CHAPTER III
EXPERIMENTAL TECHNIQUE
The general experimental set up, used for the investigation of the properties of thermo and radio electrets, is described in this chapter. The experimental set up used for the new method developed to study the behaviour of the electret, is described in the VI chapter.

III.1 Preparation of thermoelectrets

For electrets of fluorocarbon\(^+\) and mica\(^++\) the discs were cut from the commercially available sheets, of the required diameters.

For wax electrets, the carnauba\(^+++\) was melted, filtered and moulded in beakers. Discs of wax were obtained from these moulds. Plane parallel and polished faces were obtained by phasing the discs on the lathe.

The disc was sandwiched between two plane parallel aluminium electrodes. The diameter of the dielectric was kept larger than those of the electrodes in order to avoid any corona discharge from the edges of the electrodes.

\(^+\) FEP - Fluorocarbon film was obtained from Du-Pont de Nemours and Co. Ltd. (U.S.A.), having the trade name Teflon (\(\alpha\)) type A. This is a transparent film of thickness 0.025 cm. It can be bonded under heat and pressure to metals.

\(^++\) Thin sheets of natural muscovite mica were obtained from 'Electro mica Co.', Giridih (India). The sheets were transparent and colourless.

\(^+++\) Prime yellow and pale yellow carnauba wax were obtained from the Chemical Corporation of India, Limited, Bombay.
The sample was heated in an oven, the temperature of which was controlled by a thermostatic relay to within ±1°C. The forming temperature of these electrets (unless specified) was kept equal to 63°C in most of the cases, when the other parameters had to be studied. The constant temperature was attained in about two hours. Then the field was applied across the two electrodes. The temperature was kept constant for a further period of two hours after which it was cooled to the room temperature in about six hours and held at room temperature for 12 to 14 hours. The rate of cooling and the holding time under the field at room temperature were found to have negligible effect on the final behaviour of the electret (60).

The high voltage was obtained from a 40 kV transistORIZED generator of Cockroft and Walten type, shown schematically in Fig. 3.1 The experimental arrangement is shown in Fig. 3.2.

The charge density was always measured immediately after removing the electric field. The electret, thus prepared, was then wrapped in aluminium foil and kept between two heavy steel plates to ensure good contact. They were stored inside dessicators, since the humidity effect was found to be considerable in damaging the electret. It was observed that the condition of storing the electret plays an important role in the life of an electret.
FIG. 3-1 CIRCUIT DIAGRAM OF THE HIGH TENSION POWER SUPPLY

FIG. 3-2 PREPARATION OF THERMO ELECTRET
III.2 Preparation of radio electrets

The materials which were studied for their formation as thermoelectrets were also used for the formation into radioelectrets.

Ultraviolet rays and gamma rays were used to induce polarisation in these materials. Similarly, sandwiched samples were irradiated by penetrating radiations. The radiations were allowed to be incident on one of the surfaces (from +ve to -ve electrode) through the aluminium electrodes. The forming field was varied from 8 kV/cm to 300 kV/cm for different materials.

Samples were irradiated uniformly from a kilo curie $^{60}$Co source with a simultaneous application of the electric field. This arrangement is shown in Fig. 3.3. The dose rate was determined by ferrous sulphate dosimetry. The absorbed dose of any ionizing radiation is the energy imparted to matter by ionizing particles per unit mass of irradiated material at the place of interest. The unit of absorbed dose rate is the absorbed dose per unit time. In chemical dosimetry, the radiation dose is determined from the chemical change produced in a suitable substrate.

Ferrous sulphate dosimetry or Frick dosimetry, is the most widely used chemical dosimeter. The reaction involved is the oxidation of an acid solution of ferrous sulphate to the ferric salt in the presence of oxygen and under the influence of radiation.
FIG-3:3 Experimental arrangement for the formation of Radio electret.
To determine the absorbed dose (in 0.8 N sulphuric acid) a sample of the dosimeter solution was placed in the radiation field for a measured length of time, and the yield of ferric ions was measured, by the spectrophotometric analysis, comparing the optical density of the irradiated and non-irradiated dosimeter solutions at the wavelength at which ferric ions show maximum absorption (about 304 mµ or 3040 A°). The optical readings were taken soon after irradiation, so that the other oxidation of the solution is minimized. The mean absorbed dose for the volume occupied by the dosimeter solution was derived from the expression

\[
\text{Energy absorbed} = \frac{0.955 \times 10^9 (\text{O.D}_1 - \text{O.D}_0)}{\text{e.d.Q.} (\text{Fe}^{3+})} \text{ rads.}
\]

where \(\text{O.D}_1\) and \(\text{O.D}_0\) are the optical density of the irradiated and non-irradiated dosimeter solution respectively. \(\epsilon\) is the molar extinction coefficient for ferric ions at the wavelength of maximum absorption (litre/mole cm) and \(d\) is the thickness of sample used when measuring the optical density (cm). \(Q\) (product) is defined as

\[
Q (\text{product}) = \frac{\text{molecules product formed}}{100 \text{ ev of energy absorbed}}
\]

\(Q\) is the density of dosimeter solution. Substituting typical values for \(\epsilon\), \(Q\), and \(Q(\text{Fe}^{3+})\) (for Co\(^{60}\) gamma-rays) in the expression for energy absorbed, i.e. \(\epsilon = 2174\),
\[ q = 1.024 \text{, and } G(Fe^{3+}) = 15.5. \]

Energy absorbed = \( 2.80 \times 10^4 \frac{(\theta \cdot D_1 - \theta \cdot D_0)}{d} \) rads.

The maximum dose rate determined by this method was 70 rads/sec. The average dose rate obtained for most of the sample irradiated was \(~16\) rads/sec.

III.3 Methods of measuring charge of an electret

In the earliest work on thermo electrets, their charges were measured by electrostatic induction, following Eguchi's method. The charge induced on the upper electrode of an electret capacitor, was passed to an electrometer capacitor. Later this method was modified by Gross as dissectable capacitor method, and this method is still in use (17). The mechanism is shown in the schematic diagram in Fig.3.4.

Fei and Groetzinger (61) followed Eguchi's work, and measured the short circuit displacement current by thermally depolarizing the electret. The direction of the discharge current was opposite to the direction of the charging current. The total thermoelectret charge could be found by integrating the discharge current with respect to time and by passing it through a capacitor. Gross confirmed the equivalence of this result (17).

The temperature at which a thermoelectret is completely depolarized depends on the temperature at which its polarizaton was originally begun.

Binder (24) used the powder method for Litchenberg
Fig 3.4 Measurement of surface charge with dissectible capacitor. (C, parallel capacitor, V, electrostatic voltmeter, S, switch) (a) Polarized dielectric between short-circuited capacitor plates. The field of the dielectric induces image charges in the plates. (b) The switch has been opened and the upper electrode lifted. The induced charge of the electrode has been transferred to the parallel capacitor and charged it to voltage \( V \). (c) The switch has been closed again and the parallel capacitor discharged. Only the surface charges of the polarized dielectric are left.
figures, to study the homocharge, but this would only give a quantitative measure of the charge on the electret's surface.

Jevins and Reymolds (62) scanned the surface of the electret using a probe, measuring the charge with an electrometer.

Kojima and Kato (63) measured surface charge without touching the surface of the electret by a generating voltmeter.

Kallman and Rosenberger (4) used radiation discharge method, which is a non-repeatable process since it destroys the polarization.

Cross and Hart (30) suggested the measurements of the macroscopic dipole moments of an electret with a view to relating these to the surface charges of the electret.

Murphy (64) used a more sophisticated torsion balance automatic measuring system, by which he measured both conductivity and polarization of the electret.

Reedyk and Periman (65) suggested another method for measurement of surface charge, which consists in the generation of a signal by vibration of one of the electrodes of an electret condenser.

Gubkin et al. (66) and Pillai (67) have reviewed the methods of measuring the surface charge of an electret.
III.4 Present method of measuring the electret charge

Surface charge densities of thermoelectrets as well as photoelectrets were measured by the dynamic condenser system. Sessler and West (63) used this method to measure the surface charge density of the foil electrets in a microphone. The upper diaphragm was vibrated by the sound vibrations. Perlman and Needyk (65) developed a system based on the same principle to measure the surface charge of all their electrets (69).

This method consists in vibrating an electrode in the field of the electret. A signal is generated which was applied across the vibrating and fixed electrodes, of a variable magnitude sufficient to produce zero output signal. The surface charge of the electret is related to this bias voltage by the relation

\[ (\sigma_r - P_s) = K \varepsilon_0 V_B/d \]

(3.1)

It is obvious from the relation that the net surface charge \( (\sigma_r - P_s) \) so measured, is dependent on the dielectric constant of the substance \( K \), permittivity of the free space \( \varepsilon_0 \) and the thickness of the sample \( d \). It is independent of the air gap \( d_2 \).

Applying the line integral law to the two layer capacitor of Fig. 3.5

\[ d_1 E_1 + d_2 \varepsilon_2 = V \]

(3.2)
FIG. 3.6: ELECTRICAL CIRCUIT OF TWO LAYER CAPACITOR
where $d_1$ and $d_2$ are the thicknesses of the sample and gap. $E_1$ and $E_2$ are the electric fields in the sample and the gap respectively, $\Delta$ is the potential differences between the two plates. From Gauss's law

$$D_1 - D_2 = \sigma_r \tag{3.3}$$

where $D_1$ and $D_2$ are the electric displacements in the dielectric and the air gap respectively and $\sigma_r$ is the real charge on the surface of the sample. By definition of the displacement

$$D_1 = P_1 + \varepsilon_0 \frac{\partial}{\partial t} \tag{3.4}$$

where $P_1$ is the polarization factor for the sample, created by the applied field, $\varepsilon_0$ is the permittivity of free space. $P_1(t)$ can be expressed as

$$P_1(t) = P_i(t) + P_s(t) \tag{3.5}$$

where $P_s(t)$ is the component that responds slowly to changes in the internal field $j_1(t)$, and $P_i(t)$ is the component that responds practically instantaneously to changes in the internal field and can be characterized by dielectric constant $K$ i.e.

$$P_i(t) = \varepsilon_0 (K-1) \frac{\partial}{\partial t} \tag{3.6}$$

In the air gap, $P_s(t) = 0$. So combining equations (3.4), (3.5) and (3.6) we get

$$D_2 = \varepsilon_0 \frac{\partial}{\partial t} \tag{3.7}$$
and \( D_1 = K \epsilon_0 E_1 + P_s \) \(\text{(3.8)}\)

Finally combining (3.2), (3.3), (3.7) and (3.8), the field in the air gap is

\[
E_2 = \frac{KV - (\sigma_r - P_s) d_1}{\epsilon_0 (d_1 + kd_2)} \quad \text{(3.9)}
\]

where \((\sigma_r - P_s)\) is the net surface charge density. If now we apply a bias \(V = V_B\) so that \(E_2 = 0\) in the air gap, equation (3.9) yields

\[
(\sigma_r - P_s) = K \epsilon_0 V_B / d_1 \quad \text{(3.10)}
\]

Thus the net surface charge expressed by equation (3.10) is a function of the dielectric constant \(K\), the thickness of the sample and \(V_B\), the bias that produces zero field in the air gap. The measured value of \((\sigma_r - P_s)\) remains independent of the thickness of the air gap.

The schematic diagram in Fig. 3.7(a) shows the charge measuring device, employed in the present work and the photograph of the experimental arrangement is shown in Fig. 3.7(a). The electrode is vibrated with the help of an electromagnet excited by an alternating current of 50 Hz. The aluminium electrode, 8 cm in diameter provided with a guard ring, was held by a brass road, which in turn was screwed to a steel plate, kept below the electromagnet. The vibrational noise was reduced by keeping the steel
(a) Experimental arrangement of the charge measurement

(b) Charge measuring device

FIG - 3.7
disc over a ring of foam. The rod passed through two brass bushes. These provided guides for the rod in order to keep its vibration parallel to itself. The amplitude of vibration could be controlled by feeding the electromagnet through a dimmerstat. The frequency of vibration was 100 per second. Provision was made to vary the gap $d_2$. The lower electrode, insulated by a bakelite backplate, was supported by a movable stand. The lower electrode as well as the whole apparatus, were provided with levelling screws in order to obtain a parallel air gap between the two electrodes.

The signal was amplified by a transistorised, two stage AC coupled amplifier, and displayed on a Tektronix Oscilloscope model 585A. The bias was provided by a stabilized high tension unit providing a finely variable supply of 0 to 200 volts D.C. The signal was obtained across an impedance of 1 M.Ω. The photograph in Fig. 3 gives an impression of the signal, displayed on the oscilloscope.

The inherent disadvantage in the dissectible capacitor is that except for low surface charge densities, measurements must be made in vacuum to prevent discharges. The discharges in air arise because the field strength in the gap between the surface of the dielectric and the movable upper electrode can exceed the breakdown strength of the air as the electrode is raised for a measurement.

Another disadvantage is that charges are often generated
FIG-38 Display of signal on the oscilloscope
on account of the induction electrode and the dielectric. On the other hand in the present method, the upper electrode vibrates without touching the electret surface.

The present method is simple and non-destructive, and gives reliable results when compared with other techniques, and has few disadvantages. A voltage measurement and a few simple calculations yield the surface charge and its polarity with sufficient accuracy $\pm 2\%$. Measurements may be made at atmospheric pressure without fear of discharges, at a wide range of gaps and the surface charge is independent of electrical circuit parameters.

III.5 Sensitivity of the instrument

The charge density over the surface of the sample was measured by applying a biasing field equal to the field in the air gap. The accuracy with which a signal can be detected with this instrument is given by the least voltage of the bias which can bring about an appreciable change in the zero level line of the balanced signal, on the oscilloscope.

The least signal which was detectable on the oscilloscope corresponded to $V_B = 5$ volts; the instrument was insensitive below this value.

The percentage accuracy of the measurement depends upon the thickness of the sample, as well on the value of the
charge on the electret. For example a small detectable charge $\Delta q$ in the value of the surface charge $q$ amounts to a percentage accuracy of $\frac{\Delta q}{q} \times 100$. $\Delta q$ remains constant while $q$ changes with time for an electret.

The accuracy of the instrument ranges between $0.5\%$ to $2\%$ for the thinnest melinex foil of thickness $0.001$ cm. For example the accuracy with which signal could be detected was equal to $\pm 1$ volt in $50$ volts, amounting to a charge density of $142 \times 10^{-11}$ coul/cm$^2$ in $7125 \times 10^{-11}$ coul/cm$^2$ giving rise to a percentage accuracy within $\pm 2\%$.

On the other hand, the least count of $5$ volts of signal for the thickest electret of carnauba wax (1 cm thickness) amounts to a charge density of $0.955 \times 10^{-11}$ coul/cm$^2$ in a signal of $V_0 = 2,000$ volts corresponding to a charge density of $3360 \times 10^{-9}$ coul/cm$^2$ corresponding to a percentage accuracy of $0.25\%$.

**III.6 Thermal depolarization method**

Vibrating electrode method was used for measuring the surface charge of an electret, without destroying it. This method, however, does not serve the purpose of measuring the total volume polarization of an electret.

Method of thermal depolarization helps to look into the electret, and gives a fingerprint of the polarization, without the fear of dissipation of some of the charges due to imperfect shielding and other unfavourable conditions.
This is a most useful method with a limitation that the electret is destroyed. Thermal current spectra was used whenever it was required to analyse the nature of volume polarisation in the electret.

Thus the total charge of an electret was also calculated by measuring the thermal currents.

The electret was heated at a constant elevated temperature, usually equal to the forming temperature. Fig. shows the circuit diagram of the system. A vibrating re-electrometer (I.O.L. type 1880) was employed to record very low discharge currents. The sensitivity of this electrometer for measuring a current was $1 \times 10^{-13}$ ampere and the least count of the instrument to measure current was $2 \times 10^{-15}$ ampere.

III.7 Dielectric constant measurement

The dielectric constants of these electrets were measured before and after polarizing the sample. Marco circuit magnification meter was used for this purpose. The accuracy of the measurement was 0.2 pf, and the sensitivity of the instrument was $\pm 5$, in the range 40-100 kHz. To determine the accuracy of the measurements a dummy sample was used.

Fig. 3.10 shows the specially designed sample holder used for sandwitching the dielectric while measuring its dielectric constant. The movement of the upper electro
(a) Thermal depolarisation of electret

Electret
Electrode
Thermostat

(b) Equivalent circuit diagram

R₂ (Load)
E.M.F.

R₂ > R₁, V_d = i R₁

FIG-3.9 CIRCUIT DIAGRAM FOR DISCHARGE CURRENT MEASUREMENT
SAMPLE HOLDER CELL FOR CONDUCTIVITY & DIELECTRIC CONSTANT MEASUREMENT

FIG. 3.10
could be noted on the screw heads, the least count being 0.0006 cm. The diameter of the electrode was 8 cm.

III.8 X-ray diffraction studies

X-ray diffraction photographs were taken for samples in pre-excited and excited states of the dielectrets. The X-rays were incident along the direction of polarisation. The diffraction patterns were taken with a Philips X-ray unit having Tungsten target, operated in the L region of its spectrum, at voltages in the range 20-40 kV.

III.9 Measurement of conductivity

The conductivity of electret was measured by vibrating reed electrometer (I.J.L. Type 1880), which is capable of measuring currents of the order of $10^{-14}$ amp. The electrometer was operated with respect to earth as shown in Fig.3.11.

The voltage signal is considered as a source of e.m.f. symbolized by $e$ of negligible resistance, in series with a finite resistance - the internal resistance - which is referred to as $R_2$ (int) and symbolized by $R_2$. The input resistance being $R_1$, the current $i$, is given by the relation

$$i = \frac{e}{R_1 + R_2} \quad (3.11)$$

For the electret under consideration $R_1$ is small compared with $R_2$ hence
(a) Conductivity measurement

Electret

Electrometer

E.M.F.

(b) Equivalent circuit diagram

Load

$R_2 \gg R \quad \Rightarrow \quad i = \frac{e}{R_2} \quad \text{V.D. across } R_1 = iR_1$

$R_1 = 10^9 \Omega$

FIG-3.11 CIRCUIT DIAGRAMS FOR THE CONDUCTIVITY MEASUREMENTS.
i = \frac{e}{R_2} \quad (3.12)

and the voltage drop across $R$ is

\[ v.d. = i \cdot R_1 \quad (3.13) \]

That is, in the absence of feedback, the signal input departs from earth by this amount.

The stabilized voltage applied across the sample was obtained from the same electrometer.

The sample was sandwiched between two finely polished aluminium electrodes, and the distance between the electrodes could be noted. A guard ring was used to eliminate the fringing effect of the field.

The potential was increased in steps of 20 volts upto 200 volts and the corresponding current was observed. Subsequently the voltage was removed and the electret was shorted for about 10 minutes. The electret was then turned upside down, and the same procedure was repeated.

The surface of the electret in contact with the $+ve$ electrode, during polarization was termed as surface 'A' and the opposite surface was marked as surface 'B'. The current measured when the $+ve$ potential was applied to the surface 'A' is called the 'forward current' and that measured when surface 'J' is at higher potential with reference to 'A' is termed as reverse current.

The nomenclature is arbitrary, however, it indicated
the rectification properties of the electret. The measurements were taken with a view to test the non-linear behaviour of the electret. It could also test whether with the i-v characteristic the electret has any region of negative resistance.

The results obtained with thermoelectrets are described in Chapter IV.