International Journal of Multidisciplinary Innovative Research
ISSN: 2583-0228 Volume 4, Number 2 (Apr' 2024) pp. 1-5

https://www.ijmir.org
Article Received: 25 Dec' 2023; Accepted: 22 Jan' 2024.

*Research Article*

# Sol Gel Synthesis and Characterization of Barium Bismuth Titanate Ceramics at Lower Temperature

Faizal Khan[1], Soumya Mukherjee[2] and Sathi Banerjee[1]

[1]*Department of Metallurgical & Materials Engineering, Jadavpur University, Kolkata, India.*

[2]*Department of Metallurgical Engineering, Kazi Nazrul University, Asansol, India.*

*Corresponding author email: smmukherjee4a@gmail.com*

***ABSTRACT: Sol-Gel route using AR Grade precursors are used to synthesize Barium Bismuth Titanate ceramics. The intermediate gel obtained is made to undergo thermal analyses by DSC- TGA to yield information on crystallization of ceramic phase development. Heat treatment of gel is carried in air atmosphere at about 600℃, 550°C, 500C for 4hours, 6 hours and 5 hours respectively. Phase developed by heat treatment is confirmed from XRD as per JCPDS standard file. Crystallite size is estimated by Scherrer's formula while planes are indexed as per feasibility of thermodynamic stability. Optimized condition for high purity indexed phase development is obtained at about 500C for 5 hours duration. Bonding analysis is carried by FTIR analyses to determine M-O co-ordinations. Both XRD and FTIR analysis corresponds with the experimental findings to elucidate the ceramic formation. FESEM analysis exhibits the morphological features of the synthesized sample and micro-structural evolution with heat treatment temperature and duration. Dense agglomerates with irregular polygonal, spherical to flaky structure are noted in some portions of the micro-structural evolution of the synthesized sample.***

***KEYWORDS: Barium Bismuth Titanate, Thermal analyses, Phase, Bonding, Morphology.***

## INTRODUCTION

Ferroelectric materials are of vital importance for device applications in electronic components. Aurivillius family of compounds like BBT is also found to be ferroelectric in nature with versatile possible applications. This class of compound being a layered structure are ferroelectric in nature possessing good fatigue endurance which can be tailored to overcome problems associated with previous ferroelectric memories. Layered structure Aurivillius family of compound based on Bi is found to possess low operating voltages, superior polarization fatigue resistant characteristics and high Curie temperatures. Due to such properties, there is possibility for applications ferroelectric random access memory devices (FRAM), piezoelectric resonators and sensors.[1] At present scenario, ferroelectric solids are based on ferroelectric (FE) solids, which are typically poly-crystalline ceramics, for utilization in sensors, transducers, actuators, etc. based on their electrical signal. In case of photonic applications for future there is a need to couple same functional performance with the additional requirement that the materials be transparent to light. To fill this need, development of Barium Bismuth Titanate at low temperature is emerging. Among the various methods adopted to synthesis of

ferroelectric materials, the sol-gel route has attracted considerable attention in recent decades.[2, 3, 4, 5, 7]

In this paper, preparation of BBT precursor by modified sol gel process, as well as the structure and the characteristic of BBT powder have been studied by DSC-TGA, X-ray diffraction (XRD), FTIR, FESEM.

## EXPERIMENT

In the experiment, barium acetate ($Ba(CH_3COO)_2$), bismuth nitrate($Bi(NO_3)_3{\cdot}5H_2O$) and tetra butyl titanate ($Ti(OC_4H_9)_4$) were used as starting materials. Glacial acetic acid ($CH_3COOH$) selected as solvents, ethanolamine ($H_2NCH_2CH_2OH$) as complexation reagent, and acetylacetone ($CH_3COCH_2COCH_3$) as reagent to stabilize tetra butyl titanate. 99% pure Bi-nitrate pentahydrate was air dried at 60°C for 5 hours to prevent excessive hydrolysis of $Ti(OC_4H_9)_4$ followed by solvation in glacial acetic acid. The obtained solution was mixed with $Ba(CH_3COO)_2$ acetum only after the solution was transparent. Tetra butyl titanate was stabilized using acetylacetone. The resultant modified tetra butyl titanate was added to the Bi-Ba acetum mixed solution with constant stirring at room temperature. Viscosity adjustment is possible by reducing the surface tension of the precursor along with prevention of hydrolyzation of bismuth nitrate in acetic acid, ethanolamine was added to the solution under ultrasonic agitation [6], pH value was adjusted using glacial acetic acid to remain about 3.5. The resultant solution was filtered to get remains of BBT on filter paper. This BBT remains is collected in Al Boat and is heat treated at about 600°C, 550°C, 500°C for 4hours, 6 hours and 5 hours respectively. The powder collected after heat treatment was studied by XRD (Rigaku, Ultima III), DSCTGA (Perkin Elmer Make), FTIR (Shimadzu, Prestige IR21), FESEM.

## RESULTS AND DISCUSSION

Thermal analysis of sample is carried to determine the crystallization of ceramics. Thermal analyses are carried in presence of nitrogen atmosphere till 1000°C. Three endothermic peaks at about 600°C and 700°C respectively while exothermic hump centered at about 900°C is noted. Prominent weight losses are noted at 100-200°C which is about 7wt% while another in the range 500-700°C which is almost about 27wt%. Weight losses are found to diminished at around 800°C onwards. Thus, crystallization range is noted to be around 800°C for proper phase development of the material from precursors.

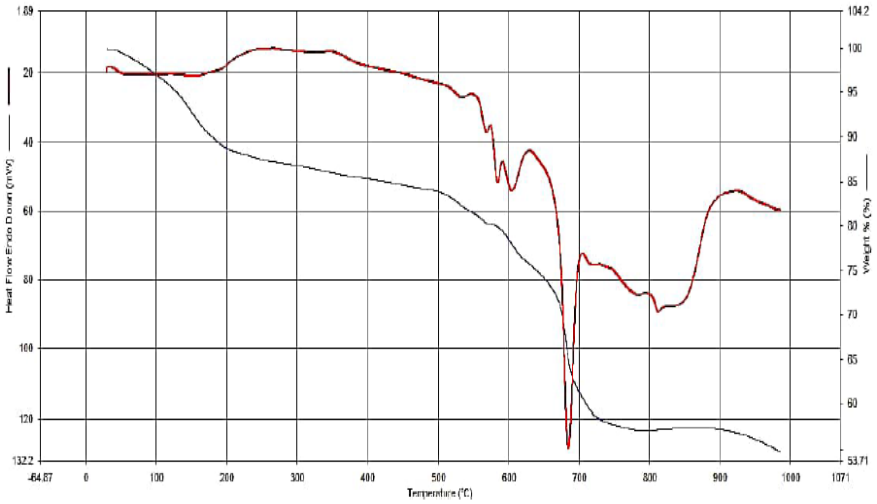


**Figure 1: DSC-TGA of synthesized samples before annealing**

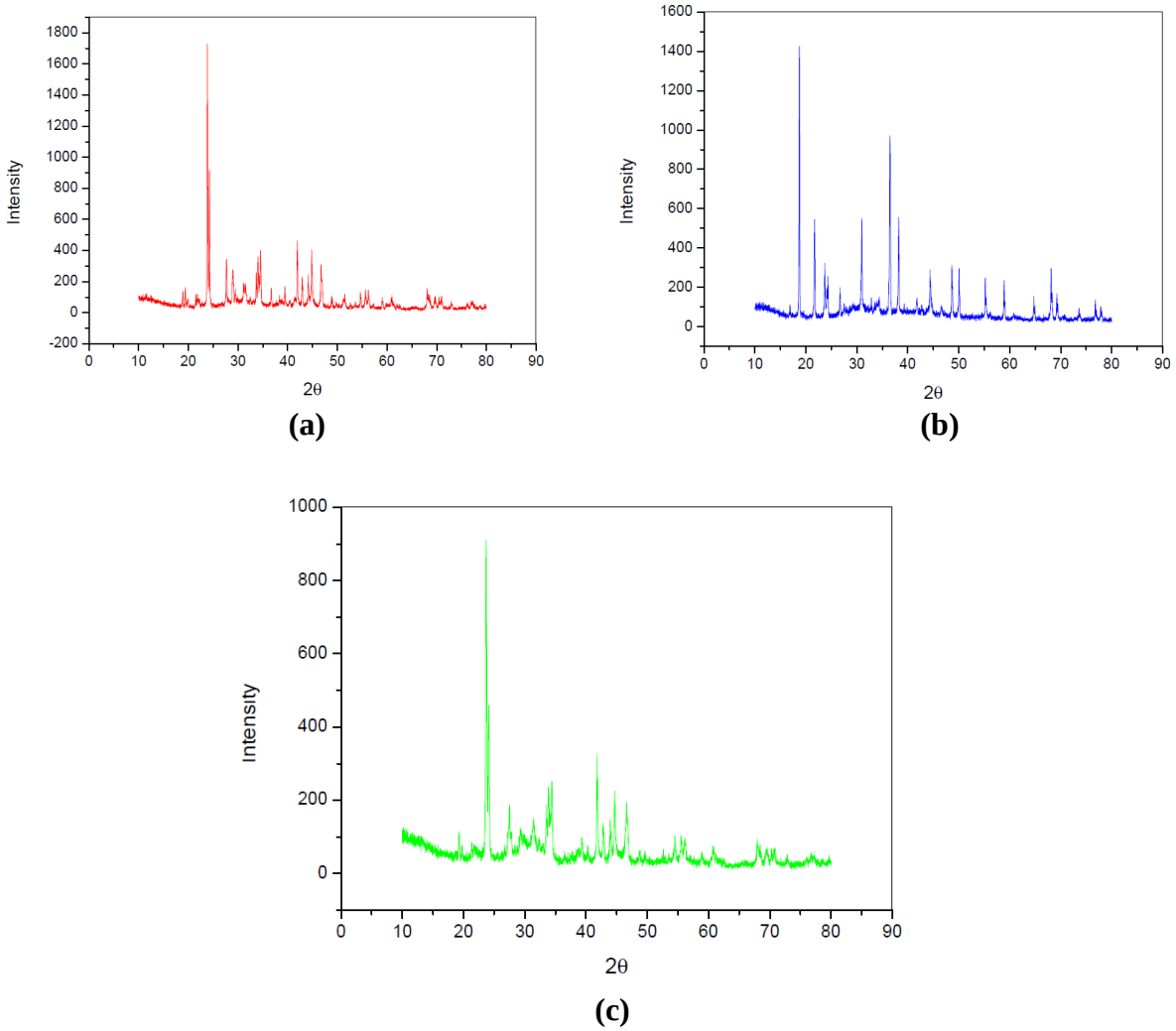


**Figure 2: XRD of synthesized samples (a) 600°C 4 hours; (b) 500°C 5hours and (c) 550°C for 6 hours**

XRD confirms the phase development after annealing at 600°C for 4 hours, 500°C for 5 hours and 550°C for 6 hours as per JCPDS card no 8-261. Amongst them 500°C for 5 hours yield the best result for phase development with all peaks got indexed as per planes from JCPDS. The other two yield some minor impurity phase but the major peaks got indexed as per planes of thermodynamic feasibility confirming the formation of barium bismuth titanate.

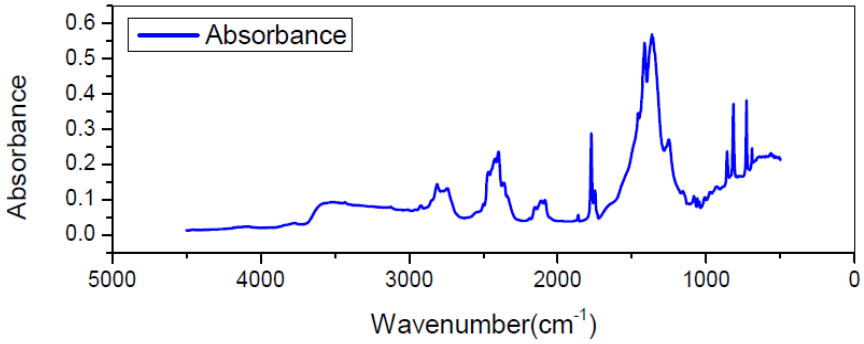


**Figure 3: FTIR of synthesized sample after annealing at 600°C for 4hours**

FTIR spectra analysis is carried using KBr as reference. The spectra positions at about 800-1100cm$^{-1}$ are due to Si-O-Si bond. The bending vibration of Si-O-Si bond is noted at about

860cm$^{-1}$ while those close to about 950cm$^{-1}$ is possibly for symmetric stretching vibration of Si-O bonds. The spectra within 480-680cm$^{-1}$ are attributed to Bi-O bonds while those around 730-750cm$^{-1}$ corresponds to asymmetric stretching of Ti-O tetrahedral units.[8]

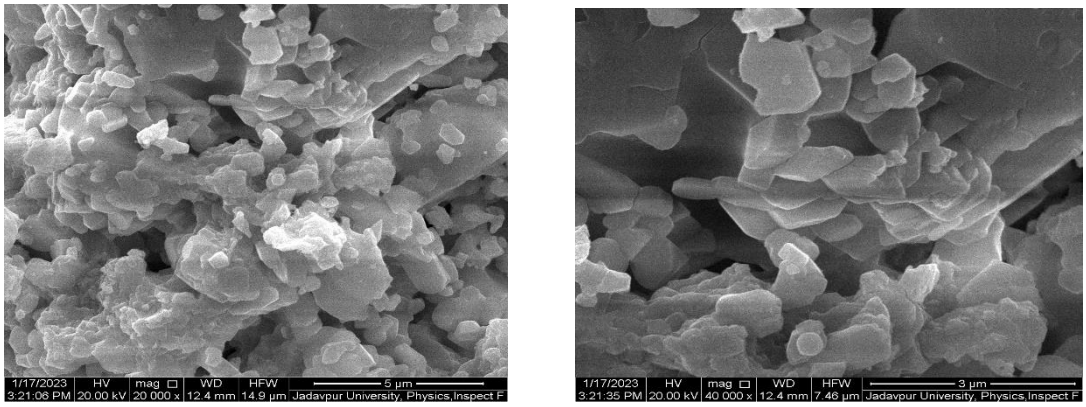


**Figure 4: SEM of synthesized sample after annealing at 500°C for 5hours**

SEM morphology indicates dense agglomerates with negligible porosity. Particulates are rectangular parallelopipeds polygon shape with some spherical. Individual particulates are close to 100-300nm in dimensions. Agglomerates are irregular shaped with slight flakes. Irregular fracture is noted in some portions with some elongated grains.

## CONCLUSION

Sol-gel route was carried to successfully synthesized barium bismuth titanate after Heat treatment. Heat treatment was carried at about 600℃, 550°C, 500C for 4hours, 6 hours and 5 hours respectively. XRD peaks of samples matches with the JCPDS data suggesting formation of BBT phase. Crystallite size decreases with increase in annealing temperature at constant soaking period. FTIR analysis exhibits the required M-O coordination of Si-O-Si bonds, Bi-O bonds and Ti-O bonds respectively. SEM morphology indicates dense agglomerates with negligible porosity. Particulates are rectangular parallelopipeds polygon shape with some spherical one in nature. Agglomerates are irregular shaped with slight flakes. Irregular fracture is noted in some portions.

## ACKNOWLEDGEMENT

Authors would like to acknowledge Department of Metallurgical & Materials Engineering, Jadavpur University and department of Physics for synthesis, Characterization facilities provided for the experiments.

### REFERENCES

[1] S. Mukherjee, M. Shahnawaz, and S. Banerjee, "Phase Development of Barium Bismuth Titanate by Modified Solid State Route." Mat. Sci. Res. India., vol. 17, no. 2, 2020, pp. 129-135.

[2] Xie, Dan, and Wei Pan. "Study on BaBi4Ti4O15 nanoscaled powders prepared by sol–gel method." Mater. Lett., vol. 57, no. 19, 2003, pp. 2970-2974.

[3] J. M. Boulton, G. Teowee, W.M. Bomnersbach and D.R. Uhlmann, "Sol-gel derived barium sodium niobate and bismuth titanate films." MRS Online Proceedings Library (OPL), vol. 243, no. 303, 1991,

[4] A. V. Murugan, S.C. Navale, and V. Ravi, “Preparation of nanocrystalline ferroelectric BaBi4Ti4O15 by Pechini method”, Mater. Lett., vol. 60, 2006, pp.1023–1025.

[5] L. Simon Sveyrat, A. Hajjaji, Y. Emziane, B. Guiffard and D. Guyomar, "Re-investigation of synthesis of BaTiO3 by conventional solid-state reaction and oxalate coprecipitation route for piezoelectric applications." Ceram. Int. vol. 33, no. 1, 2007, pp.35-40.

[6] D. Xie, W. Pan, T.L. Ren and Li. T. Liu, "Synthesis and Microstructure of Barium Bismuth Titanate Thin Films Using a Sol-Gel Technique." Key Engineering Materials. Vol. 280. 2005, pp. 845-848.

[7] J. Brendan Kennedy, Y. Kubota, Brett. A. Hunter, Ismunandar and K. Kato., "Structural phase transitions in the layered bismuth oxide BaBi4Ti4O15." Solid state communications vol. 126, no. 12, 2003, pp.653-658.

[8] A. Narayan Sah, S. Mukherjee, M. Shahnawaz and S. Banerjee, "Phase formation and Morphological features of Calcium Copper Titanate by Modified Solid State Process." Mat. Sci. Res. India., vol. 16, no.1, 2019, pp. 37-42.