SYNOPSIS

With the ever-increasing research interest in MEMS technology, the functional materials such as ferroelectric materials have received utmost attention in the recent past years. These ferroelectric materials are known for their excellent dielectric and piezoelectric properties which make them potential candidates for technological applications [4,225,226]. Lead based ferroelectric ceramics such as lead zirconate titanate (PZT) are the most exploited materials used for device applications such as sensors and actuators. The $Pb(Zr_xTi_{1-x})O_3$ system and its solid solutions exhibit exceptional high dielectric, elastic and piezoelectric properties at the 'Morphotropic Phase Boundary (MPB)' [18,107-109,190]. This MPB is a result of coexistence region of two phases viz tetragonal and monoclinic phases [19,22]. In this thesis, an effort is made to synthesize PZT ceramics by adopting low temperature calcination route.

The processing of these ceramics requires sintering temperature above 1280°C which restricts their use in practical device applications due to compositional fluctuations as a result of PbO loss during synthesis. The PbO loss not only leads to the deterioration in the dielectric, piezoelectric and electromechanical properties but is also responsible for the environmental pollution. Further, such a high firing temperature requires the expensive platinum and palladium materials for electroding purpose of these lead based perovskite devices [110, 111]. Hence, it is desired to reduce the sintering temperature for the reliable and reproducible material properties of these ceramics. The reduction in the sintering temperature also allows the utilization of less expensive electrodes such as silver for applications such as multilayer capacitors. The low sintering temperature in these ceramics can be achieved by the use of ultra fine powder [141], liquid phase sintering [142], use of additives [143], hot pressing [144] which promotes solid state sintering. In this thesis, therefore, an effort is made to lower the sintering temperature of PZT ceramics synthesized in MPB region by using Li_2CO_3 as a sintering aid.

Although these piezoceramics exhibit excellent dielectric and piezoelectric properties but they are brittle in nature and are difficult to conform to the complex shapes and structures. These limitations restrict their use in technological applications where flexibility is desired. These limitations have led to the replacement of ceramics by piezoelectric polymers known for their light weight, flexibility, fracture tolerance, ease of fabrication into desired shapes and structures [156]. Polyvinylidene fluoride (PVDF) is the most widely exploited among all the active polymers for their superior dielectric and piezoelectric properties. PVDF is a semicrystalline polymer known to exist in various crystalline forms mainly α, β and γ phases [47,162,163,168]. However, the β phase is desired in PVDF for its better ferroelectric characteristics [114-117]. Among the crystalline forms, α phase is predominant [227]. It is reported that the β phase in PVDF can be obtained from the modification of α phase by various processing conditions such as mechanical deformation [119,171,181], the application of high electric field [118], crystallization under the influence of high pressure [120,121], cooling at high rates etc. [122]. The primary intention of the present study is to optimize the thermal processing conditions in order to maximize the β phase in PVDF based films, which essentially govern the dielectric and piezoelectric properties in PVDF based films.

Although the polymer based piezoelectric materials such as PVDF has high mechanical strength, low density and high compliances, their piezoelectric and pyroelectric activities are much lower compared to the piezoceramics [57,165]. Hence, the increased interest in the search of new materials which can combine the complementary features of polymer and ceramic has led significant research in the direction of composites [188]. Piezoelectric ceramic-polymer composites constitute a new class of functional materials with combined hardness, stiffness and piezoelectricity of ceramics and the flexibility, elasticity and mechanical strength of polymers. The inter-connectivity among the different phases plays a key role in the development of composites for technological applications [188]. The material properties such as dielectric, piezoelectric, mechanical properties of a composite are governed by the composition, the connectivity, shape, volume fraction and properties of the constituent phases. For two phase composite systems, ten possible phase connectivities exist [74, 189]. Among these possible connectivities for the two phase composites, 0-3 and 1-3 piezocomposites are the most commonly studied and used for practical applications [78, 81]. Hence, in this thesis a systematic study is carried out for 0-3 and 1-3 PZT/PVDF composites. Broadly, the thesis can be divided into two parts. The first part deals with the optimization of synthesis conditions for 0-3 PZT/PVDF composites to obtain the superior dielectric and piezoelectric properties, while the second part is based on the derivation of simple analytical model of 1-3 PZT/PVDF composites which is later utilized for the optimization of device parameters of actuators used in biomedical surgical applications. In the first part of the thesis, the optimized processing conditions of PZT and PVDF materials are employed to synthesize 0-3 PZT/PVDF composites by solution cast technique. The material properties of these composites are studied with the volume content of PZT ceramics in the PVDF matrix. In the later part of the thesis, the analytical relations of material properties such as elastic and piezoelectric properties of 1-3 PZT/PVDF composites are obtained. These relations are used for achieving optimum volume content of PZT ceramics in the polymer PVDF matrix for surgical applications.

This thesis has been written in nine chapters. The introduction (Chapter 1) and procedural (Chapter 2) builds up the literature background required for this work. The results of the present study are presented in Chapters 3 to 8. Finally the significant findings of the study are presented in Chapter 9.

Chapter 1 introduces the basic principle governing the piezoelectricity phenomena and reviews the materials for device applications such as sensors and actuators. Further, the chapter also describes the criteria for material selection and highlights the objective and the scope of the present work.

In Chapter 2, the basic and intricate experimental techniques and the principles involved in the synthesis, processing and characterization of materials are discussed. The PZT ceramics are synthesized by adopting two-steps solid state reaction method while the PVDF and composite films are synthesized by solution cast technique. The phase analyses and structural characterizations are done with x-ray diffraction and Fourier Transforms Infra Red (FTIR) spectroscopy. The electrical properties such as dielectric and piezoelectric properties are studied by impedance analyzer and piezometer respectively. In the later part of the chapter, the constitutive equations required for the analytical modelling of piezoelectric PZT/PVDF are introduced. Further, the methodology adopted for the finite element modelling of these piezoelectric composites by COMSOL Multiphysics software is briefly described.

Chapter 3 deals with the systematic study of the effect of Li_2CO_3 addition on the structural, dielectric and piezoelectric properties of the PZT (Zr/Ti = 50/50) ceramics in the morphotropic phase boundary (MPB) region. The addition of Li_2CO_3 in the PZT system resulted in the improved sinterability and densification of the ceramics. 0.2 wt% Li_2CO_3 addition is found to be optimum for obtaining better dielectric and piezoelectric properties in the PZT ceramics. It is further seen that the addition of Li_2CO_3 shifted the MPB of the ceramics towards the tetragonal phase. The Zr/Ti ratio was hence, varied to regain the MPB in the PZT ceramics with an intention to obtain superior dielectric and piezoelectric properties.

(***The results of this work are published in (i) Ceramic International, vol. 41, pp. 2774-8, 2015 (Thomson Reuters I.F.= 2.605, h-index = 56, h5-index = 37, Published by Elsevier, Indexed in SCI and SCOPUS))***

Chapter 4 investigates the effect of annealing and quenching temperatures on the crystallinity, β phase fraction and dielectric behaviour of poly (vinylidene fluoride) (PVDF). The crystallinity and β phase fraction of these films are evaluated using X-ray diffraction and FTIR techniques for different annealing and quenching temperatures. It is seen that the thermal processing conditions play a crucial role in determining the dominant phase in PVDF. The β phase PVDF is the most desired phase for device applications such as sensors and actuators. Hence, the thermal processing conditions are optimized for obtaining β rich PVDF films. The β rich phase of PVDF is obtained for films which are annealed at 80°C and quenched at 20°C. The as-synthesized films for the optimized processing conditions is studied for their dielectric behavior and is found to exhibit dielectric constant as high as ~60.

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In Chapter 5, the piezoelectric composite films of 0-3 ($[PZT]_x/[PVDF]_{1-x}$), with different volume fractions of PZT ceramics in the MPB, prepared using a solution cast technique under optimized processing conditions are studied. It is seen that the increase in the content of PZT ceramic in the PVDF matrix not only increases the dielectric constant of the composites but also enhances the β phase in the PVDF polymer, thereby resulting in the overall improved dielectric and piezoelectric behavior of the system. The as-synthesized films exhibited superior dielectric and piezoelectric properties than reported in the literature.

(***The results of this work are published in (i) Ceramics International, vol. 41, pp. 8008-8013, 2015 (Thomson Reuters I.F.= 2.605, h-index = 56, h5-index = 37, Published by Elsevier, Indexed in SCI and SCOPUS))***

In Chapter 6, flexible piezoelectric composite films of ($[PZT/]_x/[PVDF]_{1-x}$ (x = 0, 0.1, 0.2, 0.3) with 0-3 connectivity synthesized from 0.2 wt% Li_2CO_3 added PZT ceramic powders in the MPB and PVDF polymer by solution cast technique under optimized thermal processing

conditions are studied. It is seen that crystallinity and β phase are enhanced with the addition of Li in the composite films and as well as with the volume content of PZT ceramics in PVDF matrix, thereby resulting in superior dielectric and piezoelectric properties. It is also seen that reasonably high dielectric and piezoelectric properties are obtained for 30% of the volume content of PZT ceramics as compared to 50% of the volume content of ceramic in the polymer matrix as reported in literature and hence, the flexibility is better in these films.
(***The results of this work are communicated in International SCI Journal)***

Chapter 7 deals with the derived simple analytical relations for the effective material properties such as elastic and piezoelectric properties of a 1-3 composite, where both the matrix and the fibres are piezoelectric materials. These relations are important for optimizing the volume fraction of piezoceramics in the polymeric matrix for the specific biomedical applications. In this study, PZT piezoceramic fillers are considered embedded in a PVDF matrix. The material properties of such composites are evaluated from the derived analytical relations and have been validated by finite element modeling using COMSOL Multiphysics software.
(***The results of this work are communicated International SCI Journal)***

In Chapter 8, the practical application of 1-3 PZT/PVDF composite in biomedical applications has been explored. The material relations derived in the previous chapter have been utilized for the optimization of device parameters and the ceramic content of the composite in cantilever configurations. It is seen that the optimized volume content of PZT ceramic in the polymer matrix is dependent on specific applications. It is found that bimorph configuration is better for actuator applications in biomedical devices as compared to unimorph cantilever.
(***The results of this work are published in (i) International Journal of Engineering Trends & Technology, vol. 4, p. 2675, 2013 and (ii) Manuscript under preparation)***

The significant findings are summarized in Chapter 9.

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