Microwave assisted High Energy Ball Milling Synthesis of SBT Nano-Ceramics

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ABSTRACT

Non-stoichiometric strontium bismuth tantalate ($Sr_{0.8}Bi_{2.15}Ta_2O_9$/SBT) ferroelectric nano ceramic powder was obtained by using high energy ball milling process. Starting precursor powders were high energy ball milled in ethanol medium for 10 hours at a milling speed of 600rpm. For single phase formation, nano size powder of SBT was calcined at 700°C for 30 minutes and pellet sintered at 1000°C for 30 minutes using microwave processing technique. Microstructure of calcined SBT powder was studied using Field emission scanning electron microscope and it reveals the formation of nano sized particles. Fatigue free behavior makes this ceramic useful for non-volatile memory application.

INTRODUCTION

- **Nanocrystalline ferroelectric ceramics are important electronic materials that provide a wide range of scientific and industrial applications such as capacitors, non-volatile ferroelectric random access memories, etc.**
- **High Energy ball Milling is a technique used to synthesize ferroelectric materials in the form of nano powders.**
- **Microwave sintering process has proved to densify materials without prompting grain growth.**
- **SBT, a Bismuth Layered Structured Ferroelectrics (BLSF) have attracted a great interest because of its promising fatigue free nature for nonvolatile memory application.**

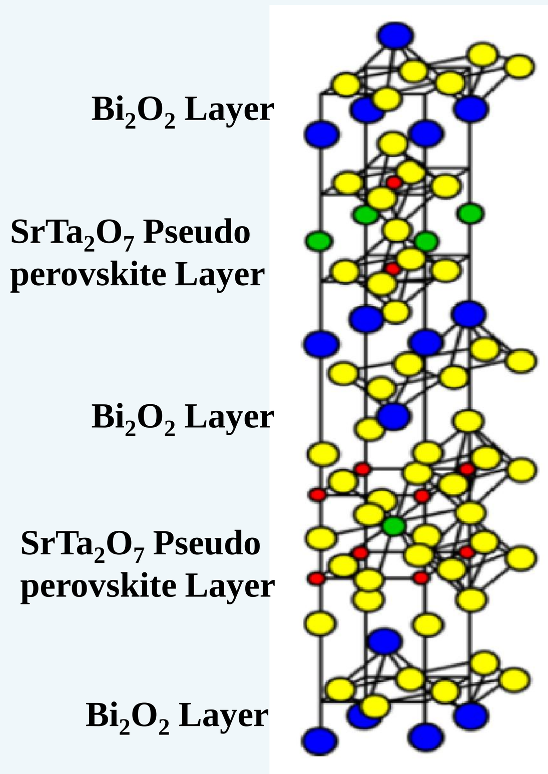

Figure1. Schematic Structure of SBT

EXPERIMENTAL PROCEDURE

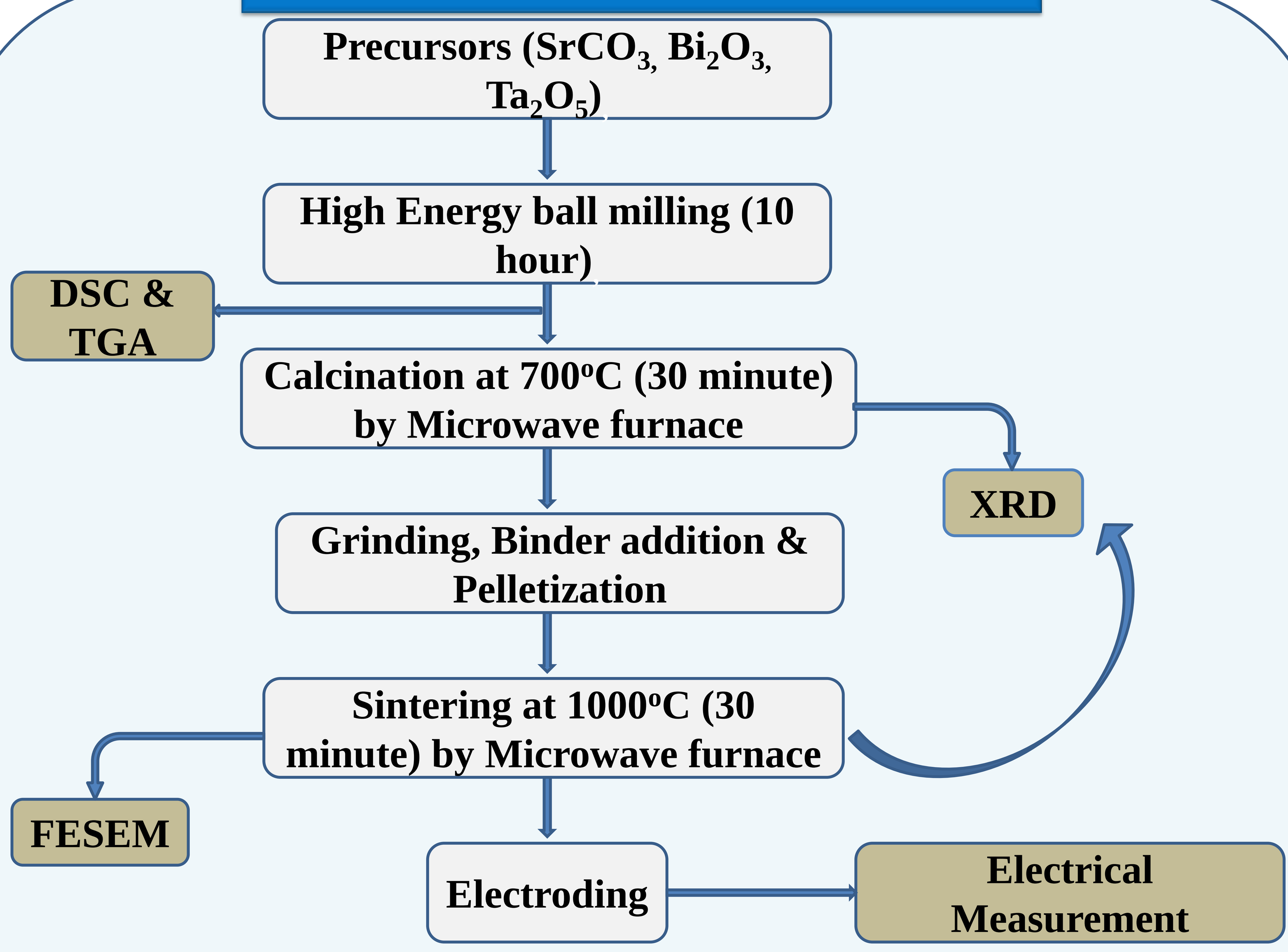

Figure2. Flow chart for Synthesis & Characterization of SBT ceramic

XRD STUDY

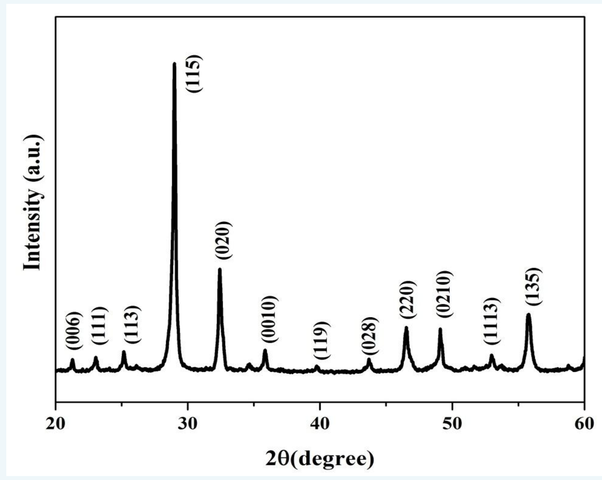

Figure5. Room temperature X-Ray diffraction pattern of SBT ceramic

- **SBT ceramic with single phase, consistent with JCPDS card No. 81-0557 having Orthorhombic Structure.**
- **Scherrer formula,**

$$D = \frac{k\lambda}{\beta cos\theta}$$

D- crystallite size, k- constant=0.94, λ- wavelength of X-Ray radiation (1.5406Å), β- FWHM (in radian), θ-diffraction angle (in radian)

Calculated D value ~45.48 nm

DIELECTRIC STUDY

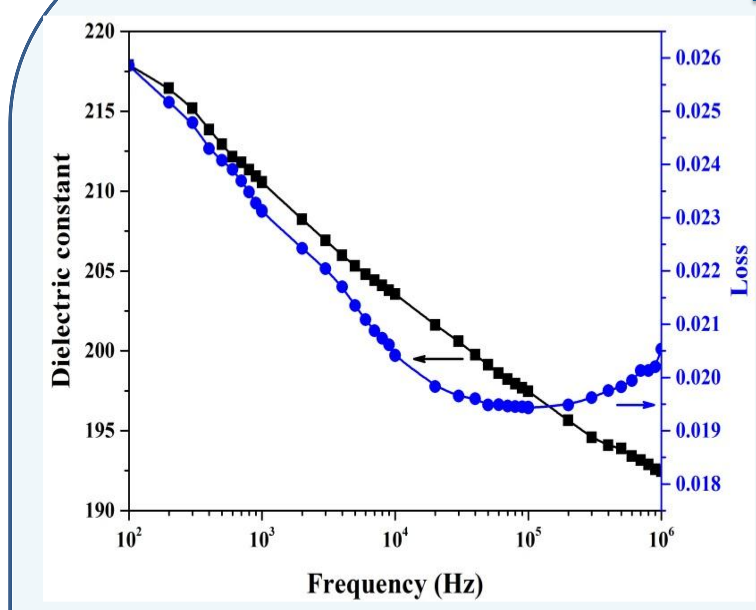

Figure6. Variation of Dielectric constant and loss (tan δ) with frequency at RT

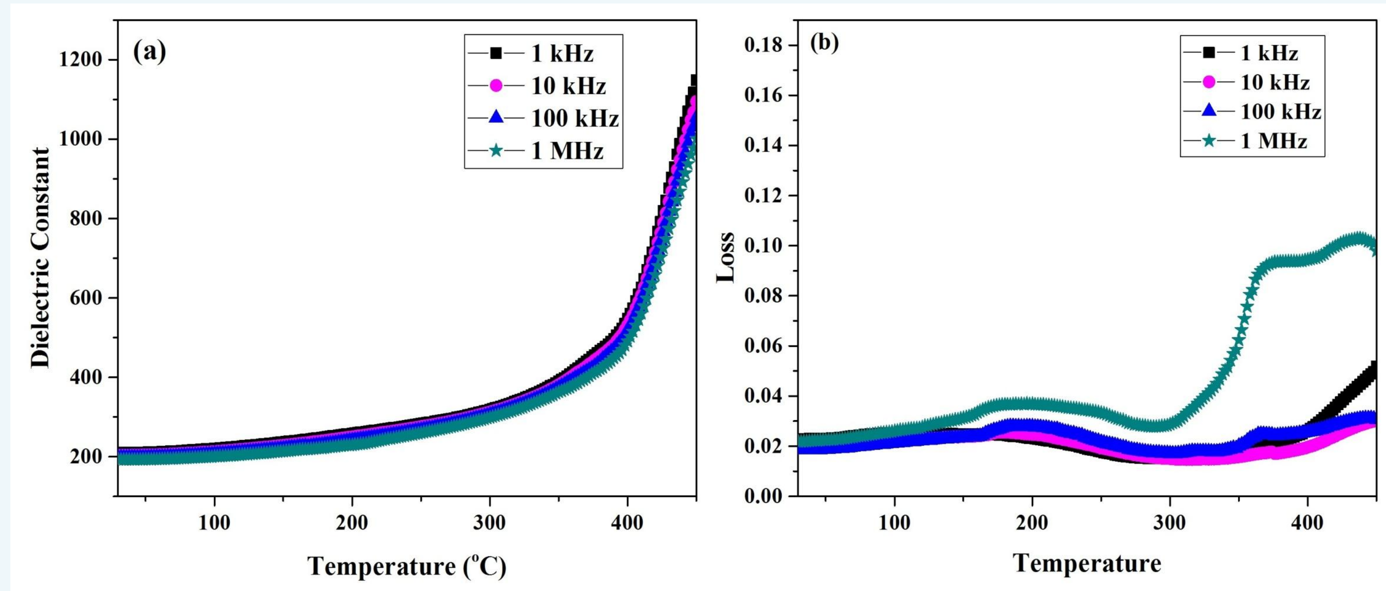

Figure7. Variation of (a) Dielectric constant and (b) Dielectric loss (tan δ) with temperature at different frequencies

Dielectric constant (ε_r) at RT (1kHz)	Dielectric loss (tanδ) at RT (1kHz)
210	0.02

- **Dielectric constant increases with increase in temperature.**
- **T_c shifts to higher temperature.**

FERROELECTRIC STUDY

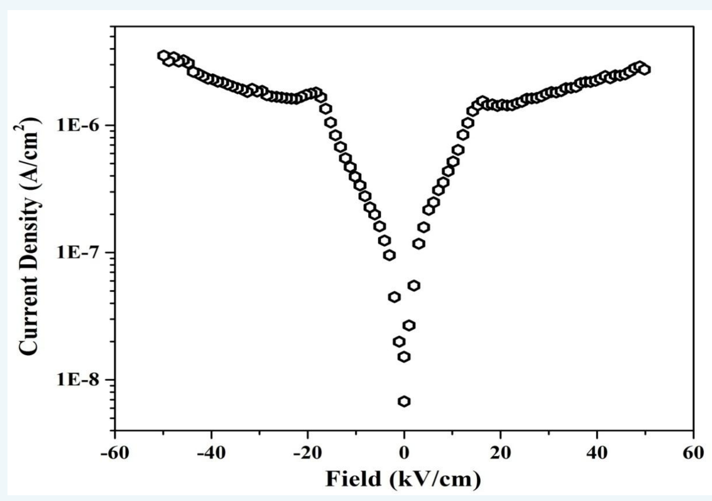

Figure8. Room temperature leakage current density vs. electric field plot of SBT ceramic

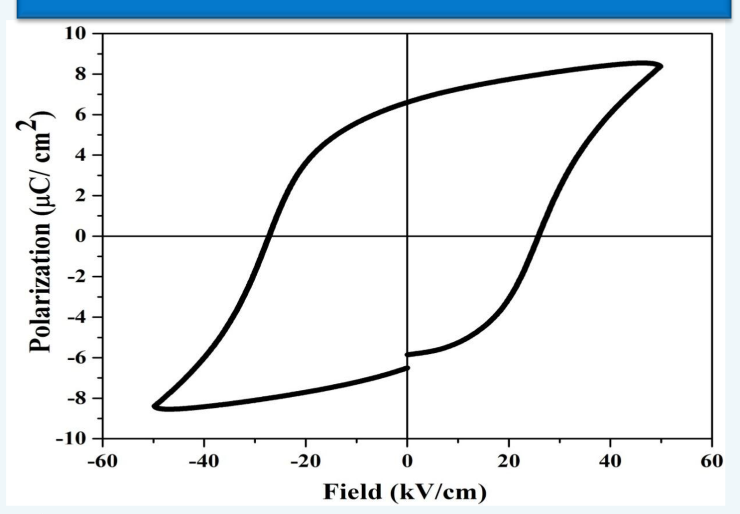

Figure9. Room temperature P-E hysteresis loop of SBT ceramic

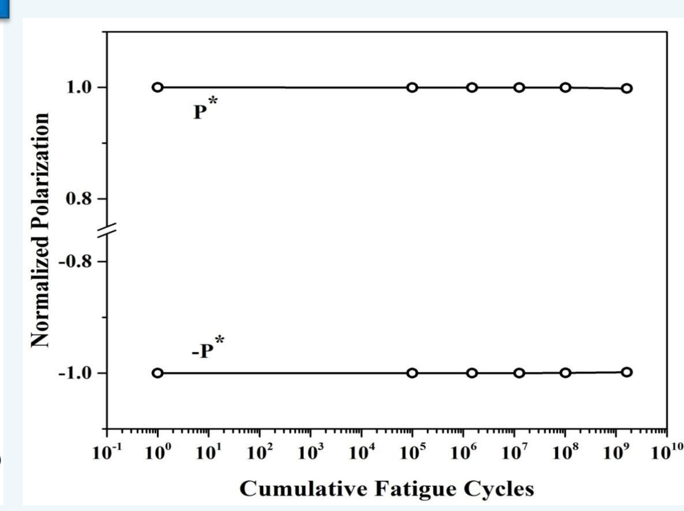

Figure10. Polarization fatigue as a function of switching cycles for SBT ceramic

Leakage current Density	Saturation Polarization (P_s)	Remnant Polarization (P_r)	Coercive Field (C)	Squareness Factor (R_{sq})	Relative Polarization Fatigue % (10^9 cycle)
~3.13×10^{-6} A/cm²	8.37μC/cm²	6.56μC/cm²	26.52kV/cm	~1.98	0.18

RESULTS & DISCUSSIONS

DSC/TGA AND MICROSTRUCTURE STUDY

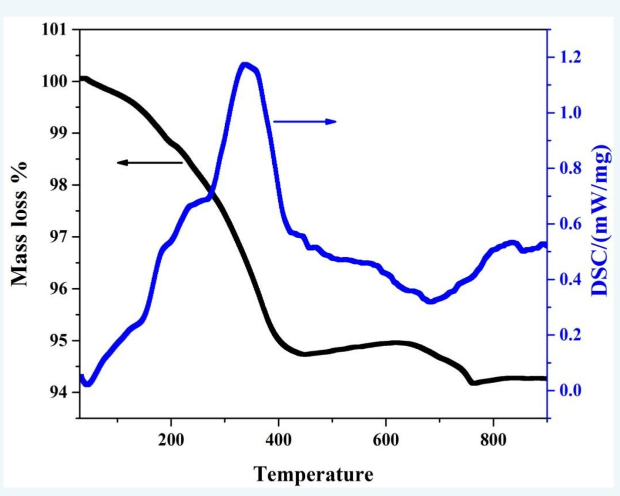

Figure3. Thermal analysis of raw SBT powder by DSC and TGA

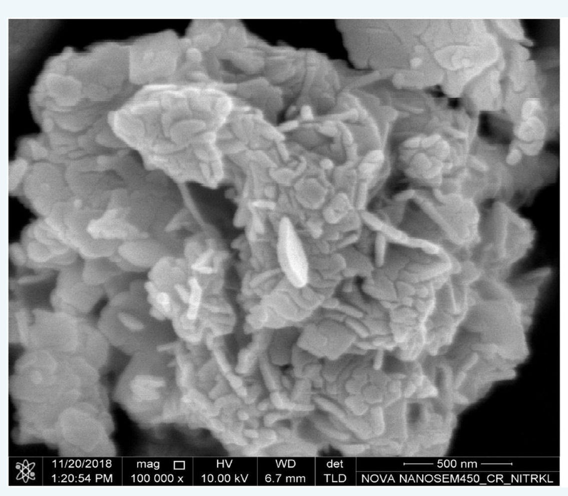

Figure4. FESEM micrograph of SBT powder

- **Particle size in the range of 60-170nm.**

CONCLUSIONS

- ✓ **Nanocrystalline single phase layered perovskite structured SBT successfully synthesized by microwave assisted high energy ball milling.**
- ✓ **Crystallite size is found to be ~45.48 nm and particle size is in the range of 60-170 nm.**
- ✓ **Excess Bismuth with cation vacancies at Sr-site tends to increase the T_c value.**
- ✓ **Better ferroelectric properties suggested that this excess bismuth SBT could be an alternative material for nonvolatile memory device application.**

References

1. P. Ganguly, A. K. Jha, Enhanced Characteristics of $Ba_5SmTi_3Nb_7O_{30}$ ferroelectric nanocrystalline ceramic prepared by mechanical activation process: A comparative study, Mater. Res. Bull. 46, 692-697 (2011).
2. I. Coondoo, N. Panwar, V.S. Puli, R.S. Katiyar, Ferroelectric and piezoelectric studies on Mo-substituted $SrBi_2Ta_2O_9$ ferroelectric ceramics, Integr. Ferroelectr. 124, 1-9 (2011).
3. S. Swain, P. Kumar, R. B. Choudhary, Electrical and ferroelectric studies of the 2-layered $SrBi_2Ta_2O_9$ based ceramics. Phys. B 477, 56–63 (2015).
4. Sugandha, A.K. Jha, Nanocrystalline non-stoichiometric SBT: Effect of milling duration on structural and electrical characteristics, Mater. Charact. 65, 126–132 (2012).
5. X. J. Lou, J. Wang, Bipolar and unipolar electrical fatigue in ferroelectric lead zirconate titanate thin films: an experimental comparison study, J. Appl. Phys. 108, 034104 (2010).