Chapter III

Development of Data Acquisition System for Ferroelectrics

Electronics Applications
3.1 INTRODUCTION

Data acquisition is the process of capturing the output of a sensor and connecting it into a suitable form for further processing and usage. A data acquisition system essentially consists of a set of transducers, signal condition the recording media and the data reduction system that include data digitization, processing, analysis and presentation system. In automation systems such a device enables the measurement faster and also avoids errors generally that occurs in manual measurements. Now-a-days with advent of computer lot of research is going on to develop data acquisition system for take measurements as per requirement. Computer, microprocessor and micro controller are tools for data acquisition system which is now a vast field of technology. [1-4]

Many author have reported computer based measurement systems for voting machine, signal representation, grain moisture measurement, Ph measurement, servo motor control, weighing machine control, temperature measurement, speed measurement etc. [5-7]

IBM PC based data acquisition system was reported as developed by Reddy\(^8\) to characterized I-V, C-V plots for semiconductor device. On the same line Patel and Bhonsle\(^9\) made a general purpose PC based Digital data acquisition system. Mathivanan\(^10\) made XYT plotter based on PC.

Mohite and Bodhe\(^11\) developed integrated weather and air pollution monitor system based software for PC data acquisition system. Spacecraft testing equipment, structure testing, vibration measurement, medical based ECG, echocardiography C.T. scanning was based on computer data acquisition. [12-14]

In field of characterizing ferroelectric material based on computer system the literature was formed to be scarce. K Sing and S.S. Bhoga\(^15\) developed computer automated Complex impedance measurement system for measurement of six dielectrics devices on same atmospheric condition. For
measurement of electrical parameters at elevated temperature using varying frequency K.K. Bamzai and P.N. Kotru used computer based set-up for impedance analyzer and HP VEE based software for recording and analyzing data. To quote a few examples Venkatarghavan et al. have designed and fabricated a versatile liquid phase Epitaxial system. Verma et al designed and fabricated successfully X-ray diffraction Topography system at NPL, New Delhi. At Pune University Shaligram and Savant et al. fabricated optical wave guide and measured parameter with computer automated system. A complete system with traversing mechanism and advanced scintillation counter and versatile turntable was established soon thereafter. An inexpensive and simple atmospheric pressure chemical vapor deposition (APCVD) reactor for preparing large area transparent conducting tin oxide thin films had been designed and fabricated by center of Electro-Chemical Research Institute Karaikudi [20] Many other systems / equipments have been developed indigenously by Indian Scientists e.g. eddy current transducer systems to locate garter spring spacers in highly radioactive coolant channels of Indian PHWR type nuclear reactors by BARC Mumbai. [21] Vibrating sample magnetometer for magnetic multiple moment detection by Dr. R.K. Krishan was developed at National Science centre for Biological Sciences etc.

3.1.1 Factors Influencing the Data acquisition System

Configuration of the data acquisition system influence parameters plays an important role as process variable to be measured and controlled. These parameters come into picture due to demands of the measurement during dynamic and static testing. [23]

Number of response channels.

How many different variables are going to attached with data acquisition card is first thing to be taken into consideration. For such a complex system, number of locations to be monitored is large to obtain sufficient data for proper understanding of specimen's behavior. [24]
Type of measurement

Acceleration, strain/stress displacement velocity are some of these types of data that can be obtained from transducers. Static or dynamic type of transducer may be required for process to be undertaken, according to their need. The signal conditioners for the sensors generally provide voltage output of $\pm 10V$ amplitude range. As sine vibration test data acquisition requires very good frequency resolution. For random data acquisition FFT techniques can be used. Along with it both time domain and frequency domain data are to be stored, the system should be able to provide the facility for the same. [25]

Frequency resolution -

Process variables and transducer output must be in same phase and time that can give correct information of process. Frequency resolution should be such that at the fundamental resonance frequency there should be at least (5-6 points) in frequency domain within frequency range. [26]

Noise Consideration -

Noise affects the measurement when the acceleration data of higher order being measured. ADC module should have the dynamic range to handle low signal to high signal. It should be wide enough to handle the maximum possible change.

Time duration of test or measurement -

These factors influence instrumentation bus speed and acquisition range both should be within range. Time parameter affects storage capacity for acquiring data. [27]
Type of analysis to be carried out -

Analysis of the acquired test data is carried out in two stages, online during acquisition and off-line post-test analysis, with adequate speed and accuracy to process signals in real-time.

Data acquisition during dynamic testing involves measurement of a large number of responses. This includes providing instrumentation acquiring the response data and analyzing it to obtain an understanding of behavior of the test specimen.

Basic on-line analysis involves amplitude and frequency estimation of the test data and digital filtering. Off-line analysis of the test data includes computation of transfer function, damping and estimation, SRS analysis, narrow band analysis, octave analysis and various mathematical operation such as integration, differentiation etc. These analysis features are provided by data acquisition software capabilities. [28-30]

3.1.2 DATA ACQUISITION SYSTEM PARAMETERS

Signal Conditioning:-

In case of low signal amplitude, the instrumentation front-end should have the capability of programmable gain for optimal usage of dynamic range of the ADC. It is ideal to have auto-ranging ADC.

Buffer Management -

For sine data acquisition to obtain good frequency resolution a large buffer size is needed.

Computational Power -

On-line analysis of more than 100 channel data puts a huge computational burden on the computer's CPU. An alternative method is to distribute the computation among multiple processors.
Instrumentation -

For bus speed Instrumentation the bus should be capable of providing data flow rate at better way. Once the signals are available and converted digital ones, vast possibilities of linking it to digital computers and using digital communication also opens up. Therefore very often unless the instrument / instrumentation system is simple and small the transducers output is converted to digital signal after minimum necessary processing and all other processing is carried out on the digital signal using digital circuits and devices.

[31-38]

3.2 System development using PCL 812 card

For developing computer based system PCL 812 card compatible P1 are fitted into one of the empty ICS slot of the PC motherboard. We used CN1 and CN2 connector having facility of analog to digital and digital to analog converter for different measurements.

Strategy of channel distribution opted for measurement is given in following table [3.1].

<table>
<thead>
<tr>
<th>Measurand</th>
<th>Channel number</th>
<th>Pin input</th>
</tr>
</thead>
<tbody>
<tr>
<td>Capacitance measurement</td>
<td>A/D 10</td>
<td>1-2</td>
</tr>
<tr>
<td>Voltage measurement</td>
<td>A/D 11</td>
<td>3-4</td>
</tr>
<tr>
<td>Temperature measurement</td>
<td>A/D 12</td>
<td>5-6</td>
</tr>
<tr>
<td>Pressure measurement</td>
<td>A/D 13</td>
<td>7-8</td>
</tr>
<tr>
<td>Voltage variation</td>
<td>D/A 15</td>
<td>13-14</td>
</tr>
<tr>
<td>Voltage control</td>
<td>D/A 16</td>
<td>15-16</td>
</tr>
</tbody>
</table>
3.2.1 About PCL 812 Card

1) It is multifunction data acquisition card 12 bit successive approximation converter equivalent to ADC (574) to convert analog inputs. Max A/D sampling rate is 30 KHz in DMA mode.

Bipolar ± 10 V Max. Range

2) 2, 12 bit monolithic multiplying D/A output range + 5 V.

3) Using CN2 Connected which contain analog input / out both.

4) Six analog inputs from different source can be connected to this connector

5) Two digital to analog converter wire can be taken out one for hysterias other for capacitance measurement circuit

6) Data is collected through software commands.

7) Utility software in high level language is used for transducer measurement.

8) PCL 812 card is fitted into PCS slot of motherboard. [39].

Block diagram of PCL812 with its connectors is represented in fig (3.1)

Details of all computer based measurement system and digital technique for measurement are given separately in each chapter. During the course of investigation the basic nature of device its, characteristics properties were measured by instruments existing in lab and other labs are now discussed in following sections.
PCL 812 Card Connections

Capacitance Measurement Circuit

Voltage Measurement Circuit

Temperature Measurement Circuit

Pressure Measurement Circuit

Voltage Variation Circuit

Voltage Control Circuit

Fig 3.1
3.2.2 CIRCUIT DESIGN FOR TEMPERATURE MEASUREMENT FOR FE TRANSDUCER

3.2.2.1 Background of Temperature Measurement

Temperature is an important parameter to be accurately monitored and controlled in all spheres of human activity. Commonly used temperature transducers are thermocouple, resistance temperature detector (RTD), and thermistor and IC sensors. Modern instrumentation uses not only extensive electronics but places great emphasis on digital circuit and techniques which have much higher reliability and immunity to noise as only two signal levels (against infinite levels in analog systems) are used and active circuit devices like transistor work in switching mode rather than analog signal processing. Storing digital information (in semiconductor and bubble memories) is far more convenient than storing analog information. There is no loss of information with time. [40-43]

Automations of industrial process control systems require development of sophisticated microcomputer system for data acquisition and control of process parameters. Computer and data acquisition based temperatures control and measurement can be effective by different manners with many ways. Direct digital control had been tried by many people. Digital control makes possible to use improved control algorithm thereby improving the accuracy of the control and provide a wider range of control setting. [44] Microprocessors based temperature control has been tried by Bhusawalwala 45 by using PID Algorithm.

An effective IBMPC based temperature controller using simple heater and coil temperature sensor PT100 was used to sense the change in temperature was given by Natraj 46 et al.

An inexpensive digital technique is used for measuring temperature developed by Bhanot 47 using 741 IC. Fiber optics based temperature sensor was fabricated by Pandit 48 et al. temperature of
semiconductor has been sensed. Thermistor based temperature control has been
tried by many author. Thermocouple based hardware for micro controller based
measurement AT89C52 was designed by Neelmegam. For the study of
capacitance, dielectric constant, tanδ variation due to temperature, provision for
temperature variations are established within the cell. For the temperature
variation in the cell used a heater filament as heat source and the temperature is
measured by using K-thermo-couple probe coupled with data logger circuit
shown in fig. (3.2) Cold junction compensation is provided by AD590
Temperature semiconductor. [50]

3.2.2.2 Thermocouple Conditioning Circuit.

The heart of a typical temperature measurement is the sensing
thermocouple. The standard K-type thermocouple was carefully stress relieved
and mounted on film in such a manner to provide good thermal transfer and
protection against moisture and the process medium. The signal output of
thermocouple is proportional to the temperature. The thermocouple is
connected directly to the differential amplifier and amplifier deciphers any
change. As the dynamic variable (temperature) is raised or lowered the signal
varied accordingly, such change is felt at the amplifier. Major functions
necessary to signal condition of thermocouple to interface it with ADC card are

* Excitation circuit
* Isolation of couple from other circuitry.
* Cold junction compensation
* Amplification
* Offset control
* Gain control
* Scaling control
* A / D conversion
Circuit for Temperature Measurement

Block Diagram for Temperature Measurement

Differential Amplifier → Offset and Gain Control → ADC Channel → PC

Fig No. [3.2]
3.2.2.3 Description of Cold Junction Compensation Circuit.

AD590 is a semiconductor sensor easily available and accurate for temperature measurement between 0°C to 150°C. It uses the fundamental property of dependence of base-to-emitter voltage on temperature. It provides an output current proportional to the absolute temperature for supply between 4 - 30 V. The circuit acts as a highly impedance constant current regulator passing 1μA/°K. Apart from being cheap it exhibits linear behavior. The inherent low cost and easy availability of monolithic IC combined with the elimination of supporting circuitry make the AD590 an attractive alternative for temperature measurement and control applications. It is available in chip form in small metal can or ceramic flat-pack package or in ready made probes. It has the following features: accuracy 0.5°C specified of 25°C low cost [51-54]

- Measurement range 5 to 150°C
- Supply voltage range 4 to 30V
- Linearity of ± 0.3°C cover the full range.

The important building blocks of temperature measurement system were used in present study

- Thermo couple junction
- Electronically cold junction compensation
- Differential amplification unit
- Buffer and offset gain control
- Output to channel of ADC

First of all thermocouple was kept in melting ice and corresponding voltage is measured by millivoltmeter. Then at room temperature and after that boiling water calibration curve is obtained in terms of milli-voltmeter. This output is very small and must be amplified to bring it up into the range where it can drive an A/D converter.
In order to make accurate measurements a second junction made of the same must be included in the circuit as reference. Adding this second junction is referred to as amplifier of gain 1000 amplifies cold junction compensation output of thermocouple. The resultant output voltages are the difference between the voltages across the two thermocouple second junction compensate electronically in the temperature of the reference junction by AD590.

3.2.2.4 Hardware description of Temperature Measurement

The block and complete diagram of temperature detection and measurement unit is shown schematically in fig (3.3). It comprises of an instrumentation amplifier. Instrumentation amplifier is made up of three op-amps namely IC741 (U1, U2 & U3) The first two op-amp U1 & U2 provide a high input impedances' because the signals go directly into the non-inverting inputs of the op-amp and the op-amp U3 represents the usual differential amplifier. The signal source of the Instrumentation amplifier is the output of the thermocouple.

Calibration Arrangement

Calibration arrangement for temperature measurement unit is shown in Fig (3.4) firstly the thermo couple was connected with the digital millimeters through shielded wire. Now the thermocouple is placed in melting ice and the output of thermocouple in terms of voltage was recorded. Then thermocouple is placed in the furnace A. Thermometer T is kept inside the furnace to measure temperature. When the furnace A is connected with power supply the temperature inside the furnace increases gradually and when the temperature becomes steady (10°C) the output of thermocouple is recorded in terms of voltage. Again the temperature of furnace is increased and output recorded in terms of voltage. Finally a graph is plotted Temperature Vs output voltage in millivolts. Which obeys the equation y = mx+C and calibrated from 0°C to 250°C.

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INSTRUMENTAL AMPLIFIER

Fig. (3.3) OP AMP user 741
Temperature Measurement through Computer Calibration arrangement

Chromel-Alumel Junction

Fig. (3.4)
3.2.3 CIRCUIT DESIGN FOR PRESSURE MEASUREMENT

PZT transducer is used for hydraulic pressure measurement. The procedure is same as used by Clark and V.R. Singh\textsuperscript{55} for measurement of pressure. We bought PZT pressure transducer by Transducers Company Pune. For very low pressure measurement Rao\textsuperscript{56} made digital micro-manometer based on capacitance principle. The calibration system was built like measurement shown in fig. (2.11) in section [2.3.4] and pressure was monitored through digital system. The pressure shown by manometer is calibrated in terms of voltage obtained in graph. For further measurement assembly was used to mount our test device and electrodes were connected to Capacitance Bridge, for dielectric measurement circuit. Measurement of these parameters by variation of pressure has been done. The circuit diagram of complete arrangement is shown in Fig. (3.5)

For the change in pressure within the cell hydraulic press was used for creating high pressure and vacuum pump for low pressure Piezo - resistive pressure sensor PZT was used for low pressure variations.\textsuperscript{57}

3.2.3.1 Component of Pressure Measurement circuit

The block diagram of pressure measurement circuit is also shown in fig. (3.5). The main blocks of pressure measurement circuits are charge amplifier as preamplifier having pressure transducer in its input, rectifier and clipper circuit are the other part of this system.

It contains three major components namely

(i) Manometer circuit is conveniently designed to use water as experimental fluid.

(ii) A capacitive transducer, the main innovation in this component is to make use of mercury meniscus to measure pressure.

(iii) Instrumentation amplifier to connect this transducer to PCL 812 card.
Pressure Measurement Circuit

Block Diagram for Pressure Measurement Circuits

Fig No. [3.5]
CLOSED LOOP FOR OBTAINING C-V CHARACTERISTICS OF FERROELECTRIC TRANSDUCER

Fig No. [ 3.6 ]
The pressure transducer is effective as piezo-capacitor, which produces a charge ‘q’ across its plate’s proportional to a pressure, applied to the crystal. If we change the water level pressure changes it will generates charge. The open circuit voltage ‘E’ out of the transducer is equal to the generated charge divided by the transducer capacitance or.

\[
E \text{ (volts)} = q \text{ (Pico coulombs)} / C_p \text{ (Pico-farads)} \quad (3.1)
\]

To condition the piezo-electric transducer output, the charge amplifier technique is employed. Since the charge generated does not change regardless of the amount of external capacitance added, the system sensitivity was unaffected by the length of the cable between transducer and amplifier. As the capacitance varies, the charge also changes according to the equation

\[
Q = C.E \quad \text{----------(3.2)}
\]

When the transducer is connected to the inverting terminal of the op-amp, this charge flows into the feedback capacitor \(C_r\). The resultant change in charge of \(C_r\) generates an output voltage.

\[
\frac{-C.E}{C_r} \quad \text{----------(3.3)}
\]

Since the op-amp requires a DC path from each input too common. For bias current flow, it is necessary to insert a resistor \(R_r\) in the feed back path.

The purpose of rectifications is to provide a DC output to the ADC card. Here a precision rectifier was used where the diode was used with an op-amp in the feed back path hence the threshold voltage was divided by the open loop gain of the op-amp, which enables to rectify even mV signals.

The DC signal through the precision rectifier was fed to ADC card, which changes the analog value in digital form directly.
3.2.4 Circuit design for voltage variation and control for ferroelectric transducer

Programmable Voltage Source

This stage consists of DAC channel inverting amplifier, summing amplifier and higher voltage amplifier circuit. This voltage source can be programmed over a voltage span ± 20 V, in any step voltage desired up to a minimum step of ± mV.

The detailed block diagram of this programmable voltage source is shown in fig. (3.7). It consists of a unity gain inverting amplifier, a summing amplifier and voltage amplifier stage.

During the voltage - capacitance measurement, the system software can generate and control the DAC1 output ladder voltage between 0 to 5V. The system software also controls the output of DAC 2 which is coupled to the inverting unity gain amplifier to generate a d. c. offset voltage from 0 to - 5V. Summing amplifier adds the ladder voltage of DAC1 and the offset voltage so that the ladder voltage of DAC1 can be set anywhere between - 5V to ± 5V level in a desired manner. Output of summing amplifier was given to high voltage amplifier stage of fixed gain 2. The interactive software allows the user to enter the upper and lower voltage limits and the increment in the voltage for sample C-V run accordingly, the start and the termination of DAC1 ladder voltage, step height of ladder voltage. The system software automatically decides offset voltages at DAC2. All gain determining resistors consists of combination of fixed resistors and variable trim pots in order to improve the accuracy of the system.

The inverting and summing amplifier were made up of IC741 is also shown in fig. (3.6)

The final output amplifier stage is built using high voltage op - amp LM 143 in non-inverting into fixed gain of 2. A separate ± 30 V supply is used for this stage and the output voltage obtained here is ± 20V. Reading it
PROGRAMMABLE VOLTAGR SOURCE

[Diagram of electronic components and connections]

Analog input
Logic input

Summing Inverting Amplifier

Fig 3.6
through ADC channel 11 to form a closed loop is monitoring system that monitors the voltage at the output of programmable voltage source. This keeps on adjusting DAC1 output till the desired voltage at the output of the programmable voltage source is obtained. This ensures exact increment in the voltage output.

The resolution of 12bit DAC with the maximum output voltage of 5V is 1.22 mV. Therefore, ideally at the lowest gain of 0.5 of PVS a minimum step voltage of 0.6 mV is achievable. However taking into consideration of the effect of noise the PVS minimum step voltage is limited to ± 10 mV. This has been tested repeatedly with good accuracy and stability. Care should be taken to eliminate high frequency noise by connecting 0.01F capacitor in the feedback loop of amplifier and bypassing all the IC supply terminals to ground with 0.47 mF capacitor. The complete circuit diagram of programmable voltage source is shown in fig. (3.7).

3.2.5 Circuit design for capacitance measurement for Ferroelectric transducers

3.2.5.1 Background of capacitance measurement

Advancement of VLSI / ULSI technologies, metal semiconductor contacts have been subject of interest due to capacitor characteristics, the Capacitance depend on voltage and play an important role in device application. [58] Capacitance methods for characterization of semiconductor involves the application of reverse biases to semiconductor junction and measuring capacitance of the junction [59] Many parameter evaluate in deep level capacitance transient spectroscopy with pulse of short duration (injection pulse). For semiconductor devices many author-tried microcomputer based capacitor measurement like D.V. Lang6⁰ C.V.R. Reddy⁶¹ and LV Reddy⁶² Measurement of capacitance with AC bridge circuit was reported by P. Holmberg⁶³.
Fig 3.7 Actual circuit of C-V Loop Characterization
Ananthi and Padamanabhan used chemical sensor placed between electrodes with a cylindrical geometry measured its net capacitance and conductance by bridge techniques interfaced to computer.

3.2.5.2 Principle of capacitance measurement

Transducer had been prepared in form of solid (bulk or film) its contact or impregnation with a base material and two foil electrodes attached on the opposite sides of the substrate. Such a transducer element behaves as combination of capacitance and conductance.

When an A.C. bridge is employed, its detector arm output yields an output that is a composite signal due to the both charges in capacitance and conductance of the sample. This is the principle of Wayne-Kerr auto Balance Bridge [65]. By following same principle shearing bridge had been build by using operational amplifier. As described the operation of Wayne-Kerr bridge all the measurement and analysis by it was time consuming and difficult. More over, effect of temperature and Pressure on dielectric properties is an important aspect for its application. In this direction an effort had been made to fabricate a setup in which data on all dielectric properties (Capacitance conductivity, dielectric constant etc) of a material is directly recorded and analyzed with the help of a computer. The effect of temperature and pressure of the applied A.C. and D.C. field on such parameters is monitored, recorded and analyzed with the help of this set up. [66]

The following sections briefly describe the hardware of set-up.

3.2.5.3 Circuit description for capacitance measurement

Material undertaken for study was in capacitive transducer consists of two surface electrode coating of aluminum film with fixed ferroelectrics material film or pallets in side. By impose of voltage, pressures or temperature capacitance changes. The change in capacitance was used to vary the frequency of oscillators or to null of capacitance bridge. The signal derived from a capacitor was produced by the dynamic variable changing the...
electrical capacitances of transducer. The transducer is then acting as a variable capacitor. The tuning capacitance of the oscillator is made of the transducer that it is an integral part of circuit. Weinbridge type oscillator was made using IC 741.

Capacitive conditioning was done by schematic block diagram in fig. (3.8) which comprised of oscillator, frequency to voltage converter, then ripple eliminator, offset control and A/D converter.

Oscillator

The purpose of the oscillator in the capacitive transducer circuit was to provide circuitry to produce an alternating current that changes frequencies as the capacitances of the transducer changes. The transducer was part of the Weinbridge oscillator tuning circuit.

The main features of Op-Amp 741 IC are [67-69]

(1) It is most versatile used for precise, low level signal amplification where low noise low drift and accurate closed loop gain was required.
(2) The offset null capability,
(3) Low power consumption.
(4) Very high voltage gain
(5) Wide power supply range provide superior performance for a wide range of instrumentation applications feasibility

Other technical specifications are

* Low offset voltages - 75 μV
* Low offset voltage drift - 1.0 μV/°C
* Low bias current - ± 2.6 nano amp
* High open loop gain - 500 K
* High common mode rejection 110dB
Block Diagram for Measuring Capacitance of FE Transducer

Wein bridge Circuit → Driver Circuit → F/V Converter → ADC Channel → PC

Circuit Diagram for Measuring Capacitance of FE Transducer

Wein bridge Circuit

Driver Circuit

R

3.3KΩ

FE Transducer

CX

+Vcc

+15 V

7

R

50 KΩ pot at 24 KΩ

0.05 μF

-15 V

0.05 μF

BC187

BC188

Frequency Out

A

R

1KΩ

R

1KΩ

3.3 KΩ

3.3 KΩ

+15 V

15 V

Fig No. [3.8]
**Frequency to Voltage Converter**

IC 9400, which is basically voltage to frequency converter, is also used to generate an output voltage that is linearly proportional to the input frequency waveform represented in fig (3.9a).

The features of the IC 9400 Frequency to voltage converter includes [70-74]

* DC to 100 - KHz operation
* OP-amp output
* Programmable scale factor
* High input impedance (> 10 MΩ)
* Capability to accept any voltage wave shape.

The input frequency is applied to the (+) input of comparator (pin 11) since the comparator hysteresis voltage was ± 200 mV. The input signal amplitude must be greater than ± 200 mV in order to trip the comparator. For unipolar input signal, it can be converted to bipolar waveform by using the offset circuit. PIN diagram of 9400 with its principal connections is represented in fig.(3.9b).

Each time the input signal crosses zero in the negative direction, the output of the comparator goes low. Three micro seconds later the $C_{ref}$ charge / discharge control circuit is enabled, which instantaneously connect the reference capacitor $C_{ref}$ to the reference voltage $V_{ref}$. It is + 5 V. This action charges $C_{int}$ each time with a precise amount of voltage until the voltage across it can no longer increase. The charging path is through the output terminal of the op-amp, through $C_{int}$, through $C_{ref}$ and finally through $V_{ref}$ on the other hand, each time the input waveform crosses zero in the positive direction the output of the comparator switches high. This disables the $C_{ref}$ charge discharge control circuit and $C_{ref}$ is shorted out. However the voltage across $C_{int}$ is retained because the only discharge path for $C_{int}$ is through $R_{int}$, which is very large. The voltage across $C_{int}$ is the output voltage $V_o$ [75-79]
Circuit Diagram for Measuring Capacitance of F E Transducer

Fig No. [3.9]
Fig. (3.9a)

Fig. (3.9b)
The amount of ripple voltage on \( V_o \) is inversely proportional to \( C_{\text{int}} \) and the frequency of the input \( F_{\text{in}} \) at the comparators input causes a precise of charge \( (q = C_{\text{ref}} \times V_{\text{ref}}) \) to be dispensed into the of op-amp's summing junction. This charge in turns flow through the feedback resistor is generating voltage pulses at the output of the op-amp. A capacitor \( (C_{\text{int}}) \) across \( R_{\text{int}} \) averages these pulses into a DC voltage, which is linearly proportional to the input frequency. The circuit diagram is shown in fig. (3.10)

The output voltage is related to the input frequency \( F_{\text{in}} \) by the transfer equation

\[
V_{\text{out}} = (V_{\text{ref}} C_{\text{ref}} R_{\text{int}}) F_{\text{in}} \tag{3.4}
\]

The response time to change \( F_{\text{in}} \) is equal to \( (R_{\text{int}} \times C_{\text{int}}) \). The amount of ripple on \( V_{\text{out}} \) is inversely proportional to \( C_{\text{int}} \) and input frequency.

[75-77]

### 3.3 COMBINATION OF CIRCUIT FOR MEASUREMENT OF CAPACITANCE

Closed loop connection was made for characterizing \( C - V \) plot of transducers material films of PZT, PLZT, TGS, TGS\(_2\) and PVF\(_2\) with different dimensions. For time domain of different capacitance and applied voltage was obtained in following manner.

The Change in the polarization of a polar material with the applied changes the dielectric constant as follows

\[
E \propto \left( \frac{\partial P}{\partial E} \right) \tag{3.5}
\]

Where \( \varepsilon = \text{dielectric constant} \quad \frac{\partial P}{\partial E} = \text{Polarization change with change in the d.c. field} \)

For a known thickness and size of a capacitor the capacitance varies is proportional to the dielectric constant of the material with applied d.c field.
Circuit Diagram for Measuring Capacitance of F E Transducer

Frequency to Voltage Ripple Eliminator

Fig No. [3.10]
C-V characterization of PZT and PLZT thin films shows the variation of Capacitance with applied d.c. voltages. The main flow chart is represented in fig.(3.11).

User-friendly software had been developed in turboC in an interactive mode with following features.

(i) When the C - V data was taken for the first time for the OUT, a pilot route was executed which applied a voltage of ± 10V in the step of 100 mV across the device.

(ii) During the pilot routine upper and lower voltage limits, corresponding to a predefined maximum device current of ± 10 mA are determined by the system software and the same voltage limits are displayed at the end of the pilot routine.

(iii) Software prompts the uses to select from the two options viz. to store data with simultaneous display of C - V characteristic on the monitor.

(iv) Display to C - V characteristics of the already stored data.

(v) It also prompts the user to enter upper and lower voltage limits, to select suitable current range and to enter the voltage increment for the C - V run.

On the same manner without disturbing capacitance measurement circuit, temperature measurement circuit was connected to channel. No.12, the device under test was instrumented with calibrated thermo couple of K-type. The output of the thermocouple via an electronic reference point was connected to temperature measuring circuit with precision multimeter measurement. Circuit was connected to A/D converter on Add - on - card in computer for further procession of signal into computer. Heating circuit was switched ON. To set a temperature gradient of predefined temperature set was accordingly to multimeter reading. Temperature measurement was taken after attaining the steady state condition. The pilot routine was run to measure capacitance value time to time. Setting through variac was done for increment of temperature.
Flow chart for main programme for DAS

START

INITIALIZE PCL 812 CARDS

ACTIVATE CHANNEL SELECTION ALGORITHM

ACTIVATE DATA ACQUISITION AND STORAGE ALGORITHM

ACTIVATE INSTRUMENT SETTING ALGORITHM

DISPLAY RESULT IN GRAPHICS MODE

TIME DELAY AND WAIT

END

Fig. (3.11)
Range settings

There are three LED's connected for setting ranges. Before connecting the sample, one should be aware of changing range otherwise display will be faulty. First sample was checked on Wayne-Kerr Bridge to get idea of range. The instrument having three type of ranges one for micro, nano and pico farad range. Each pot should be selected.

Working of sample and hold circuit

The sample and hold circuit samples an input signal and holds on to its last sampled value until the input is sampled again. It can be achieved a significant reduction in size and improved performance by using specially designed sample and hold IC LF 398, which requires only an external storage capacitor. The time period of the sample and hold control voltage Vs during which the voltage across the capacitor is equal to the input voltage are called sample periods. The time when the voltage across the capacitor is constant is called hold periods. The output of OP-amp is usually observed during hold periods. [78]

3.4 SOFTWARE DEVELOPMENT

The flow chart of the developed software for temperature measurement circuit is shown in fig (3.12). The software written in C is versatile and user friendly. It has two modes one to monitor the voltage and other is to monitor and control the voltage both as prescribed by user. The system takes uses of capacitance in every second at various voltages set by user. While running the software also generate a data file. The hard copy of which can be had on printer. The data file can be processed in Microsoft Excel to obtain a graph of capacitance voltage characterization. The flow chart is in fig. (3.13) having measurement of capacitance.

Chapter III DEVELOPMENT OF DATA AQUISITION SYSTEM FOR FERROELECTRIC ELECTRONIC APPLICATION
FLOW CHART FOR TEMPERATURE MEASUREMENT THROUGH COMPUTER

START

MAIN MENU
1. TEMPERATURE MEASUREMENT

SET START TIME STOP TIME SCAN INT

MEASURE mV VALUE USING 12 BIT ADC CHANNEL NO. 12

CALCULATE TEMPERATURE OPEN A DATA FILE

STORE THE VALUE AND TIME

DISPLAY THE VALUE WAIT FOR SCAN INT

Current time < End Time

No

STOP

Fig. [3.12]
FLOW CHART FOR CAPACITANCE AND VOLTAGE MEASUREMENT AND CONTROL

START

MAIN MENU
1. CAPACITANCE MEASUREMENT
2. VOLTAGE M & C

CAPACITANCE MEASUREMENT SET START TIME STOP TIME

VOLTAGE MEASUREMENT SET LOW LIMIT SCAN INT, STARTING TIME & END TIME

MEASURE THE CAPACITANCE VALUE ON CHANNEL NO. 11 VOLTAGE VALUE

ON CHANNEL NO. 15

CALCULATE CAPACITANCE AND VOLTAGES

OPEN A DATA FILE AND STORE THE VALUE AND TIME

DISPLAY THE VALUE

WAIT FOR SCAN INT

On Next Page

Fig. [3.13]
GET START

WAIT FOR SCAN INT

IS DATA TRANSFER TO PC REQUIRED

YES -> TRANSFER THE SCANNING PARAMETER

NO -> NEXT SAMPLE TO BE SCANNED

YES -> SAMPLE TO BE SCANNED

NO -> STOP
The main programme has following important task.

(i). having four subroutines each for one parameter to be measured and stored like capacitance voltage, temperature and pressure.

(ii). each subroutine measure process parameter and store them in data file.

(iii). Voltage control subroutine takes the predefined voltage value and control it.

(iv). Stored data can be stored in hard disk. It can take in graphical mode to plot the data.

EXPERIMENTAL

Connection of Circuits and Evaluation of data

Circuit showed in various figure has been connected with capacitor in the sample holder and connection was made for measurement of capacitance and temperature. Temperature variation system is indigenously built on sample holder. By varying temperature from RT capacitance were recorded directly on computer via software supplied by Dynalog Company various data were obtained by temperature as sensor specifically mentioned in different sections.

For measuring capacitance while applying pressure following technique had been employed. Samples of PZT, PVF$_2$ were monitored on sample holder with pressure measurement circuit with computer interfaced to it. Pressure was varied by hand press connected to sensor it is also connected via circuit to computer shown in fig.(2.10) already discussed in section [2.3.4]. High pressure can be applied by this technique.

After application of higher pressure the variation of capacitance were measured by Wayne Kerr Bridge. The variation of pressure was from Zero to 25 tones per square cm. The pressure was applied in both short circuit and open circuit condition and was retained on the sample for more then one hour.
The measurement was done after taking out the sample from the dye. In situ measurement was done with the help of dye electrode but because of piezo charges the measurement could not be taken as reliable. The reported results are for pressure effect for high pressure. The thickness of sample was measured before and after the pressure treatment by precise micrometer. The difference was negligibly small.

Comparative study was done for application of lower and higher pressure for PZT and PVF$_2$ samples.

Voltage variation plot of capacitance was done for all samples. Two types of studies were done for evaluation of data for positive voltages for poled sample were increased in controlled manner and capacitance was measured under steady condition. For obtaining loop for memory studies voltage positive as well as negative both were applied on samples then variation of capacitance was measured.

3.5 Study of ferroelectric transducer materials morphology and structure

X-ray diffraction

Analysis of the diffraction patterns of pure PZT are shown in Fig (3.5.1). The sharp peaks are observed $2\theta = 40^\circ$, which confirm the crystallinity of pure PZT. The XRD data of PLZT with various compositions are shown in Fig (3.5.2) as the composition increases there is decrease in the intensity of the peaks, indicating an increase in the amorphous nature in the composition. The relative intensity of certain diffraction patterns peaks changed significantly with weight percentage of lanthanum when composites are compared to pure PZT at $2\theta = 30^\circ$ the crystallinity is found to decrease with addition of dopants due to bonding. This nature is a clear indication of the reduction in the crystalline phase in the complex at room temperature. All the films crystallize in the perovskite structure and the orientation is in (111) direction, which shows that the orientation follows that of the substrate. Different condition during deposition does not seem to affect the structure of the films. If no sharp
X-ray diffraction patterns for PZT (a) 65/35 (b) 40/60

Fig. (3.5.1) XRD of PZT

X-ray diffraction patterns for PLZT (a) 8/65/35 (b) 15/40/60 (c) 18/30/70

Fig. (3.5.2) XRD of PLZT
Fig. Infrared spectra (IR) for (A) PZT deposited on glass substrate (B) PZT deposited on Si substrate.

Fig. (3.5.3) IR of PZT

Fig. (3.5.4) IR of PLZT
peak patterns are observed they are indicative of completely amorphous nature. Due to this amorphous nature the mobility of the ions in the material is high.\[^{[79]}\]

For PZT and PLZT solid-state sol-gel technique exhibits good homogeneity, small particle size and formation of single-phase compounds with tetragonal structure. The sharp and single diffraction peaks of the polycrystalline compound indicate homogeneity and crystallization of the samples. All diffraction lines of samples showed tetragonal crystal structure of the compound at room temperature.

Removing small quantity of the film from the substrate did the characterizations of the thin films materials. Then it is powdered and samples were thoroughly mixed separately in Nujol and investigated in Perkin-Elmer spectrometer at Nagpur National Environmental Engineering Research Institute for PZT and PLZT, for their spectra. Fig. (3.5.3) is representing IR spectrum of PZT.

Fig(3.5.4) show the IR spectrum of PLZT. The absorption at 1787 cm\(^{-1}\) is due to lead stretching vibration. The absorption at 148 cm\(^{-1}\) and 1385 cm\(^{-1}\) are due to Zr-O in plane deformation. The absorption at 1982 cm\(^{-1}\) and 1232 cm\(^{-1}\) are due to Ti-O stretching. The photomicrograph of samples shows that the grains are uniformly distributed throughout the surface of sintered pellets. A microstructure comprising of polycrystalline grain separated from each other by well-defined boundaries i.e. grain boundary has been observed in the SEM. All the SEM photographs of PZT and PLZT films are shown in fig. (3.5.5 a, b & 3.5.6 a, b, c, d).

XRD of PVF\(_2\) is shown in Fig. (3.5.7). Figure clearly depicted unpoled and poled film at 25\(^{\circ}\)C that is showing 001 and 0100 sharp peaks.

The I.R. spectra of the PVF\(_2\) thin films are shown in fig (3.5.8). The absorption peaks or the shoulders appear in the IR spectra at around 400, 550 cm\(^{-1}\) which are characterized to be due to form III crystallites and those appearing at 610, 760, 870, 1070, 1150 and around 1380 cm\(^{-1}\) are due to form II
SEM picture of Polycrystalline PZT. 65/35

Fig. (3.5.5a) SEM of PZT

SEM picture of Polycrystalline PZT. 40/60

Fig. (3.5.5b) SEM of PZT

SEM picture of Polycrystalline PLZT. 15/40/60

Fig. (3.5.6a) SEM of PLZT
SEM picture of Polycrystalline PLZT.

Fig. (3.5.6b) SEM of PLZT

SEM picture of Polycrystalline PLZT.

Fig. (3.5.6c) SEM of PLZT

SEM picture of Polycrystalline PLZT.

Fig. (3.5.6d) SEM of PLZT
Fig. (3.5.7) XRD of PVF₂

Fig. (3.5.8) IR of PVF₂
crystallization of PVF$_2$ and hence these peaks confirms the deposition of PVF$_2$ material [80].

Good quality smooth and transparent films of desired polymers have been produced by solution casted spinning disc method. The characterization studies IR - spectra of PVF$_2$ films have ascertained the deposition of these materials. The porosity was checked by the polarizing microscope and then SEM study has further undertaken. The SEM photographs of these films are shown in fig (3.5.9). These photographs are established smooth surface with uniform device structure without any porosity or inhomogeneity for PVF$_2$ films as shown in photographs.

XRD studies of TGS and TGSe established the one short hydrogen bond length of 2.44Å as reported by Hoshino. This is the reason the group III is considered as the glycinium ion and the glycine group II as the zwitter ion. The study illustrate in figure in (3.5.12) and (3.5.14).

Removing small quantity of the film from the substrate did the characterizations of the thin films materials. Then it is powdered and samples were thoroughly mixed separately in Nujol and their spectra were recorded by a Perkin - Elmer spectrometer at Nagpur National Environmental Engineering Research institute for TGS, TGSe. Infrared spectra of TGS shown in Fig (3.5.13) has peaks at round 2050, 1600 1400, 1300, 1200, 1000, 600 and 400 cm$^{-1}$. The appearance of absorption peak at 2050 cm$^{-1}$ (strong and sharp) are due to Ammonia ions (NH$_3^+$) other peaks appear in symmetrical and asymmetrical rocking of CH$_2$ and C-O-C stretching regions. Absorption peaks around 1400 cm$^{-1}$ (strong and broad) may be due to co$^-$ and CH$_2$. The absorption peaks between 1150-1030 cm$^{-1}$ (broad and strong) at 600cm$^{-1}$ (Sharp and weak) and at 400cm$^{-1}$ (sharp and weak) confirms the deposition of SO$_4^{2-}$ ions and can be compared well with the IR spectra available in the literature [82].
Fig. (3.5.9) SEM of PVF\textsubscript{2}

Fig. (3.5.10) SEM TGS

Fig. (3.5.11) SEM TGS
Fig. (3.5.12) XRD of TGS

Fig. (3.5.13) IR of TGS
Triglycine Sulphate (TGSe)

Fig. (3.5.14) XRD of TGSe

Fig. (3.5.15) IR of TGSe
An infrared spectrum of TGSe is shown in Fig (3.5.15). For TGSe, the absorption peaks have been observed at around 2075, 1625, 1500, 1375, and 1125 and around 400 cm\(^{-1}\). The appearance of peaks at around 2075 cm\(^{-1}\) (strong and sharp) at 1625 cm\(^{-1}\) (strong and weak) are due to the presence of NH\(_3^+\) ions. The appearance of the absorption peak around 150 cm\(^{-1}\) (strong and sharp) and at 400 cm\(^{-1}\) confirms the presence of SeO\(_4\) ion and the at 1375 cm\(^{-1}\) (strong and broad) may be due to the presence of COO\(^-\) and NH\(_2\) SEM photograph of TGS and TGSe are shown in Fig. (3.5.10) and (3.5.11).[83]

### 3.6 Calibration of measurements

All circuits are connected one by one and calibrated by following standard method.

Temperature measurement circuit was connected for calibration studies using K-type thermocouple with digital mill-voltmeter was combined with it. The result observed has been tabulated in the following table. From the values shown in table [3.2] measurement of temperature is quite linear. The graphical variation of temperature measured and the output reading of millimeter shows linear relation over wide range of temperature measurement. The table 3.2 shows that the percentage of error is quite low.
Table 3.2
TEMPERATURE CALIBRATION AND ERROR ESTIMATION

<table>
<thead>
<tr>
<th>Temperature measured by mill voltmete $V_0$</th>
<th>Temperature known by calibrated curve $T_0$</th>
<th>Test circuit measurement $T$</th>
<th>Dynamic temperature $q=T-T_0$</th>
<th>Recovery factor $T/T_0$</th>
<th>% Error$(T-T_0)/T_0$</th>
</tr>
</thead>
<tbody>
<tr>
<td>.15</td>
<td>20</td>
<td>19.92</td>
<td>-0.08</td>
<td>0.996</td>
<td>0.4</td>
</tr>
<tr>
<td>.22</td>
<td>40</td>
<td>40.12</td>
<td>0.12</td>
<td>1.003</td>
<td>0.3</td>
</tr>
<tr>
<td>.24</td>
<td>60</td>
<td>59.23</td>
<td>-0.77</td>
<td>0.907</td>
<td>1.20</td>
</tr>
<tr>
<td>4</td>
<td>80</td>
<td>79.21</td>
<td>-0.79</td>
<td>0.990</td>
<td>1.63</td>
</tr>
<tr>
<td>5</td>
<td>100</td>
<td>99.99</td>
<td>-999</td>
<td>0.999</td>
<td>0.99</td>
</tr>
<tr>
<td>6.2</td>
<td>120</td>
<td>120.56</td>
<td>0.56</td>
<td>1.004</td>
<td>0.46</td>
</tr>
<tr>
<td>7.8</td>
<td>140</td>
<td>139.23</td>
<td>-0.77</td>
<td>0.994</td>
<td>0.55</td>
</tr>
<tr>
<td>9</td>
<td>160</td>
<td>159.98</td>
<td>-0.02</td>
<td>0.999</td>
<td>0.012</td>
</tr>
<tr>
<td>10</td>
<td>180</td>
<td>180.42</td>
<td>0.42</td>
<td>1.002</td>
<td>0.42</td>
</tr>
<tr>
<td>11.4</td>
<td>200</td>
<td>199.87</td>
<td>0.18</td>
<td>0.999</td>
<td>0.18</td>
</tr>
</tbody>
</table>

The experimental results for pressure measurement circuit have been tabulated in table [3.3].
PRESSURE CALIBRATION AND ERROR ESTIMATION

Table 3.3

<table>
<thead>
<tr>
<th>Pressure mm of Hg of water level $P_0$</th>
<th>Corresponding mill voltmeter reading $M_1$</th>
<th>Test circuit reading in mill volt $M_2$</th>
<th>Dynamic reading $M_1 - M_2$</th>
<th>Recovery factor $M_1/M_2$</th>
<th>$%$ Error $(M_1 - M_2)/M_1$</th>
</tr>
</thead>
<tbody>
<tr>
<td>50</td>
<td>50</td>
<td>49.92</td>
<td>0.08</td>
<td>0.998</td>
<td>0.161</td>
</tr>
<tr>
<td>100</td>
<td>100</td>
<td>99.98</td>
<td>0.02</td>
<td>0.999</td>
<td>0.02</td>
</tr>
<tr>
<td>150</td>
<td>150</td>
<td>150.21</td>
<td>-0.21</td>
<td>-1.001</td>
<td>0.143</td>
</tr>
<tr>
<td>200</td>
<td>200</td>
<td>199.85</td>
<td>0.15</td>
<td>0.999</td>
<td>0.071</td>
</tr>
<tr>
<td>250</td>
<td>250</td>
<td>249.89</td>
<td>0.11</td>
<td>0.799</td>
<td>0.044</td>
</tr>
<tr>
<td>300</td>
<td>300</td>
<td>300.15</td>
<td>-0.15</td>
<td>-1.000</td>
<td>0.05</td>
</tr>
<tr>
<td>350</td>
<td>350</td>
<td>349.99</td>
<td>0.01</td>
<td>0.874</td>
<td>0.002</td>
</tr>
<tr>
<td>400</td>
<td>400</td>
<td>399.87</td>
<td>0.13</td>
<td>0.999</td>
<td>0.032</td>
</tr>
<tr>
<td>450</td>
<td>450</td>
<td>449.52</td>
<td>0.48</td>
<td>0.998</td>
<td>0.102</td>
</tr>
<tr>
<td>500</td>
<td>450</td>
<td>449.67</td>
<td>0.33</td>
<td>0.999</td>
<td>0.037</td>
</tr>
<tr>
<td>550</td>
<td>450</td>
<td>449.83</td>
<td>0.17</td>
<td>0.999</td>
<td>0.004</td>
</tr>
<tr>
<td>600</td>
<td>450</td>
<td>449.98</td>
<td>0.02</td>
<td>0.999</td>
<td>0.002</td>
</tr>
</tbody>
</table>

First of all pressure shown by water level measured by manometer is displayed at first column of table. Direct milli-voltmeter was connected to pressure transducer. Test circuit reading of millivoltmeter is presented in third column. The percentage error in measurement was calculated shown by last column. It can be seen that the pressure measured by second and third column...
Calibration curve for measurement of capacitance

Change in capacitance in nanofarad

Fig. (3.6.1)

Temperature calibration Graph

Temperature in °C

Fig. (3.6.2)
Calibration curve for measurement of pressure

Change in pressure in cm of water column

Fig. (3.6.3)

Comparison of various Films used in study

Temperature in °C

Fig. (3.6.4)
closely agree which is indication that circuit has excellent performance. Again the pressure displayed by computer by this measurement system works quite satisfactorily.

Calibration, graph for change in pressure in mm of water column and change in pressure in millvolt corresponding to output of transducer (pressure sensor) is quite proportionate. This indicates that the device is accurate and appropriate for measurement of pressure. The system shows excellent linearity for pressure ranges 50 mm of water level to 1300 mm of water level.

The devices for high-pressure measurement were made as follows. Aluminum foil was taken as lower electrode and substrate. All films of TGS, TGSe, LATGS, PVF2, PZT and PLZT were deposited on it by spin disc coating method as discussed in section [2.2.1]. After annealing the device was kept for poling and then was ready for pressure measurements.

A digital circuit for measurement of temperature, pressure and capacitance is developed as shown schematically in figure. The graph shown in the Fig (3.6.1) describes the response of the measurement system. The graph depicts the variation of capacitor with respect to the output voltage. The output voltage here means the actual capacitance value measured in terms of frequency corresponding to the graph gives information about the efficiency of circuit. The linearity of graph confirms the efficiency of system. Which is clearly indicates proportionality in the value of microfarad as change in output of bridge measurement circuit.

The calibration curve (3.6.2) shows the change in temperature verses change in output of thermo -couple (k-type) temperature transducer in millvolt. Calibration curve shows the change in pressure verses change in output of transducer [pressure sensor] in mill-volt in fig (3.6.3). Further slopes show the fast response for lower portion. The next section contains comparison of various films used in the study in fig. (3.6.4). the graph clearly shows the variation according to their transition temperature. The range of temperature is 25° C to350° C. Cmax for TGS is 1300nf, for PVF2 1700nf and for PLZT it is 2000nf. The variation is large for PLZT and less for TGS film.
3.7 RESULT AND DISCUSSION

For the measurement of the film thickness the capacitance method was not employed because of uncertainty in dielectric constant of these films at room temperature in ferroelectrics phase. The uncertainty in dielectric constant arises due to variation in density of the film and random orientations of the crystallites on the surface of the film. We, therefore, measured the thickness of these ferroelectric films by the Japan make Dial Gauge (LC = 10 μ) for measuring thick films and Baker Mercer dial gauge (least count = 1 μm). Taking four or five measurements at different parts of film, the thickness was then averaged. The error in thickness measurement by this method is expected to be.

The area of the top electrode films was either 3x5 mm² or 4x4 mm² and measured accurately by a Vernier microscope. The ferroelectrics films were polycrystalline. Their structure was seen through cross polarizing microscope. The polarizing microscope estimated size of the crystallites (L.C. = 0.1 mm) approximately. Largest crystallite size seen was ~1mm length (area ~0.05 mm²) in TGS film of 10 μ thick.

The dimensions of the single crystal plate were accurately measured by dial gauge mentioned above. The samples were annealed adequately and properly in vacuum (~10⁻³ torr) for several hours prior to all electrical measurements.

The dielectric constant (ε), the dielectric loss tan (δ) and the A.C. conductivity of the samples were simultaneously measured as functions of temperature by Wayne_Kerr-Auto balance Bridge B6442 at 1591.5 Hz from room temperature to about 90°C. BPL Megohmmeter and Keithley Digital measured the dc conductivity in the same temperature range. The measurements were performed in heating and cooling cycle runs on poled and unpoled samples. For poled and unpoled samples the heating / cooling rate was generally about 1°C / min and about 0.1°C/ min. near phase transition.
temperature. The temperature was measured by precalibrated chromel-alumel thermocouple in conjunction with OSAW Vernier Potentiometer then connected to computer with a resolution of 0.25°C.

The capacitance measurement system with voltage control unit has been developed and fabricated indigenously. It has been successfully employed to obtain C-V characteristics of ferroelectrics interfacing silicon metal oxide material device. The device chosen for studies are from material tabulated in table (3.4). The selection of material based on PZT and PLZT are from hard ferroelectrics group, TGS and TGSe related material are from soft ferroelectrics group and PVF$_2$ from polymer ferroelectrics group was chosen for making devices. Numbers of capacitor of each material had been taken for study. The average value obtained can be considered for result calculation.

**Details of Film Structure Used in Study**

**Table 3.4**

<table>
<thead>
<tr>
<th>Device parameter</th>
<th>TGS</th>
<th>TGSe</th>
<th>LATGS</th>
<th>PZT</th>
<th>PVF$_2$</th>
<th>PLZT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness</td>
<td>8μm</td>
<td>4 μm</td>
<td>5 μm</td>
<td>9.0 μm</td>
<td>3.08 μm</td>
<td>9.4 μm</td>
</tr>
<tr>
<td>Grain size</td>
<td>812 μm</td>
<td>500 μm</td>
<td>842 μm</td>
<td>745 μm</td>
<td>745 μm</td>
<td>924 μm</td>
</tr>
<tr>
<td>Area</td>
<td>0.05 mm$^2$ x 1 mm</td>
<td>0.06 mm$^2$ x 1 mm</td>
<td>0.05 mm$^2$ x 1 mm</td>
<td>0.05 mm$^2$ x 1 mm</td>
<td>0.05 mm$^2$ x 1 mm</td>
<td>0.06 mm$^2$ x 1 mm</td>
</tr>
</tbody>
</table>
DETAILS OF FE DEVICE SAMPLE PARAMETERS

Table 3.5

<table>
<thead>
<tr>
<th>Device parameter</th>
<th>TGS</th>
<th>TGSe</th>
<th>TGSP</th>
<th>LATGS</th>
<th>PZT 1</th>
<th>PZT 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness</td>
<td>2.4 mm</td>
<td>1.8 mm</td>
<td>2.2 mm</td>
<td>2.01 mm</td>
<td>0.11 cm</td>
<td>0.241 cm</td>
</tr>
<tr>
<td>Area</td>
<td>0.73 cm²</td>
<td>1.13 cm²</td>
<td>1.25 cm²</td>
<td>1.36 cm²</td>
<td>1.942 cm</td>
<td>3.01 cm</td>
</tr>
<tr>
<td>Capacitance in air (C₀)</td>
<td>15.26 nF</td>
<td>16.4 nF</td>
<td>17.1 nF</td>
<td>17.36 nF</td>
<td>22.3 nF</td>
<td>24.5 nF</td>
</tr>
<tr>
<td>AC Conductivity (σac)</td>
<td>8.8 mS</td>
<td>6.6 mS</td>
<td>8.9 mS</td>
<td>8.2 mS</td>
<td>7.4 mS</td>
<td>8.7 mS</td>
</tr>
<tr>
<td>Initial capacitance (Cₛ)</td>
<td>5.8 μF</td>
<td>8.6 μF</td>
<td>6.2 μF</td>
<td>7.24 μF</td>
<td>42 nF</td>
<td>47 nF</td>
</tr>
<tr>
<td>Humidity of device</td>
<td>55-60%</td>
<td>53-60%</td>
<td>55-60%</td>
<td>55-60%</td>
<td>60%</td>
<td>60%</td>
</tr>
</tbody>
</table>
FE DEVICES SAMPLE PARAMETERS

Table 3.6

<table>
<thead>
<tr>
<th>Device parameter</th>
<th>PELLET DEVICE SAMPLES</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>PVF₂ I</td>
</tr>
<tr>
<td>Thickness</td>
<td>1.2mm</td>
</tr>
<tr>
<td>Radius</td>
<td>1.28cm</td>
</tr>
<tr>
<td>Area</td>
<td>5.147 cm²</td>
</tr>
<tr>
<td>Capacitance in air (Cₐ)</td>
<td>18.4 nF</td>
</tr>
<tr>
<td>AC Conductivity  (σac)</td>
<td>3.7 mS</td>
</tr>
<tr>
<td>Initial capacitance (Cₛ)</td>
<td>57.8 nF</td>
</tr>
<tr>
<td>Humidity of device</td>
<td>60%</td>
</tr>
</tbody>
</table>

3.7.1 TRANSDUCER CHARACTERIZATION OF PZT AND PLZT MATERIALS

Fabrication of PZT and PLZT MFS Device

The PZT and PLZT film has been deposited by solution casted spinning disc technique that has been described earlier and as developed by our research group. The solution of these material were slowly evaporated and after evaporation the temperature of the concentrated solution was maintained between 150°C to 200°C while the rotating substrate was heated up to 200°C. The speed of rotation of the substrate was set around 4000 rpm. Few drops of the solution were dropped at the center of the rotating substrate yielding a uniform and smooth texture of the desired thin film. Varying the
C-V Characteristics for PZT Film (MFS Device)

C-V Characteristics for PLZT Film (MFS Device)
concentration and the quantity of the solution dropped on rotating substrate can control the thickness of the film.[84]

**C-V characteristics**

The variation of capacitance of 5μ PZT film with bias voltage ranges from -30 to +30 volt and shown in fig (3.7.1.1). Due to ionic conduction the step increase of capacitance rise starts from +10 positive voltages with value starting from $11 \times 10^{-10}$ farad. Maximum value obtained for PZT capacitor is $60 \times 10^{-10}$ farad. Similarly the variation of capacitance of 15 μ PLZT film with different bias conditions ranges from 30 to +30 volt is shown in Fig.(3.7.1.2) the capacitance rises from $21 \times 10^{-10}$ farad and attains highest value up to $88 \times 10^{-10}$ farad.

The relaxor nature in the film was confirmed from diffuse phase transition together with the frequency dependent $T_m$. The C-V measurements were carried out at frequency 1MHz. The C-V curve was found to be asymmetric in nature owing to the different electrode material. Aluminum and silver have different work functions. The difference in the work functions leads to a change in the value of capacitance under positive and negative bias. The presence and sign of the immobile charges in the interface region may create the shift of hysteresis in that direction on applying the $+Ve$ or $-Ve$ voltage across the interface. In the $+Ve$ voltage region (around +5V), a flat band-shift suggests a depletion layer (for the majority carriers) and leads to the continuous fall of the capacitance (up to $-5V$), which remains constant on increasing further the negative voltage. The variation has been similar attributed to the electro-strictive (Change in the dimension due to electric field; a mechanical change) mechanical coupling with the dielectric constant.

A space charge layer found at the FE-SC interface behaves as the accumulation layer at $+Ve$ voltage side because the film has been deposited on the n-type Si wafer and it causes a maximum value of the capacitance ($C_{max}$) at the $+Ve$ voltage side. It was found to be around 60 pF. The minimum value
C-V characteristics of PZT thin film capacitor at room temperature

Fig. (3.7.1.3)

C-V characteristics of PLZT thin film capacitor annealed at 500°C for 30min in O₂

Fig. (3.7.1.4)
of the capacitance \( C_{\text{min}} \) was 11 pF corresponding to the \(-Ve\) supply of the bias voltage, that can be due to depletion layer contribution.

The C-V characteristic of the metal-ferroelectric-semiconductor (MFS) device employing a PZT film of 20\( \mu \)m and 15\( \mu \)m thickness prepared by the solution casting spinning disc technique is shown in Fig. (3.7.1.3). The device characteristic shows a vertical hysteresis in the C-V plot when a fast observation has been taken by changing the bias voltage in step of 5 V, from +20 to -20 volts and again from -20 to +20 volts. The estimated value of the dielectric constants was found to be around 1785 for the maximum value of the capacitance and it is found to decrease with decreasing thickness of the film. The asymmetries in curve and bell shape are confirming the perovskite structure. The value of capacitance is changing from 538 pf to 1614 pf.

The fig.(3.7.1.4) is showing C-V curve of PLZT of 22.5\( \mu \)f and 18\( \mu \)f films. The capacitance voltage plot shows that the existence of two distinct maxima is evidence for ferro-electricity rather then for a decrease with increasing dc bias. The decrease in the capacitance with increasing dc bias has been already observed for the cubic phase of \( \text{SrTiO}_3 \), \( \text{BaTiO}_3 \) and heavily La-doped \( \text{PbTiO}_3 \) material. [85] Therefore C-V plot shows bell like shape. The asymmetry in C-V curve was further increased after temperature imposed this may be due to the redistribution of domains. The value of capacitance is changing from 1160 pf to 2220 pf.

**C-T characteristics**

The plot between the capacitance and temperature studies is shown in fig. (3.7.1.5) there is rise in the values of capacitance up to transition temperature. After that there is subsequent fall in the value as the temperature increased due to the para-electric behavior. Here the capacitance value for PZT1 pellet is \( 30 \times 10^{-10} \) F at 200\(^0\) C. For next PZT2 material the value of capacitance is \( 14 \times 10^{-10} \) F at 200\(^0\) C that is transition temperature obtained for PZT2. Here the capacitance value for PLZT1 is \( 118 \times 10^{-10} \) F at 250\(^0\) C. For next
Capacitance vs Temperature plot for PZT and PLZT Pellets

Fig. (3.7.1.5)

Capacitance vs Temperature plot for PZT and PLZT films

Fig. (3.7.1.6)
PLZT2 material the value of capacitance is $133 \times 10^{-10}$ F at $225^0$ C, which is the transition temperature obtained for PLZT.

The room temperature capacitance reduces after peak subsequently. The value shifted to higher values with increase in frequency dispersion in both imaginary parts of and below transition region is a characteristic property of relaxor film. Like any normal ferroelectric the capacitance increases gradually with rise in temperature up to its maximum value $C_{\text{max}}$ and then decreases indicating the phase transition in PLZT between room temperature and $300^0$ C. The broadened peaks indicate that the transition in all the cases is of diffused type as important characteristic of a disorder perovskite structure. It is also seen that the dielectric peak shifts towards the high temperature side with increase in frequency.

Fig. (3.7.1.6) shows the plots of different samples of capacitances Vs temperature. It is seen that anomaly in capacitor near melting temperature occurs at different temperature for different samples. In pellets and microfilms the crystallites are oriented at random. Crystallites are closely packed in films than in pellets. Due to distinct differences in crystallites conditions in different samples the anomaly occurs at different temperature. Thus due to difference in sample preparation of different origin, relaxation being a function of crystalline condition, was obtained over a wide temperature region.

For 5μ film of PZT the maximum capacitance was obtained on temperature $200^0$ C and its value had been found $24 \times 10^{-10}$ farad similarly for second PZT film of 10.5μ the maximum capacitance value obtained was $38 \times 10^{-10}$ farad at temperature $205^0$ C.

Further study for PLZT samples shows the capacitance value from range $20 \times 10^{-10}$ farad to maximum value of $95 \times 10^{-10}$ farad at temperature $223^0$ C. This is phase transition temperature of PLZT1. For second sample of PLZT2 the maxima was obtained at $232^0$ C and value of capacitance was found to be $110 \times 10^{-10}$ farad.
Capacitance Vs High pressure for PZT and PLZT Films

Pressure in tonnes

![Graph 1: Capacitance Vs High pressure for PZT and PLZT Films](image)

Fig. (3.7.1.7)

Pressure (low) Vs Capacitance for PZT and PLZT Pellets

Pressure in Grams

![Graph 2: Pressure (low) Vs Capacitance for PZT and PLZT Pellets](image)

Fig. (3.7.1.8)
C-P characteristics

Pressure transducer was studied using the some PZT pellet as a pressure sensor material at room temperature, while humidity was changing 55-60% and the equivalent air capacitance was 15.2 nF. The ac conductivity of air was 8.8 mS and initial was 5.8 μF. Pressure was applied on samples differently. First the sample was kept in low pressure cell and readings were taken. Then sample was kept in high-pressure cell and readings were taken. Thirdly sample was in air atmosphere condition and weight bars were kept one by one for the studies of variation of capacitance by applying various forces. Further pressure transducer was studied by applying low and high pressure for PZT films and PLZT Pellets and Films. Similarly for high pressure the capacitance variation was for film PZTI 86 nF to 172 nF, and for PZT2 in the range 106 nF to 182 nF. This variation of high pressure is depicted in Fig. (3.7.1.7) For high pressure variation change from 2.5 tones to 12.5 tones per cm with constant increase while for higher pressure it is independent.

The variation of capacitance at low pressure is shown in fig. (3.7.1.8) for PZT and PLZT pellets. The first sample was PZT 5A showing sharp linear variation. The pressure was applied from 100 kg/m² to 1300 kg/m². The capacitance of PZT 5A is 8x10⁻¹⁰F at 100 kg/m² and the capacitance of PZT 5J is 15.2x10⁻¹⁰ F at 100 kg/m². The value capacitance of PLZ 5H is 21.6x10⁻¹⁰F at 100 kg/m² and the of value capacitance of PLZTI is 31.2x10⁻¹⁰F at 100 kg/m². In the entire graph values of capacitances increases with pressure with constant slope. This shows sensitivity of ferroelectric materials for pressure change as involves their basic piezoelectric property.

3.7.3 TRANSDUCER CHARACTERIZATION OF PVF₂ MATERIALS

PVF₂ sheet and powder were supplied by Pennwalt Chemical Corporation (UK) under the commercial name Kynar. Pellets were prepared from Kynar powder by hydraulic press at 3.6x10 Kg/cm² pressure. The other batch of films was obtained from Kureha Chemical Company Ltd. (Japan) under commercial name KF. These samples were uniaxially stretched, biaxially
C-V Characteristics for PVF$_2$ Film (MFS Device)

29 μ Poled film

112 nF

Capacitance in Pico-farad

Voltage

Fig. (3.7.2.1)

C-V characteristics of PVF$_2$ thin film capacitor at room temperature

25 μ film

29 μ film

Bias Voltage

Fig. (3.7.2.2)
stretched and poles piezo films of different thickness. Uniaxially stretched KF
films of thickness 9µ and 29µ were poled piezo films and coated both sides
with aluminum film electrodes. Biaxially stretched KF films of thickness 16µ
and 25 µ were unpoled. Al was vacuum deposited on both sides of samples. An
electrode area 0.05x1mm³ of was taken for measurements of 25µ films were
poled at 100°C with poling field 120 KV/cm for 60 minutes.

Fabrication of PVF₂ MFS Device

The PVF₂ polymer film has been deposited by solution
casted spinning disc technique that has been described earlier and as developed
by our research group.

We used Kynar product PVF₂ powder and to obtain their
saturated solution dissolved in solvents dimethyl formamide. The solution of
these material were slowly evaporated and after the evaporation the
temperature of the concentrated solution was maintained between 60 to 70°C
while the rotating substrate was heated up to 75°C. The speed of rotation of the
substrate was set around 4000 rpm. Few drops of the solution were dropped at
the center of the rotating substrate yielding a uniform transparent and smooth
texture of the desired polymer thin film. Varying the concentration and the
quantity of the solution dropped on rotating substrate one can the control
thickness of the film.

C-V characteristics

C-V characteristics are shown in fig (3.7.2.1). A retraceable
hysteresis in C-V plot was observed in figure by changing the bias voltage
from +25V to - 25V and again in range -25V to +25V, in step of 5 volts. An
accumulation layer appears at the positive voltage side. An equal band shifts
toward the positive and negative voltage side. Negative and positive side plot
looks like mirror image of each other. This is quite symmetrical in both
directions. The minimum capacitance for PVF₂ film is 58 nf and C_max is found
to be 92nf. All the justification for the memory behaviors is fall of the
Capacitance Vs temperature for PVF$_2$ Films

Temperature in °C

Capacitance in nano Farad

- PVF2 16 micron film
- PVF2 9 micron film
- PVF2 29 micron film

Fig. (3.7.2.3)

capacitance vs temperature poled PVF$_2$ films

Temperature in °C

Capacitance in Nanofarad

- 25 micron film
- 29 micron piezofilm

Fig. (3.7.2.4)
capacitance and rotation of the hysteresis curve. Higher doping density of n-type Si-wafer provides better compensation of the polarization. Fig.(3.7.2.2) plot of C-V for 29 μ poled film and 25 μ poled film for PVF₂ represent bell shape criteria. This confirms the ferroelectricity in PVF₂. The value of capacitance obtained for 25μfilm is more than 29μfilm. The range of capacitance is 279pf to 1629pf.

**C-T characteristics**

In fig (3.7.2.3) is shows the plot of variation of capacitance versus temperature. It is seen that anomaly in electrical conduction near melting temperature occurs at different temperature for different samples. In pellet and Kynar sheet the crystallites are oriented at random. Crystallites are more closely packet in sheet than in pellets. Stretching and poling have been suggested for crystalline transformation and orientation in PVF₂. Due to distinct different samples the anomaly occurs at different temperature. The relaxation depends on the crystallinity of the sample. The anomaly at high temperature may be due to dielectric dispersion in the pre-melting regions. The value of capacitance in pellets seems to be higher. The maxima of capacitance obtained at 109°C after that capacitance decreased a little while then again starts increasing at higher temperature. The maximum capacitance is 214nf. It is observed that increasing thickness of sheet the value of capacitance decreases. The value of 29μ biaxially stretched films show that when the field is increased conduction decreased and for higher temperature the decrease in capacitance is observed. For 9μ, 16μ and 29μ sheet the maximum capacitance values were found to be 188 nf, 130nF and 102 nF respectively. Molecular motion is set at relatively low temperature due to thermal agitation change.

The variations of capacitance of 29 μ piezo film and 25 μ biaxially stretched poled and unpoled films are reported in the graph Fig. (3.7.2.4) along- with their temperature dependence. The piezo film has high dielectric constant. Its capacitance varies from a value of 88nf at RT to 97nf at
130°C. From a careful look of these two graphs two dispersive regions can be deciphered around 60°C and 105°C respectively from the change in slope of capacitance. The dispersive region around 60°C is associated with earlier reported - relaxation in PVF₂ in case of solution-casted film. As inferred, at this temperature the dispersion is associated with molecular motion in the crystalline region and hence depends on the crystallinity of the sample. However, the change in slope in a capacitance variation of 29 μ piezo film at around 60 and 105°C is of much lesser magnitude than in the solution casted film, suggesting the crystallinity in the solution casted 25 μ film to be of lesser order than 29 μ piezo film. In both, 29 μ poled piezo film and 25 μ poled and unpoled biaxially stretched film the change in slope of the capacitance variation around 105°C again seems to be related with different sort of molecular motion other than the one responsible for α - relaxation. Alternatively, it denotes the temperature regain where this molecular motion drastically change.

The measurement in 25μ biaxially stretched films shows additional effect in the slope of poled and unpoled samples. In both, the dispersion in the region of relaxation is obvious from the change in the gradient around 55°C and 105°C. It is interesting to note here that the nature of the slope is opposite to each other in unpoled and poled sample plots at these temperatures. It is essentially an effect of ordering in the crystalline structure on poling. In the unpoled 25μ samples an additional change in the slope observed around 68°C is completely absent in the poled sample plot. The plot of loss mainly indicates for the 25 μ samples that the unpoled sample is lossier than the poled one particularly in the region of high temperature. The poled sample is more dispersive than the unpoled around 115°C.

It can be summarized that there are two regions of dielectric dispersion in 29 μ piezo film and 25 μ biaxially stretched poled film which are mainly due to different nature or changes in the molecular motion at the folded portion of the polymer.
Pressure (High) Vs capacitance for PVF₂ film

- PVF₂ 16 Micron film
- PVF₂ 25 Micron film

Pressure in tons

Fig. (3.7.2.5)

CAPACITANCE Vs PRESSURE FOR PVF₂ FILMS

- 9 micron PVF₂ film
- 15 micron PVF₂ film
- 29 micron PVF₂ film

Fig. (3.7.2.6)
C-P characteristics

Since the mutual transformation of the three morphologies of crystallites has been suggested also with an effect of pressure, it has been tried to investigate the effect of uniaxial pressure on the capacitance on 25 μ piezo film. The result is shown in Fig.(3.7.2.5) as the variation of capacitance with pressure. The value of capacitance of about 58.1 nf at room temperature with zero pressure reaches a value of 59.2 nf after applying a 25 ton uniform pressure on this sample. Two alternative interpretations of this observation can be suggested. Since the Form I crystalline phase is polar, the increase in dielectric constant is obvious. The nearly saturation region in the ε and tanδ value after 15 tones of pressure suggests that after this pressure the transformation to Form I crystalline is very small or negligible and so also change in dielectric constant and loss become negligible. Alternatively, it may be also due to piezoelectric effect that will release piezo charges at stress condition in the polymer matrix reaching a saturation value at 15 tonnes of pressure. These additional frozen-in charges can account for the initial increase in dielectric constant and loss both. It cannot be ruled out that the behavior shown in the graph depends on both the mechanisms playing their respective roles, howsoever small. The value of capacitance low varied for 16μ film from 58 nf to 59.8 nflow. The value of capacitance for 25μ film varied from 60.8 nf to 61 nf for 5 tones per cm pressure.

For application of low pressure from 100 Kg/m² to 1300Kg/m² shown in fig (3.7.2.6) for films of 9μ, 16μ and 29μ samples of PVF₂. The value capacitance of PVF₂ is 56.3 nf at 700 kg/m², for 29 μ film the value capacitance of PVF₂ is 47.3 nf at 700 kg./m² and for 16μ film the value capacitance of PVF₂ is 40 nf at 700 kg./m² for 9μ film. In the entire graph values of capacitances is increasing with pressure with constant slope. This shows sensitivity of material towards pressure and suitability for pressure sensor device of these ferroelectric PVF₂ films.
3.7.3 TRANSDUCER CHARACTERIZATION OF TGS AND RELATED MATERIAL

Triglycine sulphate (NH$_2$CH$_2$COOH)$_3$H$_2$SO$_4$ (TGS) is known as (TGS). It is crystallizes in the monoclinic structure with space group P2 in the ferroelectric phase with Curie temperature of 49$^\circ$C above that it transforms to the para electric phase with space group P2.[86]

There were two types of sample used for on studies. TGS, TGS (doped) and TGSe in pellets form and other in thin film form substrate is glass slides. For our study it was placed inside the vacuum chamber as described in Fig. (2.12). The electrical measurement like a.c. conductivity was studied using Wayne - Kerr-Auto Balance Bridge. Afterwards other studies like dielectric constant dielectric loss were measured. Various types of circuit like temperature measurement circuit and capacitance measurement circuit was connected with sample holder. Then pressure measurement circuit was connected and samples were placed inside pressure chamber.

Fabrication of TGS MFS Device

The C-V characteristic of the metal-ferroelectric-semiconductor (MFS) device employing a TGS film of 5 µm thickness was prepared by the solution casting spinning disc technique shown in Fig. (3.7.3.1 ). The device characteristic shows a verticle hysteresis in the C-V plot by changing the bias voltage in step of 5 V, from +30 to -30 volts and again from -30 to +30 volts. The direction of the hysteresis was found to be counter clockwise. The estimated value of the dielectric constants was found to be around 220 for the maximum value of the capacitance is found to decrease with decreasing thickness of the film.

A space charge layer found at the FE-SC interface behaves as the accumulation layer at +ve voltage side because the film has been deposited on the n-type Si wafer and it causes a maximum value of the capacitance ($C_{\text{max}}$) at the +ve voltage side. It was found to be around 75 pF. The minimum value of
C-V Characteristics for TGS Film (MFS Device)

Fig. (3.7.3.1)

C-V Characteristics for TGSE Film (MFS Device)

Fig. (3.7.3.2)
the capacitance \( (C_{\min}) \) was 40 pF corresponding to the –ve supply of the bias voltage, that can be due to depletion layer contribution.

The presence and sign of the immobile charges in the interface region side may create the shift of hysteresis in that direction on applying the +ve or –ve voltage across the interface. In the +ve voltage region (around +5V), a flat band-shift suggests a depletion layer (for the majority carriers), that leads to the continuous fall of the capacitance (upto –5V), which remains constant on increasing further the negative voltage.

The electron transfer process across the interface may also be associated in this observation with the ionic defects in the ferroelectric in addition to the presence of the domain structure.

The small flat band shift due to the shift of charge states located either in ferroelectric film or at semiconductor film side of the interface is also possible. However in the TGS film the presence of extrinsic carrier SO₄ ion is not ruled out because the crystals were grown in the acidic medium ph~4 and used for deposition of films, which has been pointed out earlier.

The presence of hysteresis in the capacitance voltage characteristics demonstrates the presence of memory behaviors in the TGS film. The presence of a space charge layer (either accumulation layer or depletion layer, which will depend on the nature of the applied voltage) at the film-semiconductor interface will also influence the real dielectric constant and the conductivity of the TGS film. Hence the depletion surface layer decreases the real dielectric constant of the film. The depletion capacitance \( C_D \) comes in series with the \( C_{\max} \) and can be calculated by using the following relation.[91-94]

\[
\frac{1}{C_{\min}} = \frac{1}{C_{\max}} + \frac{1}{C_D} \quad [3.5]
\]
By using the known values of $C_{\text{max}}$ and $C_{\text{min}}$, the depletion capacitance $C_D$ comes out to be 85.71 pF that is higher than the $C_{\text{max}}$ and hence will influence the real part of the dielectric constant and hence the measured part of the capacitance will be dominated by the $C_D$. Therefore the apparent part of the dielectric constant will be lower than its real value. The estimated value of the dielectric constant 220 is less than its measured real value around 254 in the present study. The dielectric constant of a ferroelectric film can also be influenced by the presence of a non-ferroelectric surface layer effect which may be presented over the ferroelectric thin film.

The occurrence of the C-V hysteresis and the direction of rotation of the hysteresis curve can be explained on the basis of ferroelectric field effect, because the sense of rotation of the curve is towards the direction of polarization. The memory behaviors observed by us is analogous to that which has been observed by Park and Granneman\textsuperscript{87} for BaTiO$_3$, Krupanidhi\textsuperscript{88} for Pb$_3$Ge$_3$O$_{11}$ film on n-type Si wafer and Matsui et al\textsuperscript{89} for PLZT film deposited on n-type Si substrate. But our result is contrary to what has been reported by Wu\textsuperscript{90} for Bi$_4$ Ti$_3$O$_{12}$ film on n-type Si wafer with respect to the direction of rotation of hysteresis loop. The C-V hysteresis observed by us and other workers in FE-SC (n-type) interface has the same sense of hysteresis rotation, i.e. the counter clockwise rotation.

**Fabrication of TGSe MFS Device**

In Fig.(3.7.3.2) the device characteristic curve of solution casted TGSe (4 μm) deposited on n-type Si wafer is shown. A retraceable hysteresis in the C-V plot was observed by changing the bias voltage from +30 to −30 V and again from −30 to +30 V, in the step of 5 volts. In the figure the C-V characteristic curve shows a similar anticlockwise path for rise and fall of capacitance as observed in case of TGS film-SC interface study.

An accumulation layer appears at the +Ve voltage side. The $C_{\text{max}}$ was found to be 87 pF and the $C_{\text{min}}$ as 42 pF. An equal flat band shift towards
the +Ve voltage as well as -Ve voltage side can be seen around + volts in the trace and retrace of the C-V characteristics. The equal shift in the flat band voltage suggests that both electron and hole are participating in the effect. For the trace and retrace at either side of the bias voltage, it is indicated that a contribution of depletion layer around +5 V a depletion layer appears for the majority carriers and around -5V, it appears for the minority carriers. This layer depends upon the nature of the charge carriers and applied voltage.

The depletion capacitance $C_D$ was estimated around 83 pF which is close to the $C_{\text{max}}$ value and hence it will decrease the real value of the dielectric constant found to be around 206 which is less than the reported value dielectric constant ($\sim 250$) of the TGSe film.

All the justifications for the memory behaviors, fall of the capacitance and rotation of the hysteresis curve, are similar to those for TGS as they belong to the same ferroelectric group of Triglycine sulphate family, where the selenate group substitutes the sulphate group in the crystal structure. TGSe remains ferroelectric at the room temperature and its transition temperature ($31^\circ C$) is lower than the TGS ($\sim 49^\circ C$). Hence by analogy, all the features and characteristics of the C-V plot for TGS are seen applicable in the case of TGSe. This study further substantiates the underlying principle of non-volatile memory property of ferroelectric-semiconductor interface as investigated in this study of the two members of the ferroelectric TGS family which are interfaced in the form of micron order thin film on n-type Si semiconductor

Fig. (3.7.3.3) shows the measurement of capacitance with respect to voltage variations. Voltage is bias voltage applied to the device in both negative and positive polarities, which is actually the bell shape of different bias curve is ascertaining the ferroelectricity in TGSe.

However, with reference to the TGSe film-semiconductor interface study, Batra et al. have suggested that the ferroelectric TGSe thin
Bias Voltage

C-V characteristics of TGS thin film capacitor at room temperature

Fig. (3.7.3.3)

Bias Voltage

C-V characteristics of TGSe thin film capacitor at room temperature

Fig. (3.7.3.4)
films are not useful for the nonvolatile memory devices because of their instability and also due to their depolarization effect. They found that these films are not unidomained and hence the observed depolarization effect neutralizes the polarization causing the bending of conduction and valence band and a degeneracy of the carrier concentration at the interface occurs. Hence the band bending at the ferroelectric-semiconductor interface will not exceed the band gap of the semi-conducting electrode. In another study they found that by utilizing a ferroelectric TGSe film between a metal and a Si electrode for a polarization direction requiring inversion, the minority carriers would need about 10 sec until they are in thermal equilibrium. It is very long time for any device application. In our technique however we could not supplement this conjecture.

However, in our study the depolarization effect and the instability at the surface of the TGSe film cannot be ruled out to some extent.

Although TGSe is ferroelectric at below room temperature and its Ferroelectricity is also retained in the thin film form, the ferroelectric properties in film form may not be as ideal as in bulk crystals. Though this study may have limited scope with respect to practical applications in MFS devices since the fabrication technique is not compatible with the Si-IC technology, but it establishes the memory capability in understanding the specific principle underlying the non-volatile memory devices, when a ferroelectric film is interfaced with a semiconductor.[96-99]

Fig. (3.7.3.4) is shows the measurement of capacitance with respect to voltage variations for TGSe film. Voltage is bias voltage applied to the device in both negative and positive polarities, which is actually counter value of DAC input to the circuit for 5µ and 2.5µ films. The counter value here is the decimal equivalent of value present in memory location that is proportional to the input voltage on device. The value of capacitance obtained
C-V characteristics of LATGS thin film capacitor at room temperature

Fig. (3.7.3.5)

Comparision of TGS and TGSe Pellets

Fig. (3.7.3.6)
for these films are in range of 42 pf to 90 pf. When thickness increases the curve shifted towards left side.

Similarly plot for LATGS thin film C-V characteristics in fig(3.7.3.5) for 2.5 \( \mu \) and 9\( \mu \) are showing same criteria except that its capacitance values are changing from 53 pf to 101 pf. Which indicates that doping is increases the value of capacitance.

The fig.(3.7.3.6) shows comparison of TGS and TGSe pellets that indicate capacitance increases up to 72°C. Anomaly occurs for TGS pellet at 120°C. After that capacitance decreases sharply. Anomaly occurs for TGSe at 60°C. The value of capacitance decreases again after 140°C it starts increasing. The Triglycine sulphate pellets of thickness 2.4 mm and area 0.73 cm\(^2\) was used for our studies. The value of equivalent air capacitance was 15 and 36 nf respectively. These studies were conducted with initial temperature from 30°C and humidity of sample at initially was between 55-60%. The ac conductance in air was 8.8 mS and the capacitance in air was 5.8 \( \mu \)F. In this section these material show their ferroelectric behavior near the room temperature (\( T_c \) for TGS is 49°C and \( T_c \) for TGSe is 22°C and \( T_c \) for LATGS is 51°C).

For further study of comparison of related films the plot in fig. (3.7.3.7) is describing the same thickness variation of capacitance with temperature. It clearly indicates the parallel lines with constant slopes. The doping in TGS enhances the value of capacitance in TGSe and LATGS. TGS 22.5\( \mu \) film and TGSe 12.5 \( \mu \) films of capacitance obtained maximum 160 nano-farad and 120 nano-farad are respectively for temperature variation 20°C to 170°C.

C-T characteristics

The capacitance versus temperature plot in fig(3.7.3.8) show the fall in the values of capacitance after transition temperature. This fall in the values can be justified due to the para-electric phase at Triglycine Selenate
Comparision of TGS, TGSe, LATGS Films

Capacitance Vs temperature plot for TGS films

Fig. (3.7.3.7)

Fig. (3.7.3.8)
Capacitance Vs pressure (High) for TGS and TGSe films

![Graph showing capacitance vs pressure for TGS and TGSe films.]

Fig. (3.7.3.9)

Capacitance Vs pressure (low) for TGS and TGSe Films

![Graph showing capacitance vs pressure for TGS and TGSe films.]

Fig. (3.7.3.10)
because $T_c$ of Triglycine Selenate is $22^\circ C$ it shows the para-electric behavior. It is also seen that lines are quite parallel reflecting the same internal activities during the rise in temperature. Dielectric studies were performed by using Triglycine sulphate doped Triglycine sulphate and Triglycine Selenate separately.

C-P characteristics

The C-P characterization variations of capacitance at different pressure were found for films of TGS and TGSe in fig. (3.7.3.9). For films 12$\mu$ TGS the capacitance variation from 136 nf to 188 nf for high pressure of 2.5 tones to 30 tones and for 20$\mu$ TGSe film the capacitance variation is 166 nf to 202nf respectively. The plot indicates the variation with constant slopes.

The next graph in fig. (3.7.3.10) is for 22.5$\mu$ TGS and 12$\mu$ TGSe and their change in capacitance for low pressure of 100 Kg/m$^2$ to 1300 Kg/m$^2$. The value of capacitance for TGS is increases constantly up to 1300Kg/m$^2$ from 86 nf to 172 nf for low pressure. The value of capacitance is changing from 106 nf to 182 nf for TGSe film.

We observe that for the potentiality of sensor or transducer devices amongst the pure ferroelectric material films PVF$_2$ has described by C-T characteristics. This is mainly because of its high dielectric strength for being a polymer. It is also for pyro devices having pyro-electric effect (92) which is quite substantial in poled and biaxial drawn films. This can enhance being a polymer and it will have properties suitable for all weather conditions. It shows good mechanical strength too in film form directly obtained from manufacture in different thickness.

The detail analysis of C-V characteristics C-P characteristics and C-T characteristics of reported for all studied materials PZT, PLZT, PVF$_2$, TGS, TGSe and LATGS. The characterization and Measurement reported by us ascertain proper and right choice of transducer materials for different applications. For successful instrumentation system desirable in measurement we have presented in this chapter the need of good methodology for installation of the sensor and signal conditioner and required range setting as necessary factors.
3.8 REFERENCE

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