CHAPTER 5

CONCLUSIONS AND FUTURE WORK

Sol-gel process which is a method of preparing inorganic materials via chemical routes was used to fabricate PZT ferroelectric thin films and composite materials. Sol-gel PZT thin films with a composition at the morphotropic phase boundary (52/48) prepared using metal alkoxides and 2-methoxyethanol as a solvent have been fabricated and characterized in this study. Processing parameters, the amount of excess Pb and annealing conditions, which enhanced perovskite formation and optimized electrical properties, were studied. In addition, sol-gel based composite thick film prepared by using sol-gel PZT solution and PZT powders have been fabricated. The following observation were made as the followings:

1) Sol-gel PZT thin films with a Zr/Ti ratio of 52/48 were successfully fabricated by spin-coating solution of polymeric complex Pb,Zr,Ti-methoxyethoxide onto Pt-coated Si substrates. Upon deposition, films were pyrolyzed at 300°C and further annealed to promote crystallization and densification. In this study, it can be achieved by conventional furnace annealing at heating rate of 5°C/minute. PZT films with good electrical properties were successfully fabricated by the addition of 10 mole% excess Pb and annealing at 700°C for 30 minutes.

2) The intermediate-perovskite phase transformation in PZT films was investigated by using XRD. The as-deposited film after pyrolysis at 300°C on the hot plate was still in amorphous state. During heat treatment, an

intermediate or pyrochlore-like structure formed and then transformed to perovskite phase at higher temperature. In order to obtain pure-phase perovskite, 10 mole% of excess Pb was incorporated in stock solution. The observation confirmed that an addition of 10 mole% excess Pb was necessary to promote the transformation.

3) The microstructure evolution was examined using FE-SEM. SEM micrograph showed nearly a single perovskite phase. The results were in good agreement with XRD data. In addition, the uniform and dense microstructure were obtained. The cluster of sol-gel PZT thin films was approximately 1 μm.

4. The reasonable values for electrical properties were obtained in this study. The remanent polarization was 19.2 $\mu C/cm^2$ for an applied voltage of 7 volts. The coercive field was 46.4 kV/cm for films thickness ~ 3000 Å. The dielectric constant was measured to be 940. This results indicated that controlling the microstructure evolution and the phase development by exploration of annealing conditions and the amount of excess Pb were very important for the optimization of electrical properties.

5. Paste composition of thick film can be fabricated by dispersing 75%by weight of PZT powders in 0.88M PZT sol-gel solution. This result indicated that the rheology of paste was pseudoplastic. Film viscosity showed no signs of any difficulty to be able to be screen-printed.

FUTURE WORK

This work presents numurous opportunities for further research. Some suggestions are listed below.

1) The effects of alternative PZT composition: The role of different Zr/Ti ratio in controlling crystallization and microstructure should be studied. Moreover, the role of PbO overcoated and PbO atmosphere powders which can compensate lead-loss during heat treatment and lead to the crystallization of phase-pure perovskite PZT films at lower annealing temperature should be further examined.

2) The orientation of PZT thin films: The electrical properties of PZT films seemed to be sensitive to the crystallographic orientation of the films. Therefore, the factors that influence the orientation of PZT films should be studied, including types of substrates, seeding layer and pyrolysis condition.

3) Rapid thermal annealing (RTA) technique: This novel technique can reduce the processing time to only few seconds. An advantage of the short rise time may be the reduction in surface damage and minimization of the film-substrate interaction even at annealing temperature above 700°C.

4) Development of composite materials: ferroelectric composite materials for incorporating macroscopic device based on piezoelectric applications such as ultrasonic high frequency transducers and fiber optic modulator should be considered. These potential applications which require film thickness of 1-30 μm and geometries of μm^2 to cm^2 still exist. The use of

binder/plasticizer systems, special drying and firing techniques should be investigated.