CHAPTER 8

SUMMARY AND CONCLUSIONS

7.1 GENERAL

The thesis work mainly deals with the investigation of structural, morphological and dielectric properties of solid solution of Sn doped $BaTiO_3$ and their composites with nickel ferrite, CNTs and Fly-ash. For the present study, all samples were prepared in single phase by solid state technique. Different experiments were carried out and results were carefully analyzed to monitor the changes in the structure and dielectric properties of Sn doped $BaTiO_3$ and their composites. This chapter presents a concise summary based on the results and discussions presented in the previous chapters. It also provides a brief note on the scope for the future work.

1. Synthesis of a few compositions of $BaTi_{1-x}Sn_xO_3$ (x=0.0, 0.05, 0.15, 0.30, 0.40 and 0.50) using $BaCO_3$, TiO_2 and SnO_2 via conventional solid state ceramic route has been described. Mechanism of the formation of solid solution $BaTi_{1-x}Sn_xO_3$ has been studied in detail using thermal analysis (TGA/DSC) and X-ray diffraction techniques. The X-ray diffraction studies of calcined powders confirmed that the formation of single phase solid solution $BaTi_{1-x}Sn_xO_3$ proceeds through the formation of $BaTiO_3$ and $BaSnO_3$ phases and reaction between them at temperatures > 1100 ^{o}C. A change in the crystal structure from tetragonal to cubic is observed with increasing concentration of dopant, Sn. Dissolution of Sn ion in the lattice of $BaTiO_3$ has further confirmed by Fourier transform infrared spectroscopy (FTIR) technique. Effect of Sn doping on microstructure and dielectric properties has been studied in detail. In this chapter, we have compared our results with the previous results reported in the literature.

2. Synthesis of a few compositions of $BaTi_{1-x}Sn_xO_3$ (x=0.0,0.10,0.20,0.30,0.40 and 0.50) using $Ba(NO_3)_2$, TiO_2 and $SnCl_45H_2O$ via solid state ceramic route has been described. Mechanism of the formation of solid solution $BaTi_{1-x}Sn_xO_3$ has been studied in detail using thermal analysis (TGA/DSC) and X-ray diffraction techniques. X-ray diffraction studies demonstrated that the single phase powders of nano-sized (40-50 nm) could be obtained by calcination at temperature 800^{o}C which is approximately 400^{o}C lower than temperature requires for conventional solid-state ceramic route. Therefore, we can conclude that replacement of $BaCO_3$ by $Ba(NO_2)_3$ as a raw material for the mass production of nanopowders of BTS for industrial applications is a cost efficient and straight forward method as compared to other methods reported in the literature. The properties measured of samples synthesized using $Ba(NO_3)_2$ has been compared with properties of same compositions synthesized by conventional ceramic route mentioned in chapter 3. Moreover, we have compared properties reported in the literature on $BaTi_{1-x}Sn_xO_3$ system synthesized by different methods.

3. Synthesis of $NiFe_2O_4$ by the gel-combustion method has been described. Further, synthesis of composites of optimum composition $BaTi_{0.85}Sn_{0.15}O_3$ and $NiFe_2O_4$is discussed. Four composites of (1-x) $BaTi_{0.85}Sn_{0.15}O_3$ - x$NiFe_2O_4$ (x=5, 10, 15 and 20 weight %) have been synthesized, and their properties such as crystal structure, microstructure, dielectric, electrical and magnetic are discussed. Properties of composites are compared with properties of constituent phases $BaTi_{0.85}Sn_{0.15}O_3$ and $NiFe_2O_4$. The formation of the composites and their constituents was confirmed by X-ray diffraction, FTIR and SEM analyses. Conduction in the composites is governed by hopping of electrons among the different ions of the elements Sn, Ti, Ni and Fe present in the samples. Variation of dielectric constant with respect to frequency shows a dispersive behaviour due to Maxwell-Wagner effect. The interfacial polarization has a significant contribution for composites as compared to their end members.

A relaxation phenomenon in the high-frequency range was observed in the composites which are intrinsic properties of the composites. Measurement of magnetic properties indicated that composites are also magnetically ordered systems with hysteresis loop in their M-H plot at room temperature. The value of saturation magnetization (M_s) increases linearly with increasing concentration of ferrite phase in the composite. However, the coercive field (H_c) and remnant magnetization (M_r) for all the composite samples is larger than that of pure $NiFe_2O_4$ phase. The value of saturation magnetization and coercive field indicates that composites are soft magnetic materials. The composite may be used in transformer, inductor cores, recording heads, microwave devices and magnetic shielding.

4. Four composites of different concentration of BTS and CNT; (1-x) $BaTi_{0.85}Sn_{0.15}O_3$ - x CNT (x=5, 10, 15 and 20 weight %) have been synthesized by solid state ceramic route. X-ray diffraction analysis confirmed the presence of both the constituents in the composites and ruled out the reaction between BTS and CNT even at sintering temperature 1350^{o}C. Micro structural characterization using SEM indicated that CNT is distributed uniformly in the composites. Average grain size is small while density is higher of the composites as compared to BTS. The thickness of the CNT in the composites is approximately 100 times more than the thickness of CNT used in the preparation of the composites. EDXA analysis confirmed in the composites a thick layer of BTS had been deposited on the CNT during synthesis and sintering. Dielectric constant is smaller while dissipation factor is larger for the composites as compared to BTS. The electrical conductivity of the composites systematically increases with increasing concentration of CNT in the composites.

5. In this study we demonstrated that the fly-ash generally is the wastes which were used to enhance the properties of the barium titanate stannate. The analysis of composites was studied through TG-DSC and for preliminary study on the phase formation and determination of their crystal structure; the powder x-ray diffraction technique was adopted. The thermal

analysis of the mixture of the composites shows the thermal stability of the samples with in the investigated range of temperature, while the XRD pattern shows no reaction among the two phases. The morphology of composite materials was studied by scanning electron microscopy (SEM) and analysis of grain size of composite (BTS-FA) carried out through "**Image J**" software which shows smaller grain size than barium titanate stannate (BTS). The dielectric constant and dissipation factor of composites was low as compared to BTS which reflects the insulating behaviour of fly ash. The activation energy also decreases with increasing the fly ash content in barium titanate stannate samples, which increases the locally migration of electron in between the respective sites of multivalent ion Sn/Ti and Fe. Composites exhibit small value of dielectric constant and dissipation factor at higher frequency hence, they are more suitable for thermally stable capacitor fabrication as compared to BTS.

7.2 FUTURE SCOPE OF STUDY

- Study of oxidation state of various ions using XPS will be useful in understanding the defect structure and resulting dielectric properties.
- Study of electrical properties of composites.
- To understand the hopping mechanism in composites materials.