

Title FABRICATION AND CHARACTERIZATION OF BZT AND BNT-BASED CERAMICS PREPARED VIA THE COMBUSTION TECHNIQUE

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ABSTRACT

This work constitutes a major study of phase formation, microstructure and electrical properties of BZT and BNT-based ceramics which were synthesized through the combustion route. The experimental processes are focused on four parts. The study of the first part concerns the effects of firing temperatures and dwell time on phase formation, microstructure and dielectric properties of $\text{BaZr}_{0.10}\text{Ti}_{0.90}\text{O}_3$ or BZT10 ceramics prepared via the combustion technique. The topic of next study is the effects of zirconium content on phase formation, microstructure and electrical properties of $\text{BaZr}_x\text{Ti}_{1-x}\text{O}_3$ with $0.025 \leq x \leq 0.150$ ceramics which were synthesized via the combustion technique. In the third part, the study is focused on the effects of firing temperature on phase formation, microstructure and the dielectric properties of $\text{Bi}_{0.5}(\text{Na}_{0.74}\text{K}_{0.16}\text{Li}_{0.10})_{0.5}\text{TiO}_3$ or BNKL1610 ceramics synthesized via the combustion route. In the last part, the study focuses on the composition effects on phase formation, microstructure and dielectric properties of the $(1-x)\text{Bi}_{0.5}(\text{Na}_{0.74}\text{K}_{0.16}\text{Li}_{0.10})_{0.5}\text{TiO}_3 - x\text{BaZr}_{0.05}\text{Ti}_{0.95}\text{O}_3$ or BNKLT-100xBZT with $0.025 \leq x \leq 0.150$ ceramics prepared via the combustion technique.

The results of the first part indicated that the pure perovskite phase is found in powders calcined at 1000 °C for 5 h. The XRD patterns indicated that BZT has a

mixture of cubic and tetragonal phases and that the cubic phase content increased with increasing firing temperatures and dwell time. The microstructures of the BZT10 powders exhibited an irregular shape and an agglomerated form. The average particle size increased with the increase of calcination temperatures and dwell time. The average grain size, density and maximum dielectric constant increased with increasing sintering temperatures to a maximum at 1400 °C and thereafter it decrease.

In the next part, the XRD results suggested $\text{Ba}(\text{Zr}_x\text{Ti}_{1-x})\text{O}_3$ with $x = 0.025$ exhibits an orthorhombic structure at room temperature. The crystal structure was transformed to rhombohedral, tetragonal and cubic phase, respectively with increasing zirconium content. The effects of x on the dielectric properties have been studied intensively. It has been found that the phase formation, which was affected by zirconium substitution, strongly influences the dielectric behavior. The rhombohedral phase decreases the maximum dielectric constant while the tetragonal phase enhances it. An extrapolation studied revealed that the phase transition peaks merged into one peak at $x \sim 0.094$. The highest diffuseness constant of 1.95 was observed in BZT with 0.075 mol% zirconium. This was caused by the broadest dielectric peak of ferroelectric phase transition and the imminent diffusion between ferroelectric phase transition peak and Curie phase transition peak. The ferroelectric properties were sensitive to the phase exhibited in BZT system.

The results of the third part demonstrated that firing temperature highly influences the secondary phase formed on BNKLT1610 powders and ceramics. The vaporization of raw materials and products caused the formation of a secondary in calcined powders and sintered ceramics, respectively. The XRD patterns suggested the BNKLT1610 ceramics show the coexistence of rhombohedral and tetragonal phases and the relative amount of each phase correlates with the sintering temperature. The dielectric peak at depolarization temperature (T_d) was exhibited in all samples while the dielectric peak at maximum dielectric temperature (T_m) was observed only the samples sintered at high temperatures. The dielectric results persuade core-shell structure is formed in prepared samples. Increasing sintering temperature caused the grain core to enlarger and the grain shell to become thinner. The dielectric constants are highly affected by secondary phase formation and densification. The highest value

ϵ_r of 1,210 was obtained from the sample sintered at 1,000 °C, which is significantly higher than BNKLT1610 prepared via the solid-state reaction method.

The results of the last part showed how BZT content affects typical properties of a BNKLT-100xBZT system such as phase formation and dielectric properties. The XRD investigation revealed that BNKLT-100xBZT exhibits the coexistence of rhombohedral and tetragonal phases. By BZT addition, the rhombohedral and tetragonal were decreased and increased, respectively. From a dielectric properties investigation, BNKLT-100xBZT showed two dielectric loss peaks at ~ 190 °C and ~ 320 °C and were defined as T_d and T_m , respectively. Increasing BZT fraction caused T_m to shift to a lower temperature while insignificantly shifting T_d . The maximum ϵ_r and ϵ_{\max} with the values of 1,380 and 4,050 were observed from the sample with composition of BNKLT-5BZT. This can be attributed to this composition being located near the MPB region.

