.



(a) $PbTiO_3$ powders at 500 °C (average grain size = 260 nm)



(b) $PbTiO_3$ powders at 600 °C (average grain size = 275 nm)



(c) $PbTiO_3$ powders at 700 °C (average grain size = 320 nm)

Figure 5(a- c) The SEM images of $PbTiO_3$ powders at 500 °C, 600 °C and 700 °C

Conclusion

$PbTiO_3$ powders with different temperatures were successfully prepared by solid state reaction method. In the XRD analysis produces the information on crystallite sizes of several peaks were 23.992 nm at 500 ºC, 21.613 nm at 600 ºC and 27.245 nm at 700 ºC respectively. The crystal structure of the prepared $PbTiO_3$ powder was perovskite type with tetragonal symmetry. From SEM analysis, the average grain size of $PbTiO_3$ powders were found to be about 260 nm at 500 °C, 275 nm at 600 °C and 320 nm at 700 °C respectively. The grain size of $PbTiO_3$ powders seen to be uniform but some of the grain size were slightly large. As these results, the crystallite size of $PbTiO_3$ powders at 600 ºC was smallest than other two temperatures and the average grain size at 500 ºC was smallest grain size of these three temperatures. According to research data, the lead titanate ($PbTiO_3$) powders were quite suitable for cost effective and uncomplicated trends of ferroelectric materials such as random access memory (RAM).

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