Phase transition and ferroelectric properties study of lead-free bulk $0.95BaTiO_3$-$0.05CaSnO_3$ ceramics synthesized by high energy ball milling method

B. Kar[1], S. Swain[1], P. Kumar[1*] and S. Panigrahi[1]

[1] Department of Physics and Astronomy, NIT Rourkela, Odisha, India, 769008

Email id : pawankumar@nitrkl.ac.in

ICPN 2021

Introduction

- Room temperature ferroelectric properties in Pb-free $BaTiO_3$-based ceramics have attained considerable attention as replacement of Pb-based PZT materials due to environmental issues.
- Substitution of Ca^{2+} at Ba^{2+} site and Sn^{4+} at Ti^{4+} site in pure $BaTiO_3$ develop a multiphase co-existence which improves piezoelectric property.
- Calcination and sintering temperature of $BTCS_{x=0.05}$ system prepared by high energy ball milling method is expected to be lower in compared to other solid state reaction route preparation.

Experimental Methods

Starting precursors (in powder form) + Zirconia balls

↓

High energy ball milling (300 rpm, 5 hrs.)

↓

Sample collected and grinded

↓

Calcination (1100°C/3hr) → XRD

↓

Pelletization, sintering (1250°C/3hr)

↓

SEM Study | Raman Study | Dielectric, PE Study

Results and Discussion

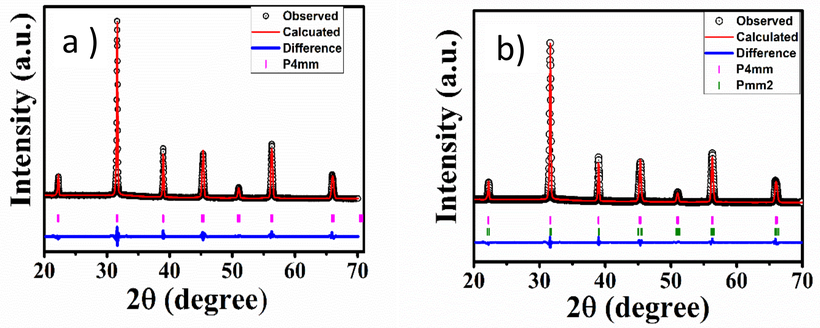

Figure 1. Reitveld refinement of $BTCS_{x=0.05}$ powder a) tetragonal phase b) tetragonal+orthorhombic phase

	a(Å)	b(Å)	c(Å)	α=β=γ	χ^2	R_p	R_{wp}
a)	3.99857	3.99857	4.01421	90	5.32	4.93	6.53
b)	3.99962	3.99962	4.01185	90	3.51	4.05	5.30
	3.98747	3.98517	4.02811				

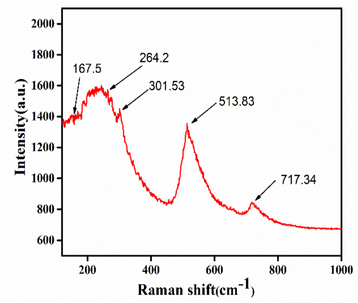

Figure 2. Raman spectrum at room temperature

- Some tetragonal peaks found to be diffused in compared to pure $BaTiO_3$ system

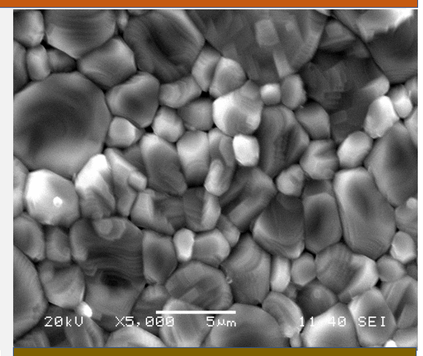

Figure 3. SEM image of 1250°C/3hr heated pellet

- Closely packed grains with average grain size of 2.31μm observed

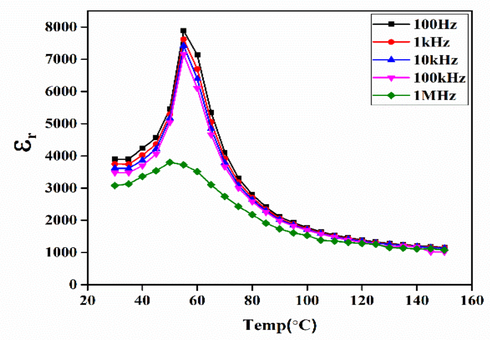

Figure 4. Variation of ε_r with temperature at different frequency

- Maximum ε_r of 7886.6 was observed at T_c (=55°C) at 100 Hz
- No relaxor behaviour was found

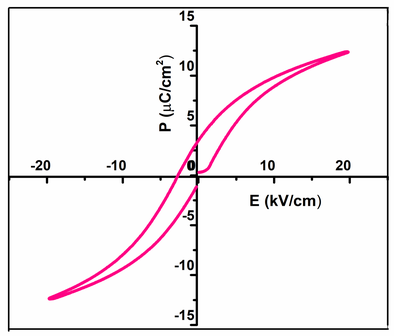

Figure 5. PE (hysteresis) loop

- Nearly saturated hysteresis loop at room temperature.
- P_s =12.394 μC/cm², P_r =2.1376 μC/cm², E_c =1.667 kV/cm

Conclusions

- Relatively low calcination and sintering temperature of $BTCS_{x=0.05}$ ceramics highlights the necessity of HEBM method.
- High values of ε_r, P_S and P_r makes $BTCS_{x=0.05}$ ceramic a good room temperature ferroelectric material.
- By changing Sn^{4+} percentage piezoelectric property can be improved.

References

1. Saito Y, Takao H, Tani T, Nonoyama T, Takatori K, Homma T, Nagaya T and Nakamura M 2004 *Nature* 432 84
2. Swain S, Kumar P and Sonia 2021 *Ceram. Int.* 47 26511-26518
3. Sahu R and Kumar P 2020 *Phase Transitions* 93 91–99