CHAPTER VII

SUMMARY

First system, lead free BNKFT ceramics were synthesized by the combustion technique. The optimal calcination andsintering conditions were found to be 750 °C for 2 h and 1050 °C for 2 h, respectively. The calcination and sintering temperatures directly affected the phase formation, microstructure, density and electrical properties. The highest density (ρ = 5.85 g/cm^3), superior dielectric properties at T_c (ε_r =7,846 and tanδ = 0.02) and piezoelectric constant (d_{33} =213 pC/N) were obtained from the sample sintered at 1050 °C. With superior electric properties, this work indicates that the BNKFT ceramics prepared by the combustion technique are better than the BNKFT produced by solid stated method.

The content of x and y have a strong influence on the crystal structure, microstructure, lattice parameter and percentage of the perovskite phase of the calcined powders. The lattice parameter of a increased with increasing x and y content. The ceramics possessa pure single phase of perovskite structure, indicating that K^+ and Fe^+ have diffused into the lattice. The SEM images indicated that with increasing x content, particle size decreases. But with increasing y content, the variation of the particle size is opposite. BNKFT powders can be successfully synthesized by the combustion technique at a lower calcination temperature than those prepared by the solid-state reaction method.

The variation of x and y content affects directly crystal structure, microstructure, density, dielectric, ferroelectric and piezoelectric properties of the BNKFT-x/0.03 and BNKFT-0.18/y ceramics. The XRD indicated that the ceramics possess pure single phase of perovskite structure, indicating that K^+ and Fe^+ have diffused into the lattice. With increases x and y contents, grain size decreases and increase, respectively. The optimum electric properties can be obtained at x=0.18 and y =0.03, as follows: ρ = 5.85 g/cm^{-3}, ε_r = 7,850, tanδ = 0.02, P_r=20.1 μC/cm^2 (measured at 40 kV/cm) and d_{33} = 213 pC/N. With superior electric properties,

this work indicates that the BNKFT-x/y ceramics prepared by combustion technique are better than the BNKFT-x/y ceramics prepared by solid state.

Second system, BNKLT ceramics were prepared by the combustion technique with optimum calcination and sintering of 750 and 1025 for 2 h. The firing temperatures have direct effect on phase formation, grain size, densification microstructure and dielectric properties of ceramic samples. The structure exhibited the coexistence of rhombohedral and tetragonal phases, which is consistent with the nature of the specimen with an MPB composition. The optimum sintering temperature significantly promoted the grain growth and microstructure densification. The highest dielectric at T_c (ε_r = 4,344) and density (ρ = 5.79 g/cm^{-3}) were obtained from the sample sintered at 1025 °C. These results were higher than the sample obtained by solid state reaction method and confirmed that the combustion technique has potential to fabrication of high purity BNKLT ceramics using lower firing temperature.

The addition of BKT (x) and BLT (y) directly affect the crystal structure, microstructure, density and electrical properties. The crystal structure shows the coexistence of rhombohedral and tetragonal phases at the MPB composition BNKLT-0.20/0.03. The SEM result indicated with increasing x content, the grain size decrease. But with increasing y content, the variation of the grain size is opposite. The dielectric spectra show two distinct phase transitions approximately 110 °C and 310 °C. All the samples show relaxor behavior which is linked to the presence of disorder on the A-site. The coexistence of a polar and non-polar phase at the MPB was indicated by a pinched P-E loop and a large strain of ~0.36% at the composition BNKLT-0.20/0.03 with S_{max}/E_{max}~729 pm/V. These large strains in combustion-prepared samples represent a significant improvement on solid-state prepared samples and shows great promise for actuator application.

Third system, BNNKT powders and ceramics were successfully prepared by combustion technique with calcination and sintering of 750 °C and 1025 °C for 2 h. The calcination and sintering parameters have direct effect on phase formation, grain size, densification microstructure and dielectric properties of ceramics samples. The BNNKT powder showed the rhombohedral structure. The BNNKT sintered

ceramics indicated the coexistence of rhombohedral and tetragonal phases, which is consistent with the nature of the specimen with an MPB composition. The result shows that the increase of sintering temperature helped the growth of grain size. The density and shrinkage increased with increasing sintering temperatures from 900°C to 1025°C, and decreased after further sintering at a higher temperature (1050°C).BNNKT powders and ceramics can be successfully synthesized by the combustion technique at a lower calcination and sintering temperatures than those prepared by the solid-state reaction method.

The variations of x and y contents directly affect the crystal structure, microstructure, density and electrical properties on BNNKT-x/y ceramics. The structure identified the phase transition from rhombohedral and pseudo-cubic symmetry. The addition of KNN content decreased rhombohedral phase and the structure became more pseudo-cubic. With increase of x and y content, the average grain size decreases. The ε_r show pronounced dependence on frequency. With increased frequency, ε_r for all ceramics decrease. The BNNKT-x/y solid solutions exhibited diffuse phase transition behavior. The change in P-E loops indicated that the long-range ferroelectric order of the sample was disturbed and turned to the PNRs with increase of x and y contents. The polarization hysteresis loop transformed from well saturated typical ferroelectric to pinched and then to relaxor state with increase in x and y contents. The largest S_{max} of 0.25% corresponding to a high-field effective d_{33}* of 509 pm/V was found in the composition with y=0.01.