Chapter - VII

Conclusion of This Thesis on Characterization of FE Transducer Material for Electronic Applications
7.1 HIGHLIGHTS OF FERROELECTRIC TRANSDUCERS CHARACTERIZATION

After more than 60 years of the discovery of ferroelectric materials it has become well established for electronics devices. The special features of FE materials being high dielectric, pyro-electric, piezo-electric, the bistable characteristics has leads to very large number non-linear device applications.

During these studies a special credit goes to the presented work in the preparation of PZT and PLZT of different configurations for transducer studies. These characterizations were done under various physical conditions of stress, temperature and pressure. The transducer is made up in capacitor form placed in special designed holder in forms of thin films, crystals or pellets. Measurement of the changes in capacitance, dielectric constant and tangent lost were performed with useful application results.

Very prominent outcome of the research has been the development of various circuits for measurement purposes. Study begins with electronic temperature measurement circuit for temperature variation of the sample for transducer characterization. Another circuit was developed for pressure measurement. Unique circuit was developed for capacitance measurement which is compatible for PCL 812 card. For characterizing transducer material Data acquisition system was developed using DIO add-on-card PCL 812. Digital technique of measurement with cheaper integrated circuit was used. Results with these system instruments built in our laboratory when compared with conventional standard costly equipments. It works out that our all circuit developed work excellently and precise characterization results are obtained.

7.2 CONCLUSIONS OUT OF CHAPTER III

The solid-state reaction technique used for preparation of some of our compounds; the structural characterization and the verification of single phase were done through XRD and surface studies by scanning electron
microscope and by vibration spectroscopy. The observed XRD pattern shows
the preparation of single phase and good quality compound supported by the IR
spectra. The SEM analysis shows the homogeneous grain size of the sample.

This thesis begins with a brief introduction of Data Acquisition
system. A brief review of the literature relevant for circuit application and
related compounds are also presented in each section. In third chapter
temperature and pressure measurement circuits were reviewed. Existing
calibration techniques error checks the circuit performance; efficiency and
resolution are also calculated. Calibrations of all circuits designed were done
by the standard methods. Calibration curves show linearity over wide ranges.

C-V measurement circuit characterizes for the ferroelectrics
semiconductor devices PZT, PLZT, TGS, TGSe and PVF₂ with MFS devices.
For this purpose, these films were deposited on an n-type Si wafer by solution
casting or evaporation technique. Thereafter applying a bias voltage of ± 30 V
C-V characteristic was investigated. All the films have shown memory
behavior when the measurements are undertaken in the step of ± 2 Volts. The
effect of depletion capacitance $C_D$ for each film has also been noticed for all the
devices.

The third chapter contains the overall DAS development, its
circuit operation adjustments, block diagrams, circuit schematics, calibration
and reading taken by these instruments developed.

All the material chosen for studies are characterized by C-T and
C-P curves obtained by various circuit connections interfaced to computer. It is
seen that anomaly in capacitor near melting temperature occurs at different
temperature for different samples. C-V characterization of PZT and PLZT thin
films shows the variation of Capacitance with applied d.c. voltages.

For 5µ film of PZT the maximum capacitance was obtained on
temperature $200^0C$ and its value had been found $24x10^{10}$ farad similarly for
second PZT film of 10.5µ the maximum capacitance value obtained was
38x10^{10} \text{ farad} \text{ at temperature } 205^\circ \text{C}. \text{ For high pressure the capacitance variation was for film PZT1 } 86 \text{ nf to } 172 \text{ nf, and for PZT2 in the range } 106 \text{ nf to } 182 \text{ nf.}

The value of capacitance for PVF_2 16\mu film ranges from 58 \text{ nf to } 59.8 \text{ nf. The value of capacitance for } 25\mu \text{ film values varies from } 60.8 \text{ nf to } 61 \text{ nf for } 5 \text{ tones per cm pressure. In the entire graphs values of capacitances are increases with pressure with constant slope. This shows sensitivity of material towards pressure variation and suitability for pressure sensor device of these FE PVF_2 films.}

Comparison of TGS and TGSe pellets on the basis of capacitance versus temperature indicates that capacitance increases up to } 72^\circ \text{C. Anomaly occurs for TGS pellet at } 120^\circ \text{C. After that capacitance decreases sharply. Anomaly occurs for TGSe at } 60^\circ \text{C. The value of capacitance decreases again after } 140^\circ \text{C it starts increasing.}

This is first time hard and soft ferroelectric compared on basis of their dielectric behaviors.

- The temperature dependent change in capacity with constant frequency comes out to be maximum for PLZT and minimum for TGSe transducer.

- The pressure dependence is more for PZT and PLZT material in comparison to PVF_2 and TGS related group showing that they are more suitable for piezo device.

\textbf{CONCLUSIONS OUT OF CHAPTER IV}

This chapter contains a success story of obtaining plot of computer aided very simple combination hardware and software system that performed the task for obtaining hysteresis with very simple circuitry. But here again to measure spontaneous polarization and field we employed other circuitry. In spontaneous polarization plot for PZT and PLZT temperature dependence confirmed that it is decreasing with temperature.
At temperature 25°C, 80°C, and 150°C it is clear indication of the reduction of spontaneous polarization with the different ratio combination 8/65/35, 15/40/50 and 18/30/70 for PLZT films. The hysteresis loop show good symmetrical shapes. Varying frequency did not have significant influence on the hysteresis loop. The device shows shift along polarization as well as field axes. The value of remanant polarization $Pr = 7.4 \mu\text{C/cm}^2$ and $-Pr = 11\mu\text{C/cm}^2$ and electric fields are $+Ec = 45.4 \text{kV/cm}$ and $-Ec = 38.2 \text{kV/cm}$ thus the polarization shift has been reported in “graded ferroelectrics”. However in a homogeneous PLZT film, the shift may be also due to electrode asymmetry which may be in the case of our PLZT films also.

All the samples show single phase with tetragonal structure. It is observed that the transition temperature decreases linearly with lead concentration. Also the degree of squareness $(Pr/Ps)$ decreases with higher lead concentration.

The Piezo film exhibited the hysteresis loop. In the solution casted PVF$_2$ film an elliptical loop was observed at low fields. Some sort of nonlinearity was observed at high fields FE can not be feasible and shows with one proper poling stretching and orientation. These observation suggest that the ferroelectrcity in PVF$_2$ is due to Form I crystallites which are present in the piezo film after it is stretched biaxially or poled uniaxially.

Spontaneous polarization and coercive fields were calculated for PZT, PLZT, PVF$_2$, TGS, and TGSP at different temperature and on different bias voltages. Curves for pure TGS at different temperature confirmed second order nature of phase transition. Hysteresis curves of phosphate doped TGS crystals with two different concentrations have been traced on computer. The Spontaneous polarizations have been determined at different temperatures. It was found that the transition temperature shifted towards lower values with increasing concentration of the dopants. The temperature dependence of spontaneous polarization is also confirmed for all materials. Coercive field calculation was also done from reading of samples from 0°C to 50°C where

CONCLUSION OF THE THESIS
hysteresis loop reduces to a single line at transition temperature. It is seen that spontaneous polarization of pure TGS decreases with increasing temperature. At \( t = T_c \) at the Curie temperature its value become zero. After that gradual variation indicate second order transitions.

Advancement in thin film ferroelectric integrated circuitry have proved suitability for high density non volatile random access memories devices by tailoring the polar properties of the bulk material in thin film forms of PZT and PLZT lead to develop higher density memories. Hysteresis loop studies of these materials with different compositions on silicon substrate resulted in improved ferroelectric properties as characteristics parameters to be used in devices. The work conducted in the study for PVF₂, TGS and TGS related group to obtain hysteresis parameter has good potential for characterizing these materials.

CONCLUSIONS OUT OF CHAPTER V

Chapter focuses on the development of novel circuit designing based on 555 for measurement of dielectric constant and capacitance for direct read out by digital technique. Details of circuit description are presented. Capacitance measurement is compared with existing instrument in our laboratory. The dielectric constant and dielectric losses are measured with frequency from 50 KHz to 10 MHz by using source-detector. In further study of PZT1 and PZT2 transducer is done for the variation in frequency. For lower temperature region it is almost parallel; it means it does not depends on temperature rise. This study is done with frequency measurement circuit. From the changes in frequency for PZT it became quite linear and then there is sharp rise in the frequency after 135°C as it goes on increasing unto 250°C. Dielectric constant increase up to certain concentration then starts decreasing, while the trend is reverse for dissipation factor.

The material is characterized for pressure and stress transducer. The PZT, PLZT and PVF₂ are mostly suitable as pressure and stress transducer for electronic application. The dependence of dielectric constant and dielectric constant increase up to certain concentration then starts decreasing, while the trend is reverse for dissipation factor.
loss on frequency of the applied a.c.field was studied in the temperature range of 30°C-150°C and frequency range of 50 kHz-10MHz for PVF₂. For higher temperatures exceeding 100°C the value of decreases continuously at frequency till 10MHz beyond which it rises again. The dependence of on frequency shows similar behavior for all temperature exceeding 100°C.

This variation for 16 µ and 25 µ poled films of PVF₂ with its comparison of dielectric constant with 25 µ un-poled film. That indicates dielectric constant sharply changes with temperature for poled 25 µ film. As a consequence of molecular motion at elevated temperature the change in dielectric constant occurs this causes the change in frequency according to mathematical formula. It is also seen that lines are quite parallel, reflecting the same internal activities during the rise in temperature.

The variation of frequency with pressure the initial value was 15x10⁵ Hz at room temperature after applying a uniaxial pressure on the sample the frequencies linearly increases up to 20x10⁵ Hz for per Newton meter square, thereafter increase is slight for PVF₂.

The dielectric constant then decreases slowly in para-electric phase. Here it is to be emphasized that our εₘₐₓ is the highest value amongst the εₘₐₓ reported on TGS films so far. The occurrence of dielectric anomaly in the film clearly shows the existence of ferro-electricity in it the dielectric instability at Tc being one of the important criteria

It is to be emphasized here that preparation and dielectric properties of TGSe films are being reported here for the first time. The dielectric constant increased with rise in temperature as in bulk sample and a peak occurred at ~ 23.5°C. The dielectric constant was 25 at 19°C. εₘₐₓ is 30 at 23.5°C and 39 at 39°C. Sharp dielectric anomaly indicates that ferroelectrics b-axis is preferably oriented perpendicular to the plane of the films. Further, the high value of E for the polycrystalline films at Tₑ (23.5°C).
The dependence of dielectric constant ($\epsilon$) and loss tangent ($\tan\delta$) on temperature in the temperature range $30^0C-150^0C$ was studied in the frequency range 100 KHz to 10 MHz for PZT and PLZT and have been reported. In this chapter it can be seen that the slope decrease with the rise in temperature, suggesting, that the electronic hopping conduction diminishes with the rise of temperature.

The detailed analysis of dielectric constant and dielectric loss as a function of frequency indicates dielectric anomaly indicating consequent structural phase transition from ferroelectric phase to para-electric. It is also show that this anomaly is represents a second order normal ferroelectric phase transition.

It was observed the plot between frequency and temperature increases due to para electric behavior of Triglycine selenate. The fall in curve above the temperature $22^0C$ justified due to the para electric phase of Triglycine selenate because $Tc$ of Triglycine selenate is $22^0C$ and beyond $22^0C$ it shows the para-electric behavior.

**CONCLUSIONS OUT OF CHAPTER VI**

Chapter VI deals with vibration analysis measurement employing piezo transducers by PZT and PLZT pellets. The vibration meter by simple circuit design is successfully made. The vibration measurement system designed by me is based on piezo-accelerometer, which is interfaced with our electronic circuit design to read vibration of any vibrating system in the range of 10-1000Hz. Each stage of circuit was checked by computer simulation on work bench. The proposed design is an easily implementable and low cost solution for vibration measurement. The computer results are encouraging and portray the feasibility of the circuit design for practical vibration measuring system for mechanical parts.
The present characterization of Capacitance transducer in this thesis has shown many of their desirable properties-

1. Most of them show linearity over wide range of temperature dependence as described by C-T characteristics as desirable device characteristics.

2. They can be used as pressure transducer PLZT and PZT may be used as vibration sensor.

3. The circuits developed by us for measuring system have wide scope of adaptability as being simple and affordable in all measuring laboratories. They present low error and high proficiency.

4. C-V characterization showing hysteresis properties. Interfacing with metal and semi conductor these ferroelectric materials applicable for memory devices.

5. For transducer characterization of PVF2 its capacitance shows the effect of poling and stretching conditions and biasing field ferroelectric polymer.

6. The sensors involvement in study we tried to implant it in circuit capacitor.

7.3 FURTHER PROSPECTS OF TRANSDUCER CHARACTERIZATION

The first circuit developed by author is based on op-amp 741 employing some resistances and 3 IC chips for measuring temperature. Micro-electronics technology can miniaturize this whole circuitry into single chip. This very simple circuit in conjunction with thermocouple in build can serve as very precise contact temperature measurement system that can be interfaced with PC.

The second circuit developed is based on LM108 and two Op-Amp 741 with, some resistances and capacitors and interjection with ferroelectric material can be served as pressure measurement contact system. This all circuit devices can give rise to new single chip form in
microelectronics ferroelectronic technology as recent trend of ferroelectric application.

The third circuit developed based on op-Amp 741 in conjunction with IC 9400 that can be served as capacitance measuring system in single chip form.

New part of circuit is based on frequency and vibration measuring system IC 555 in conjunction with CD 4033 and FND 543 in a single chip form can serve as frequency measuring system having digital read out with various ferroelectric devices potentiality.

Vibration measuring system can also be minimized to make a single chip with some modifications in circuit.

1. By measuring vibration it can be possible to improve the condition of machinery by active controlling it. This gave way to realize the ultimate goal of a "smooth and Noisless machine."

2. Only with the advancement in electronics instrumentation in electronics, instrumentation, materials and system integration techniques, smart structures came into existence. To make a structure smart, a smart system is to be integrated to it. A smart system comprises transducers and controlled networked together. Interfacing of transducers to different networks involves enormous efforts to built efficient system.

A vibration sensor development can help in fabrication of a seismic mass supported by thin silicon hinges using bulk micro machining. Movement of the seismic mass in the presence of vibration and can be monitored to sense vibration.

Vibration sensors have become an integral part of many MEMS which consist of mechanical components, sensors and electronics all integrated in the same environment. Development of MEMS requires fabrication of smart sensors using micro fabrication of mechanical as well as electronic component
on the same Chip. Miniaturization of components has many desirable factors e.g. small size, low cost & high precision.

7.4 FUTURE SCOPE OF STUDY

The role of ferroelectric in electronics has vast potentialities for sensors, transducers N/E devices and memory devices. Our work was to develop new circuit of characterizing ferroelectric materials and their potential device applications. Presentation given by us will be further significant of technological development for ferroelectric based electronic applications.

The study of frequency and vibration can be employed best for:

- To verify machine condition.
- To check the condition of rocket, aircraft.
- To check the railway track by studying vibration of track.
- To check the train accidents.

C-P and C-T characterization of material suggest that they can be utilizing in a single device transducer form in industry, factory and many other electronic application in laboratories for research development.

Mechatronics is the new emerging field to implement technological development of different fields as mechanics hydraulics and pneumatics, sensorics and control where in sensorics ferroelectronics has major role. Mechatronics system are modern means such as FE sensors actuators or combined solutions so as to FE to implement in robotics, aerospace control and industrial automation which combines classical mechanical system with embedded modern by the intelligence of electronics called intelligent smart system.

The electronic intelligence can be defined as integrated system using logic control and algorithms, neural network, fuzzy systems and genetic algorithms that improves the performance of the system in modern instruments and system.

CONCLUSION OF THE THESIS
By interfacing a ferroelectric device directly with a semiconductor material, it is possible to construct several types of interesting devices, in which the basic electrical properties of the semiconductor enhanced by the polarization state of the adjacent ferroelectric by the utilizing the ferroelectric field effect. Ferroelectric field effect semiconductor C-V characterization suggests core memories and DRAM memories may have potentiality in ferroelectric technology.

One of our largely studied PVF₂ poly vinylindene fluorides is one of the first industrially important ferroelectric polymers. The C-V characterization of PVF₂ shows possibility for application in MEMS. Its C-P characterization observed at different pressure shows electrical variation with pressure shows electrical variation with pressure. Its C-T characterization observed variation of temperature changes the corresponding capacitance and can have special devices applications.

PVF₂ being piezoelectric has wide scope in sensors applications like telephone, microphone, acoustic transducer and pyro-electric detector one has special features for implanting capability in body system.

The temperature, pressure, poling and stretching of device samples largely affects their characteristics properties. Hence it is important to take into account all these varying parameter in design of the device made of PVF₂ for application with all suitability.

Ferroelectronic sensor and transducer interfaced with electronic circuit design show the technological developments and future contribution towards a change from 'structural' to 'functional' and further on to 'smart' material concept. They may be termed as functional materials in the sense that their properties can be changed by proper selection of ferroelectric material with right Integrated Chips, thus affording the prospect of developing ferroelectric materials for diverse applications as in new field of ferroelectronics.

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