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Barium Titanate ($BaTiO_3$) synthesized by sol-gel auto-combustion method

[1]Gaikwad AS, [2]More SS, [3]Kathare RV, [4]Mane ML, [1]Borade RB, [5]Vijapure YA and [1*]Kadam AB

[1]Physics Department, Jawahar Mahavidyalaya, Anadur, Dist. Osmanabad (M.S.) India
[2]Physics Department, Y.C. College, Tuljapur, Dist. Osmanabad (M.S.) India
[3]Physics Department, KMJ Mahavidyalaya, Washi, Dist. Osmanabad (M.S.) India
[4]Physics Department, SGRG Shinde Mahavidyalaya, Paranda, Dist. Osmanabad (M.S.) India
[5]Chemistry Department, Shrikrishna Mahavidyalaya, Gunjoti, Dist. Osmanabad (M.S.) India
*Corresponding Author: drabkadam@gmail.com

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ABSTRACT

$BaTiO_3$ (BTO) has been synthesized by sol-gel auto-combustion method. The prepared nanoparticles were sintered at 700 °C for 2 hrs. The X-ray diffraction (XRD) pattern was used to study the phase transformation and crystal structure of BTO. All reflections XRD are corresponding to the cubic structure of BTO indicating polycrystalline and single phase formation BTO nanoparticles without the formation of any other secondary phases. The calculated lattice parameter of BTO is found to be a = 3.99 Å and crystallite size was determined as t_{xrd} = 75 nm. Scanning electron microscopy was used to investigate the grain size and their morphology. The smaller crystallite size and grain size confirmed the nano-crystalline nature of prepared BTO powder. Spontaneous electric polarization with applied electric field hysteresis loop was measured to investigate the ferroelectric properties of BTO.

Key words: Sol-gel, XRD, PE loop

Principal
Jawahar Arts, Science & Commerce College,
Andur Tal. Tuljapur Dist. Osmanabad

INTRODUCTION

Materials possessing a large dielectric constant and electric polarization have gained a great deal of attention. Since the discovery of ferro-electricity in a single crystal of Rochelle salt in 1921 [1], there have been many attempts to find new materials which possess a high dielectric constant and electric polarization. The urgent demand for ceramic capacitors with high electric polarization has been a key issue leading to the development of ceramic capacitor technology. High dielectric constant materials are desirable for the miniaturization of capacitors required for integrated circuits in electronic devices [2]. Ferroelectric materials are used in many modern devices, such as piezoelectric actuators and electro-optic modulators. Materials of the ABO_3 family (A and B are cations and O is oxygen), such as $BaTiO_3$, $PbTiO_3$ and $SrTiO_3$, have received a significant amount of attention due to their ferroelectric and electro-optic properties. $BaTiO_3$ (BTO) is of fundamental and technological importance due to the strong coupling of its electric polarization to the electric field and stress field. It is a well-studied prototype displacive ferroelectric system offering a good platform to investigate novel effects. It has many applications such as in ferroelectric and multilayer ceramic capacitors (MLCC) [3–5] owing to its high dielectric constant and tunable microwave devices, which in turn is due to its high and field dependent dielectric constant in its paraelectric state. Semi-conducting BTO can be used for CO gas sensor by making use of its PTCR effect [6,7].

In ferroelectric fine particles, ferroelectricity decreases with decreasing particle grain size and disappears below a certain critical size [8,9]. Other physicochemical factors, such as the density, shape, presence of impurities and structural defects, also affect this property. During the last few years, studies to improve the quality of perovskite powders have concerned the sol-gel process [10] and chemical methods [11,12]. These studied are directed mainly to the synthesis of fine grained powders [13], thin coatings [14], and monolithic bodies [15] of controlled particle size and morphology and with controlled final microstructure [16]. The sol-gel method is a chemical technique including the following stages: mixing of metallic alkoxides independently or in combination with acetates, oxalates and inorganic salts; addition of distilled water under which hydrolysis and condensation reactions take place. These are finalized to obtain an amorphous gel under drying and heating. This method allows additives to be introduced into the synthesized powders in order to modify the properties of the ceramic. The advantages of this procedure are high purity, homogeneity and low sintering temperatures. Dutta et al. [17] have proposed that a relationship exists between the tetragonal BTO and the type of counter ion used in the synthesis. Although the exact mechanism of this process is not yet known, the authors suggested that most soluble salts can promote the dissolution of BTO, disturbing the dissolution recrystallization process. Thus, a better understanding of the structure-property relationship of perovskite Nano crystals is highly desirable, which is also necessary for developing high- performance electronic devices. In this work we are reporting the synthesis of BTO nanoparticles by low temperature sol-gel auto-combustion method.

METHODOLOGY

The powders of BTO were synthesized by sol-gel auto-combustion. Analytical grade citric acid Titanium butoxide $Ti(OC_4H_9)_4$ (99.5% pure) was diluted in absolute ethanol (99.5% pure) and barium acetate $(Ba(OOCCH_3)_2)$ were dissolved in acetic acid. These solutions were mixed together and then stirred in order to make them homogeneous. Reaction procedure was carried out in air atmosphere without protection of inert gases. The molar ratio of metal nitrates to citric acid was taken as 1:3. The metal nitrates were dissolved together in a minimum amount of double distilled water to get a clear solution. An aqueous solution of citric acid was mixed with metal nitrates solution, then ammonia solution was slowly added to adjust the pH at 7. The mixed solution was kept on to a hot plate with continuous stirring at 90 °C. During evaporation, the

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solution became viscous and finally formed a very viscous brown gel. When finally all water molecules were removed from the mixture, the viscous gel began frothing. After few minutes, the gel automatically ignited and burnt with glowing flints. The decomposition reaction would not stop before the whole citrate complex was consumed. The auto-combustion was completed within a minute, yielding the brown-colored ashes termed as a precursor. The as prepared powder then annealed at 700 °C for 2 h.

The samples were powdered for X-ray investigations. Part of the powder was X-ray examined by Phillips X-ray diffractometer (Model 3710) using Cu-K_α radiation (λ=1.5405Å). Surface morphology and grain size were observed using scanning electron microscope (SEM, JEOL JSM-6300). Ferroelectric properties of BTO sample was studied by using P-E hysteresis loop tracer system.

RESULTS AND DISCUSSIONS

The structure and phase purity of the samples were confirmed by analyzing the observed powder X-ray diffraction patterns. Figure 1 represents X-ray powder diffraction patterns of BTO nanoparticle sample. It seen that crystallized BTO phase can be obtained at heating temperature 700°C for 2 hrs, indicating that single phase of BTO presented in the prepared pure samples. It is evident that peaks at 2θ = 22.21, 31.58, 38.92, 45.31, 50.73, 56.11, 66.08, 70.48 and 74.86, 79.23° indexed as (1 0 0), (1 1 0), (1 1 1), (2 0 0), (2 1 0), (2 1 1), (2 2 0), (2 2 1) and (3 0 1) diffraction line of cubic phase appeared, respectively.

The lattice parameter '*a*' was calculated using the following equation [18],

$$a = d\sqrt{(h^2 + k^2 + l^2)} \qquad (1)$$

where, *d* is the inter-planer spacing and (*hkl*) is the index of the XRD reflection peak. The calculated lattice constant is *a* = 3.99 Å, revealing the crystal structure to be cubic.

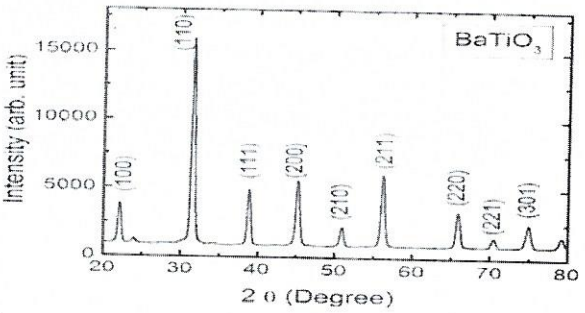

Fig. 1: XRD pattern of $BaTiO_3$ nanoparticles prepared by sol-gel auto-combustion method and sintered at 700 °C for 2 hr.

The average crystallite diameter 'D_{XRD}' of powder estimated from the most intense (311) peak of XRD and using the Scherrer method [18],

$$D_{XRD} = \frac{C\lambda}{B_{1/2}\cos\theta} \qquad (2)$$

where $B_{1/2}$ is the full width of half maximum in (2θ), θ is the corresponding Bragg angle and C = 0.9. The calculated crystallite size is 75 ±5 nm, confirming the nanoparticle formation of BTO sample synthesized by sol-gel method. The bulk density (d_B) measured using the formula

$$d_B = \frac{m}{\pi r^2 h} \qquad (3)$$

where m is mass, r is the radius and h is the height of the pellet. The percentage porosity is calculated using following the relation,

$$P = \left(\frac{d_X - d_B}{d_X}\right) \times 100 \qquad (4)$$

where dx and d_B are the X-ray density and bulk density respectively. It is observed that the bulk density is 5.3 g/cm^3 whereas percentage porosity is 15%.

The surface and grain morphology of the prepared sample is studied by scanning electron microscopy (SEM). Scanning electron micrographs (SEM) of the surfaces of the BTO sample is shown in Fig. 2, which is characterized by a typical porous structure and small rounded grains. It can be observed from the SEM images that the prepared samples are amorphous and

porous in nature. The particles were well distributed and slightly agglomerated. The observed grain size is around 150 ±5 nm.

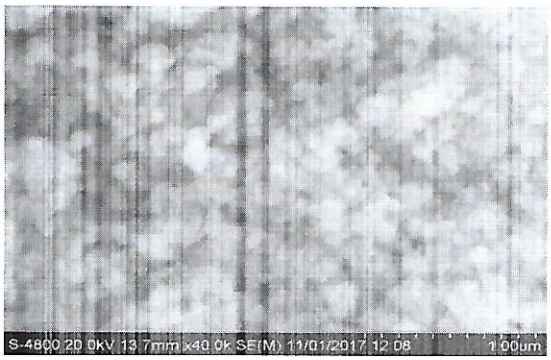

Fig. 2: SEM image of $BaTiO_3$ nanoparticles prepared by sol-gel auto-combustion method and sintered at 700 °C for 2 hr.

To investigate the ferroelectric properties of $BaTiO_3$ at room temperature P-E loop is recorded and shown in fig.3. The observed P-E loop is lossy and not saturated. The leakage current may be due to porous nature of sample. The maximum polarisation of BTO is observed 2.23μc/cm².

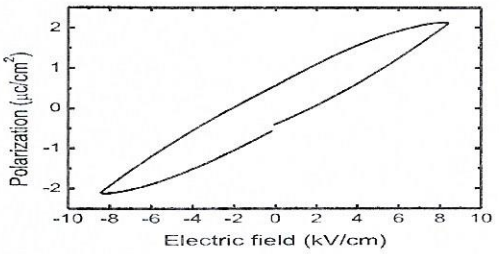

Fig. 3: Polarization (P) versus electric field (E) hysteresis loop of $BaTiO_3$ nanoparticle prepared by sol-gel auto-combustion method and sintered at 700 °C for 2 hr.

CONCLUSION

Nanocrystalline $BaTiO_3$ has been successfully synthesized by sol-gel auto-combustion method. XRD analysis revealed the formation of single phase BaTiO3 without the presence of any other secondary phases. The crystalline size is calculated and found to be t_{xrd} = 75 nm which confirm the nanocrystalline nature of prepared $BaTiO_3$ powder. Using scanning electron microscopy the grain size and morphology is investigated. Ferroelectric properties of $BaTiO_3$ are studied by using P-E loops.

REFERENCES

1. Valasek J. Piezo-electric and allied phenomena in Rochelle salt, Phys. Rev.,17,1921,475–481.
2. Laxman Singh, Uma Shanker Rai, Kam Deo Mandal, Byung Cheol Sin, Hyung-il Lee, Hoeil Chung, Youngil Lee. Materials Characterization,96 ,2014, 54–62
3. Tsurumi T, Hoshina T, Takeda H, Mizuno Y, Chazono H. IEEE Trans. Ultrason., Ferroelectr. Freq. Control,56,2009,1513–1522.
4. Okino Y, Shizuno H, Kusumi S, Kishi H. J. Appl. Phys.,33,2004, 5393.
5. Choi KJ et al. Science, 306,2004,1005–1009.
6. Bi Z, Aga H, Ramanan SR. J. Electroceram.,28,2012.109–117.
7. Tang Z, Zhou Z, Zhang Z. Sens. Actuators, B 93,2003,391–395.
8. Hoshina T, Wada S, Kuroiwa Y, Tsurumi T. Applied Physics Letters, 93,2008, 192914–192917.
9. Sundaresan A, Bhargavi R, Rangarajan N,.Siddesh U, Rao CN. Physical Review, B.74, 2006, 161306–161310
10. Mazdiyasni KS. Ceramics Bull., 63(4), 1984, 591.
11. Chaput F, Boilot JR. High Tech. Ceramics, 1987, 1459.
12. Hayashi Y, Kimura T, Yamaguchi T. J. Mater. Sci., 21, 1986, 757.
13. US Patent 3,647,964 (1972).
14. Yanoskaya MP et al. Inorganic Materials, 17(2),1981, 307.
15. Rehspringer L, Poix P, Bernier JC. J. Non- Cryst. Solids, 82, 1986, 286.
16. Mosset A, Guatier-Luneau I, Galy J. J. Non-Cryst. Solids, 100 , 1988, 339.
17. Dutta P, Asiaie R, Akbar S, Zhug W. Chemistry Material, 6, 1994, 1542–1548.
18. Gurav SK, Shirsath SE, Kadam RH, Patange SM, Lohar KS, Mane DR. Materials Research Bulletin, 48,2013,3530.