CHAPTER 6

Conclusions and Suggestions for Further Work

The present work deals with the preparation and characterization of lead magnesium niobate (PMN)-lead titanate (PT) both in powder and ceramic forms. Accordingly, the research contribution of this thesis has been made under three major aspects, i.e. preparation and characterization of (i) PMN (ii) PT and (iii) PMN-PT.

6.1 Conclusions

6.1.1 Preparation and characterization of PMN

(a) A mixed oxide synthetic route to single-phase PMN powder has been developed via a Columbite method. The optimal calcination conditions for single phase PMN is 800 °C for 1 h with heating/cooling rates of 20 °C/min.

(b) Single phase of perovskite PMN ceramics of high density (the relative density of ~ 96%) with the average grain size ~ 4.56 μm may be produced by employing straightforward sintering condition at 1240 °C for 2 h with heating/cooling rates of 5 °C/min. It is found that slow heating/cooling rates of 1 °C/min can provide enough time for occurring of lead volatilization and result in the formation of pyrochlore $Pb_3Nb_4O_{13}$.

6.1.2 Preparation and characterization of PT

(a) A new process has been developed for the preparation of PT powders with 100% perovskite phase by employing a combination of simple mixed oxide synthetic route and a rapid virbro-milling technique.

(b) Even though the simple mixed-oxide method was used, the highly dense and stable PT ceramics were successfully formed by the careful control of processing parameters. It is found that the slow heating/cooling rates are important parameters in preparing dense PT ceramics. The well defined-grain ceramics with the average grain size of about 0.6 μm could be prepared by heating them to 1225 °C using the considerably slow heating/cooling rates of 1 °C/min and dwell time of 2 h. Moreover, it was also found that the relative densities of these ceramics would be as high as 97%.

6.1.3 Preparation and characterization of PMN-PT

(a) A simple stage mixed oxide process has been developed to synthesize (1-x)PMN-xPT powders ($0.1 \leq x \leq 0.9$; $\Delta x = 0.1$). A series of continuous solid solution of perovskite structure, whose degree of tetragonality increased, while optimum firing temperatures decreased continuously with increasing PT content, were found.

(b) Only ceramic compositions in the (1-x)PMN-xPT ($0.1 \leq x \leq 0.5$; $\Delta x = 0.1$) have been successfully fabricated from the calcined (1-x)PMN-xPT powders, employing a normal sintering method. The optimum sintering conditions of 1240 °C for 2 h with 15 °C/min, were observed in samples at compositions of x between 0.1 to 0.3 whilst for compositions with PT content of $x = 0.4$ and 0.5 optimum condition was found at 1220 °C for 2 h with 15 °C/min. It is seen that the tetragonality factor (c/a) and the degree of angularity for the grain shape increase with PT content.

6.2 Suggestions for further work

There are a number of issue arising from the results of this study. It is believed that this could be partly filled by the following suggestions for further work.

(1) In general, some improvement may be achieved by increasing the density of the materials in this study by using techniques such as sintering in oxygen or

hot iso-static pressing (HIP). However, the costs of sophisticated processing and manufacturing would need to be considered.

(2) Further work on the electrical characterization especially the dielectric measurement of the materials in this study would facilitate a deeper understanding of perovskite ferroelectrics in general.

(3) The further investigation should be carried out in order to find out exactly what inhibits grain growth in these PT ceramics while they are subjected to a long heating schedule of slow heating/cooling rates.

(4) Further development work arising directly from this study would focus attention on the compositions within the MPB region of the (1-x)PMN-xPT system where $0.3 < x < 0.4$. Careful investigations of the effects of sintering condition on the phase formation, densification, microstructure and dielectric properties would provide knowledge and baseline for further academic work.