CHAPTER II

APPARATUS, METHOD OF PREPARATION
AND

PRESERVATION OF ELECTRETS

1. Introduction.

Electrets are usually fabricated by the conventional method adopted by the pioneer worker Eguchi (1), by heating to melt and subsequent cooling of a suitable dielectric material while subjected to a high electric field. Although electrets can be prepared even at low temperatures, much below the melting point of the substance used, the magnitude of the charge and the permanency of the electret will be comparatively poorer to that of an electret prepared from the molten state (2, 3 and 4). Thus, for obtaining best results, electrets are prepared at or near about the melting point of the electret material. To this effect, most of the workers devised their own methods for preparing electrets at high temperatures. The important principle in the method of preparation of electrets has already been described briefly in Chapter I of this thesis.

Good and Stranathan (5) devised a method for making strong, smooth and uniform electrets. According to them the mode of mixing of the different components of the electret materials and the manner of cooling the mixture in the electric field have definite influences on the formation and subsequent behaviour of the electret.
Baumann and Wiseman (10) have designed a special type of dissectible capacitor inside a cylindrical oven in such a way that the dielectric could be melted, cooled with the electric field applied, and then measurements carried out, without removing the electret from the oven.

Majority of measurements of the electret strength have been confined to charge transfer method. The effective surface charge of the electret is transferred to a charge sensitive instrument such as the electrometer, electroscope or low capacity electrostatic voltmeter.

Eguchi (1) and Adams (6) have measured the surface charge of electret by means of an electrometer. Gemant (as reported by Gutmann (7) ) used the spark gap method to measure the magnitude of charge. Gross (9) studied the characteristics of electrets by means of a new method of analysis, which was based on the simultaneous measurement of the external current and of the induced charges on the capacitor containing the dielectric. For this purpose he devised the dissectible capacitor method. Some of the succeeding workers (8 & 10) found this method as a very suitable and effective device for electret measurement and utilised it in their investigations. Wieder and Kaufman (11) measured the charge density by means of electrostatic induction method using a commercial
electronic electrometer and a shunt capacitance. Kakiuchi (12) used a quadrant electrometer while Kojima and Kato (13) utilised a generating voltmeter for charge measurement. Froiman and Fridkin (14) used a lamp-electrometer connected by the method of constant deviation in their investigations of the heterocharge of carnauba wax electrets. Partington (15) and others measured the surface charge of electrets by using a Lindemann Electrometer. Freedman and Rosenthal (16) devised an apparatus capable of continuously measuring and recording electret strength. This apparatus serves as a completely automatic measuring system. Many others followed the charge transfer method for the measurement of charge of the electrets (17, 18, 19 & 20).

2. **Apparatus Used For The Preparation Of Electrets In The Present Investigations.**

In this laboratory Bhatnagar and Bhawalkar (22), Chandy and Bhawalkar (23) and later on Mathew and Bhawalkar (24) had prepared electrets by using a simple type of apparatus which was found suitable specially for the preparation of thicker electrets from melted substances. This apparatus mainly consisted of an assembly of two parallel copper electrodes, the connecting ends of which were bent at 90°. These electrodes were fitted to an ebonite lid which was placed over a pyrex beaker containing the melted electret material. An ebonite spacer with a
Figs. 1 & 2: Apparatus used for the preparation of electrets

Figs. 3 - 13: Showing different parts of the apparatus separately
Figs. 3-13: Showing different parts of the apparatus separately
slot in it was inserted in between the electrodes and the electret was formed in between the slot. Field was applied at the ends of the copper rods. When the wax solidified, the electret was taken out from the electrode assembly.

However, this apparatus was found to be unsuitable for the preparation of thin electrets of thicknesses of the order of 1.0 mm. or less without any cracks and other surface defects and irregularities. Hence, in the present work a different apparatus was designed and utilised for the preparation of electrets throughout these investigations. Figure (1) shows the side view of the apparatus. Figure (2) shows a three dimensional view of the same apparatus. The different parts of the apparatus have been shown separately in figures (3 to 13).

The apparatus essentially consisted of two parallel electrodes in between which the electret was prepared in a mica holder. The lower electrode was a plane copper sheet of size (7.5x5x0.15) cms. Three holes of 0.5 cm. diameter were drilled in the middle of three sides of this plate and on the centre of the fourth side a portion of 1.5 cm. square was cut (fig.3). This sheet was planed and polished. A tin foil of 0.001 cm. thickness and of the same size as that of
the above copper sheet was taken and four squares of 1.5 cm. size were cut from the middle of all the four sides (fig. 4). Actually this was used as the lower electrode. An electret holder of proper thickness was prepared with mica sheet which was also having the same dimensions as those of the lower electrode (fig. 5). The thickness of the mica sheet was adjusted by peeling off layers of mica sheets or by pasting thinner layers by means of suitable adhesive. In this mica sheet, a slot of 1 square cm. was cut exactly at the centre and at the middle of all the four sides holes were drilled as shown in fig. (5). The electret was moulded in the slot of this mica sheet which also served the purpose of insulation between the high voltage electrodes. This mica sheet was also used as a holder for the electret after its preparation. It was of uniform thickness and was plane, so that the molten material may not leak from the slot during preparation. A mica piece (7.5x5x0.2) cms. with a rectangular cavity (3.3x2.2) cms. in the centre was used for pressing the electret holder with the lower electrode (fig. 6).

A copper rod of 1.5 mm. diameter and 2.5 cms. in length was taken and welded at the centre of the upper surface of a plane copper piece (3x2x0.2) cms.
This was used as the upper electrode. The copper rod served the purpose of a holder for the upper electrode and also was used as the terminal for applying high voltage to this upper electrode. After cleaning and polishing, it was covered with rectangular tin foil (fig. 8). Particular care was taken to avoid air pockets in between the tin foil and the copper electrode.

A square piece of mica (1.5x1.5x0.2) cms. with a hole drilled at its centre was taken and inserted in the cavity of the lower electrode. Another mica strip of size (5x1.5x0.2) cms. with a hole at its centre was used as a support (fig. 10). A steel strip having the shape as shown in figure (11) was used for clamping the upper electrode tightly on the surface of the electret.

Assembling Of The Apparatus.

First of all, all the different parts of the apparatus were cleaned with benzene. The lower electrode was placed on a plane surface and the tin foil was pasted over this by pressing it with a glass rod. All the four squares at the sides of the tin foil were just above the holes of the copper electrode in this position. The mica holder was placed over the tin foil so that the drilled holes were in line with the lower electrode system. Thereafter the mica presser was kept over this electret
holder. One screw was inserted in the hole of the mica supporter to pass through the mica square fitted in the cavity of the lower copper electrode, the electret holder and the mica presser. In the same way three other screws of suitable lengths were inserted through the holes on the three other sides. The above assembly was tightened by nuts and bound together. The apparatus was now ready for the preparation of electret.


A small beaker (50 ml.) was taken and cleaned with benzene. A little quantity of the electret material was taken in the beaker and heated on an electric heater till it melted completely. The casket was also placed on the heater and allowed to reach a temperature two or three degrees above the melting point of the electret material. The casket thereafter was removed from the heater and kept on a plane surface. The cavity on the mica holder was rubbed with cotton in order to press the tin foil uniformly on to the copper sheet. This was essential to avoid any air trapped between the tin foil and the copper sheet, which would expand during heating and cause bulging of the electret. Small quantity of the molten mixture in the beaker was poured into the slot in the mica holder with the lower electrode as the support, care being taken to avoid any air bubbles getting into the liquid.
The upper electrode which was covered with tin foil was also placed on the heater on a clean mica piece. The temperature of the upper electrode was allowed to reach three degrees above the melting point of the electret material. The time required to reach this temperature was noted earlier by trial. When the electret material solidified, the heated upper electrode was placed over it very slowly, without applying any force. Some of the material was allowed to melt and the excess quantity was squeezed out on all the four sides of the upper electrode. In this condition the upper electrode touched the mica frame uniformly and hence the electret formed had a uniform and plane upper surface. The upper electrode was kept fixed in this position by means of a steel spring which was clamped to one of the screws as shown in figure (2). The thickness of the electret formed in this way in the mica slot was the same as that of the mica frame.

The casket was placed on a glass plate inside an oven. The oven was adjusted to give a constant temperature at which the electret was to be prepared. This polarising temperature was kept constant throughout the formation of electret. Two connecting wires, which were connected to a high voltage generator (in this case a 5 KV Ionisation Tester) were connected to the screws marked A and B in
figure (2). A was in contact with the upper electrode and B with the lower electrode. In all the cases in this investigation the positive terminal from the high voltage supply was connected to the upper electrode and negative terminal to the lower electrode of the system. This high voltage generator was giving constant voltage.

Sufficient time was allowed for the casket and electret material to attain the temperature of the oven. After reaching thermal equilibrium, the high voltage was applied across the terminals A and B for the desired polarising time. After a fixed time of keeping the sample in the above condition, the oven was switched off and the sample allowed to cool to 35°C, i.e. slightly above the room temperature. Then the high voltage was switched off and the casket was taken out from the oven after disconnecting it from the high voltage terminals. Immediately all the nuts and screws were taken out and the steel spring which was connected to the upper electrode was also removed. The mica presser was taken out by applying a little force. The upper electrode, after unfolding the tin foil from the sides of it was also removed. The tin foil from the upper surface of the electret could now be easily peeled off. After this, the mica holder with the electret was gently taken out from the lower electrode. While dismantling the electrode assembly,
Figs. 14 - 17: Showing the details of the method of preservation of electrot
maximum care was taken to avoid formation of cracks and scratches in the electret. Also a fixed time was always allowed before taking out the electret from the casket. It was kept immediately in a measuring device and the charge was measured by the method of induction using a Lindemann electrometer. The descriptions of all the instruments used in the preparation and measurement of electrets are given at the end of this chapter separately. The measuring procedure and the details of charge studies are given in the third chapter. After the first measurement, the electret was taken out from the measuring instrument and short-circuited with a tin foil and preserved in a desiccator as described below.


The necessity and importance of short-circuiting the electret and preserving it under controlled conditions are already described in the first chapter. The mode of preservation adopted in these investigations is as follows.

Immediately after taking out the electret from the electrode assembly, it was short-circuited with a piece of tin foil of size (13x3) cms. as shown in figures 14 & 15. The short-circuited electret was kept in between two ebonite sheets of the same size as the electret holder. Over the ebonite sheets, thin layers of cotton
were pasted for smoothness and to ensure better contact. A paraffin wax slab of moderate weight was placed over the upper ebonite sheet which helped to give better contact between the electret surfaces and the short-circuited tin foil. Afterwards the electret with the above described accessories was placed in a dessicator in which freshly dehydrated calcium chloride was kept. The dessicator was covered with a freshly greased lid and kept in a cool place for further studies.

5. **Instruments Used For The Preparation And Measurement Of Electrets.**

(1) **Wide Range Oven.**

For heating the samples and cooling them under controlled conditions, a wide range oven, manufactures by Baird & Tatlock (London) Ltd., was used throughout this work. Three heating circuits are incorporated in this oven which provide a temperature range up to 300°C which was sufficient for the purpose of this work. A special control is also provided with an arbitrary scale 0-100. Before its actual use a calibration chart was prepared so that the adjustment pointer could be set at any figure appropriate to the temperature required. In position No. 4, the desired temperature could be obtained very quickly also.
5kV Ionisation Tester
A red and a green lamp are provided with the oven. The green lamp signifies operation of the control. If the temperature rises beyond or goes below the setting temperature, the control would light up the red lamp thereby giving a warning. Hence slight fluctuations of temperatures can be adjusted by adjusting the knob in the appropriate direction. The temperature can be read by introducing a mercury thermometer inside the oven through a special device given on the upper wall of the oven. For adjusting little variations of temperatures, a hole and lid arrangement is also provided on the upper wall of the oven. By using this, sufficiently accurate and constant temperature can be obtained for a long time, variations being within about ± 0.5°C.

(ii) **High Voltage Supply.**

For getting high voltage in this work, a 5 KV Ionisation Tester, Type 732, supplied by Airmec Laboratories Ltd., Cressex, High Wycombe, Bucks, was used. This instrument was capable of giving D.C. voltage continuously variable from less than 250 volts to 5000 volts. By adjusting the voltage control, any desired voltage may be obtained for experimental work. This instrument operates from 200-250 volts 50 c/s main, the total power consumption being approximately 50 watts. The D.C. high
Fig. 18-a: Lindemann Electrometer
slotted on the top of the instrument. The whole device
voltage is produced by means of the R.F. type power pack and the order of output current is 5 microamperes. A constant voltage transformer is also used in the circuit for getting constant voltage and to avoid fluctuations.

(iii) **Lindemann Electrometer.**

Surface charge studies of electrets was one of the principal purposes in this investigation. The charge measurement was carried out by means of a Lindemann Electrometer manufactured by Cambridge Instrument Co., London.

Figure (18) shows the connections in the electrometer. The leads from the two sets of plates are carried out at one end insulated by silica tubes (A and B) and the lead from the needle is carried out at the other end (C). The earthing terminal D on the box helps eliminating the effects of stray electrostatic fields. The pneumatically operated earthing switch can be worked by pressing a small rubber bulb connected to the instrument by rubber tubing. A drying chamber E, containing a suitable drying material is slotted on the top of the instrument. The whole device is enclosed in a small metal box, in the centre of the top and bottom of which are holes about 10 mms. in diameter closed with microscopic cover slides.
Fig. 19-a: Charge Measuring Instrument
For use, this box is placed on the stand of an ordinary microscope of suitable magnification. The pointer at the upper end of the needle is observed by illuminating it through the hole in the bottom of the box. The graduation on the eye-piece scale was 1 division = 0.1 mm. This sensitive instrument had a capacity less than 2 cms. The leakage was practically nil in dry atmosphere which made the charge measurement possible. The details of setting the electrometer and the circuit diagram employed for the measurement of charge are given in chapter III.

(iv) Measuring Device.

In these investigations the charge measurement was carried out by the method of induction. For this purpose, a special measuring device was prepared to enable the transfer of charge from the surface of the electret to a moving electrode and then from it to an electrometer. The constructional details and the working of the measuring device are given below.

Figure (19) shows the cross-sectional view of this apparatus. It essentially consists of a fixed lower electrode on which the electret is placed to measure the charge, and another movable electrode
which is made to rest on the surface of the electret for a short period by a suitable mechanism. When the movable electrode is released, the charge which has been transferred to it from the electret surface, is immediately transferred to an electrometer by means of appropriate connections. The lower electrode is earthed.

The frame of this apparatus is prepared by using a hollow brass cylinder of 10 cms. outer diameter, 16 cms. in length and 4.0 mms. of wall thickness. Both sides of this cylinder are closed by plane brass sheets of 4.0 mms. thickness. At the lower side of this, a window of (6x7) cms. is made by cutting the brass cylinder, which is meant for inserting the electret in between the electrodes. The lower electrode is made by a circular brass disk of 5.0 cms. of diameter and 4.0 mms. thickness which is fitted to the bottom of the apparatus by rivets at the centre. The moving upper electrode has a surface area of 0.5 sq. cm. and has a stem 1.2 cm. long. This is screwed into an ebonite solid disk of 1.75 cms. diameter and 2.5 cms. length in order to insulate it from the rest of the instrument. On the other side of the ebonite cylinder a brass rod of 5.0 mms. diameter and 16.0 cms. length is screwed in. To enable the lifting of the
electrode, a special two spring mechanism is devised. These two springs are inserted on the brass rod which passes through a collared hollow brass cylinder which is fitted in a hole on the upper cover of the apparatus. The brass rod is capable of moving freely and the two steel springs are spreading on either side of the collared cylinder on the rod. The ends of these springs are also properly fitted to the rod by means of screws. A copper bracket with 0.8 mms. width and 12 cms. length, which is bent four times as shown in figure (19) is also inserted on the upper end of the rod above the spring. This is meant for pressing and lifting the upper electrode by means of pressing and releasing the spring system. Finally an ebonite solid cylinder is screwed at the end of the rod just above the bracket which serves the purpose of a handle.

For measuring the charge of the electret, the bracket is pressed and clamped by means of the clamping device fitted at the top of the cylindrical case. At this position, the upper electrode is in contact with the upper surface of the electret which is already resting on the earthed lower electrode of the measuring device. The upper electrode is also immediately earthed. After keeping the upper electrode like this for about
a minute, the bracket is released and the stronger spring pushes the upper electrode system upwards till the compression of the lower spring is enough to stop the upward motion of the rod. Thus the charge which has been transferred to the moving electrode from the electret surface is in turn communicated to an electrometer by suitable connections. The details of the charge measurements are given in chapter III.
References to Chapter II

(1) Eguchi, M.
    Phil. Mag.; 42; 198; (1925).

(2) Thiessen, P.A.;
    Winkel, A.; and Hermann, K.
    Physik Zeits.; 37, 511, (1936).

(3) Mikola, S.

(4) Groetzinger, G.; and Kretsch, H.
    Zeits. f. Physik; 103; 337 (1936).

(5) Good, W.M.; and Stranathan, J.D.

(6) Adams, E.P.;
    J. Franklin Inst.; 204, 469 (1927).

(7) Gutmann, F.

(8) Wiseman, G.G.; and Fester, G.R.

(9) Gross, B.

(10) Baumann, N.P.; and Wiseman, G.G.

(11) Wieder, H.H.; and Sol Kaufman;
    J. Appl. Phys.; 24, No. 2; (1953).

(12) Yoshinoba Kakiuchi;

(13) Shoji Kojima; and Kiyoe Kato;

(14) Froiman, A.I.; and Fridkin, V.M.
    Kristallografiya, 1(3), 342-50; (1956).

(15) Partington, J.R.; Planer, G.V.; and Boswell, I.I.
(16) Lorry A. Freeman; and Louis A. Rosenathalk,
(17) Bhadra, T.C.;
Ind. J. Physics; 32, No.6; 281-96 (1958).
(18) Vancalker, J and Vander Zinde, L;
Z. Phys; 155, No.4; 413-21; (1959).
(19) Seiwatz, H;
(20) Gross, B.;
(21) Groetzinger, G; and Kretch, H.;
Zeits. f. Physik; 103; 5-6; 337-349, (1936).
(22) Bhatnagar, C.S.;
Theesis accepted for Ph.D. degree by the
University of Sagar, Sagar (M.P.), India; (1956).
(23) Chandy, K.C.;
Theesis accepted for Ph.D. degree by the
University of Sagar, Sagar (M.P.), India; (1957).
(24) Mathew, A.C.;
Theesis accepted for Ph.D. degree by the
University of Sagar, Sagar (M.P.), India; (1960).