CHAPTER - 2

DESCRIPTION OF ACTUAL EXPERIMENTAL ARRANGEMENT AND MEASUREMENTS

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2.1 INTRODUCTION

Cerium dioxide, 99.99% pure, was obtained from Indian Rare Earths Limited, Bombay and films of different thicknesses were prepared by vacuum evaporation technique. For routine structural identification of the powder material used, X-ray diffraction was used. Electron microscopy was used for detailed structural characterisation of the film. Thickness of the film was measured by using Tolansky's interferometric method, and also by using quartz crystal monitor. The current voltage characteristics were obtained to identify the mechanism of conduction, while dielectric studies were carried out to get an idea of some relaxation mechanism. These studies are described in detail in Chapters 3, 4 and 5, while this chapter describes the actual arrangement used for experimental work in the present investigation.

2.2 FILM DEPOSITION BY VACUUM EVAPORATION

2.2.1 Basic Unit

In the present investigation, we have used 'vacuum evaporation technique' utilizing resistive heating mode.
Theoretical aspect of vacuum evaporation has already been dealt with in Chapter 1. We have used 'VICO' Vacuum Coater Model VC-12 for most of the depositions, while samples for low frequency measurements and for electron microscopy were fabricated using Hind HiVac Vacuum Coating Unit '19 F9'.

The coating unit consists of a mild steel cabinet upon which is mounted a cylindrical work chamber and control panel as shown in the photograph 2.1. Inside the cabinet, a rotary pump, a diffusion pump, control valve and electrical accessories are housed. Schematic lay out of the coating unit is shown in Figure 2.2 and described below.

The desired vacuum in the work chamber is obtained by the use of a diffusion pump. The diffusion pump by itself cannot evacuate the chamber unless proper fore-pressure and back pressure are established. To this end, rotary vacuum pumps are used. The pump used in the unit is a single stage oil immersion vane pump with air ballast, driven by an induction motor. When the pump is directly connected to the work chamber through the 'Roughing Valve', it establishes fore-pressure for the diffusion pump. And when it is connected to the diffusion pump through 'Backin Valve', it creates and maintains backing pressure. A magnetic isolation valve is included in the vacuum pipe line above the rotary pump. This valve isolates automatically the rotary pump from the rest of the system, thus preventing oil contamination, of the vacuum line during
FIG. 2.1 VACUUM COATING UNIT
Fig. 2.2 Schematic diagram of vacuum coating unit

1. Chamber, 2. Air admittance valve
3. Penning gauge, 4. Roughing valve
5. Pirani gauge, 6. Backing valve
power failure and also at the same time admite air into the pump to normalize the pressure inside the pump.

Fractionating type of three stage diffusion pump using silicon oil 704 has been used in the coating unit. When the oil is heated and vaporized, the vapour consists of light, medium and heavy fractions travelling upwards inside the jet system. The vapour is forced through the jet apertures and is deflected downwards by the jet deflectors. Gas from the vacuum chamber diffuses into the vapour stream and is carried away with it, thereby increasing in pressure as it proceeds from stage to stage, and finally, it is removed to the atmosphere by the Backing pump, while the oil vapour condensing on the water cooled pump walls drains to the boiler where it is re-evaporised. A water flow relay is used to prevent the over-heating of the pumping system and to maintain the desired rate of flow.

The metallic vacuum chamber is well polished on the inner surface and is provided with circular port holes with toughened glass windows at the top and on the side for visual observation of the coating process. The chamber is supported on a L-shaped neoprene gasket ring. A water cooled Baffle Valve isolates the work chamber from the diffusion pump and a fine control needle valve is provided for admitting air in the chamber.
The arrangement inside the vacuum chamber depends on the nature of coating application. For resistive heating, two electrodes carry basket/boat and are connected to a low voltage high amperage power supply. Generally the tungsten or molybdenum is used as the refractory material for the evaporating source.

Pressure measurement is done with the help of Pirani and Penning gauges. The Pirani gauge works on the principle, that heat conductivity of a gas in a chamber decreases linearly with the pressure in the chamber. Operational range is approximately $1 \text{ to } 10^{-3}$ torr and is generally used to measure fore-pressure and backing pressure. Penning gauge is a cold cathode ionization gauge which measures glow discharge current and is used to obtain a reliable indication of operating pressure inside the work chamber. It is calibrated to give the corresponding pressure. Its operational range is approximately $10^{-2}$ to $10^{-5}$ torr.

2.2.2 Evaporating Source

In the present investigation, we have used molybdenum boats and tungsten baskets (made of three strand wire). With the boat, the charge material cerium dioxide was in the form of powder itself, but when tungsten basket was used either of the two techniques were adopted.
(i) A paste of cerium dioxide was prepared using double distilled water, and then the basket filament was thoroughly impregnated with this paste with a previously cleaned fresh brush. Then it was mounted in the work chamber.

This technique was used for the preparation of films for (I-V) measurements, and dielectric measurements in the range $100 \text{ KHz}$ to $2 \text{ MHz}$.

(ii) Pellets of cerium dioxide were prepared in a previously ultrasonically cleaned (solvent acetone) die at a pressure of 10 tons. This pellet was placed in the basket. This technique was used for preparation of films for dielectric measurements in the low frequency range upto $100 \text{ KHz}$ and also for electron microscopy.

2.2.3 Substrate

The substrate influences the properties of the films deposited on them to a large extent (Holland, 1964) as already pointed out in Chapter 1. For polycrystalline films, pyrex glass and quartz substrates are commonly employed as they exhibit large smooth area. In the present investigation, we have used Blue Star super deluxe micro slides manufactured by Polar Industrial Corporation, Bombay. The slide is processed from optically flat microglass. Its dimensions are 75 mm length, 25 mm width and 1 mm thickness.
2.2.4 Substrate Cleaning

When preparing thin films on glass substrates, by vacuum evaporation, the cleanliness of the plate is of primary importance, as it greatly influences the film adhesion on the substrate. An excellent review on the subject is available due to Holland (1970). Various methods are available in the reported literature for the chemical cleaning of the substrates but with the advent of the modern detergents many of the early procedures based on drastic treatment of the glass surface with acids have become out of date. They can be readily cleaned by immersion in a detergent solution, and by utilizing ultrasonic cleaner with appropriate solvent. A well cleaned glass surface should show an uniform breath figure. In the present investigation an eight step procedure was adopted to ensure a clean surface.

(i) Glass slide was immersed in sodium hydroxide solution, rinsed with distilled water, and rubbed with fresh clean cotton wool.

(ii) It was, then, immersed in dilute nitric acid solution and again rinsed with distilled water, followed by rubbing with fresh clean cotton wool.

(iii) It was immersed in distilled water in which detergent 'Lab. wash' (E. Merck) was proportionately mixed and was ultrasonically cleaned.
(iv) Rinsed with hydrogen peroxide solution.

(v) Rinsed with benzene (AR grade).

(vi) It was immersed in acetone (AR grade) and ultrasonically cleaned.

(vii) It was rinsed with double distilled water.

(viii) Lastly the cleaned slides were dried in vacuum.

Finally cleaned glass substrates gave an uniform breath figure. The substrate was fixed in the substrate holder in the vacuum chamber for deposition and the chamber was sealed.

2.2.5 Masks

To prepare the experimental cell during a single pump down cycle, i.e., without breaking the vacuum, two movable masks as shown in Figures 2.3 and 2.4 were prepared. The mask was attached to a rotary vacuum seal, which could be rotated from outside and the mask set at the appropriate position between the evaporating source and the substrate. Position marked '1' was used for the deposition of base electrode (Al), '2' for the dielectric film and '3' for the upper electrode (Al). The finished form of the sandwich structure is as shown in Figures 2.3 and 2.4.

2.2.6 Preparation of Experimental Cell

(Al/CeO\textsubscript{2} film/Al)

The rotary pump was started after closing all the valves, for evacuating the inside of the pipe line, thereafter it was
Fig. 2-3 showing (A) mask and finished form of (B) thin film capacitor.
FIG 2.4 SHOWING (A) MASK AND FINISHED FORM OF (B) THIN FILM CAPACITOR.
connected to the work chamber through the roughing valve to achieve fore pressure ($\sim 10^{-2}$ torr) for the diffusion pump. When the desired fore pressure was established, roughing valve was closed and backing valve was opened and simultaneously diffusion pump was switched on. Water flow was adjusted for proper cooling. When the desired backing pressure ($\sim 10^{-2}$ torr) was achieved, diffusion pump was connected to the work chamber through the baffle valve. When the pressure inside the chamber stabilized in the range $10^{-6}$ torr, chamber was ready for deposition.

In order to have uniform deposition and at approximately desired steady pressure the charge material was initially degassed by heating it slowly in vacuo at a low temperature, well below the actual temperature of deposition.

The construction of the experimental cell was completed in three steps using an appropriate mask.

(i) A bottom layer of aluminium (99%) pure was evaporated onto the substrate.

(ii) A layer of dielectric material, cerium dioxide was deposited on the bottom layer.

(iii) A top layer of aluminium was evaporated over the dielectric to complete the experimental cell.
2.2.7 Annealing

The (Al/CeO₂ film/Al) cell assembly was annealed in vacuum for 3 hours at nearly 125°C. Then it was cooled and finally taken out of the vacuum chamber and placed in the measurement chamber on a small heater specially designed for the substrate.

2.3 HEATING ARRANGEMENT

Temperature is an important parameter and can be controlled by direct heating as well as by indirect heating. In some studies, direct heating of the specimen has been used for rapid determination of temperature variation of resistivity (Ehrenberg and Hirsch, 1951). For obtaining conductivity data and dielectric measurements in the frequency range 100 KHz to 2 MHz at different temperatures, we have used direct heating of the experimental cell. To this end, a small heater was designed as shown in Figure 2.5 and was prepared in the departmental workshop. It consists of two flat smooth and well polished metal plates of size 6 cm x 4 cm between which a 125 watt heating element is sandwiched. On the upper surface two metal blocks are fixed as shown in the figure forming a slot to hold the substrate. The heating element is connected to AC mains via a variac. The instrument can be calibrated in terms of current flowing through the filament and a steady substrate temperature established. This gives a fair approximation of the substrate temperature and is useful in adjusting
FIG 25  ILLUSTRATING DESIGN OF HEATER AND PLACEMENT OF CAPACITOR.
the temperature at the desired value. The actual temperature was measured by using a Chromel Alumel thermocouple.

For dielectric studies in the range 100 Hz to 100 KHz, the temperature was controlled by keeping the sample in an electric air oven.

2.4 MEASUREMENT CHAMBER

Diagramatic sketch of the measurement chamber is as shown in Figure 2.6. It consists of a smooth nickled steel base plate with three levelling screws. It supports a glass ball jar which is placed on it utilizing L-shaped neoprene gasket ring. Inside the chamber in a corner, there is an electric feed-through sealed to the base. Six thin metal rods insulated from each other pass through it. Both ends of the rod are threaded. They serve as electrical leads, two for film, two for heater and two for thermocouple. There is another small opening which is connected to a rotary pump via a vacuum stopper. In the centre of the base plate, a small centre table made from asbestos plate is positioned as shown in the diagram. Heating arrangement is placed on this table. The asbestos table has also an arrangement to clamp the thermocouple properly on the substrate.

2.5 LEAD CONNECTIONS TO THE ELECTRODES

For fixing connecting leads to the upper and lower aluminium electrodes, a thin aluminium foil was wrapped on the
FIG 26  MEASURING CHAMBER WITH SET UP FOR I-V MEASUREMENT
electrodes. Thick aluminium strips were also wrapped with aluminium foil. These two were, then, held firmly by crocodile clips. These gave satisfactory contacts for DC current voltage measurements, but were not satisfactory for dielectric relaxation measurement at higher frequencies. For obtaining rigid contacts, for these measurements, the following procedure developed by Chaturvedi et al was adopted. The various steps are detailed below and are shown in Figure 2.7.

(i) Clean glass slides were drilled at desired places by an IMECO ultrasonic drill.

(ii) Glass slides were cleaned by adopting steps (i) to (iii) of the cleaning procedure given earlier and then conical holes were etched using electronic grade hydrofluoric acid.

(iii) Slides were cleaned following the steps (iii) to (vii) of the cleaning procedure. For contact leads, 30 SWG enamelled copper wire was used and was cemented using silver paste after removing enamel from the contacting end.

(iv) The pasted contacts were dried at 150°C in an oven for about 10 hours. The substrate was cleaned following steps (iii) to (viii).

(v) Bottom electrode was deposited using mask position 1 (Fig.2.4).

(vi) Cerium dioxide film was deposited using mask position 2.
CLEAN GLASS SLIDE

ULTRASONIC DRILLING OF HOLES
CLEANING AND HOLE ETCHING

LEAD BONDING
VACUUM DRYING
CLEANING FOR DEPOSITION

ELECTRODE DEPOSITION
(USING MASK OPENING - 1)

ALUMINUM FILM

DIELECTRIC FILM DEPOSITION
(USING MASK OPENING - 2)

ELECTRODE DEPOSITION
(USING MASK OPENING - 3)

FINISHED CELL ASSEMBLY

FIG 27 PREPARATION OF EXPERIMENTAL CELL FOR DIELECTRIC STUDIES.
(vii) Upper electrode was deposited using mask position 3.

Mask positions were so adjusted that on metallisation bonded leads got connected with the respective electrodes. The contacts prepared by this method are claimed to give contact resistance typically less than 0.01 ohms. They are ohmic in nature and mechanically very strong.

2.6 (I-V) MEASUREMENT

Many excellent accounts of various measuring techniques for obtaining conductivity data are available at present and reference may be made to Geiger and Scheel (1927), Meissner (1936), Becker and Horovitz (1952), Gerritsen (1956) etc. Main factors affecting the suitability of various methods and precision attainable include contact resistance and form of sample, i.e., whether in the form of single crystal, film, powder, or small crystallites. The simplest method and also widely used is the 'Voltmeter-ammeter' method which follows from the definition of conductivity. Direct measurement of the current and voltage together with accurate measurements of the dimension are needed to determine the conductivity. In the present investigation, we have used this method utilizing the circuit shown in Figure 2.8.

We could have connected the voltmeter across the sample itself but then it was found to interfere with the current measurements. This circuit ensures that we are measuring that
FIG. 2.8  CIRCUIT FOR I-V MEASUREMENT.
part of the current which is flowing through the sample and not that current which is sum of the currents flowing through the sample and that flowing through the voltmeter. Specification of the instruments were so chosen that in either way of connecting the voltmeter, its reading was practically unaffected.

Equipment actually used in the experimental set up is shown in the photograph 2.9. Transistorised power supply type 6/2 manufactured by Systronics was used for stabilised voltages. It gives a highly stable DC supply, the output of which can be varied from zero to 30 volts at a maximum current of 500 ma. The output voltage is covered in 3 overlapping ranges with fine continuous adjustment over each range. Front panel meter is provided to monitor the output voltage setting. Impedence claimed is less than 0.05 ohms upto 1 KΩ/s.

For voltage measurements three meters were used, Phillips DC Microvoltmeter (PP 9004), Electrometer Amplifier EA 815 (manufactured by Electronics Corporation of India, Hyderabad) and Motwane multimeter. Motwane multimeter was used for measuring thermo e.m.f. for recording the temperature of the substrate. Phillips micro voltmeter was used to adjust the rheostat’s movable point to desired voltage so that required voltage across the film was applied when the film was put in circuit. The instrument has automatic indication of polarity of test voltages. The range carrying an input impedance of 100 MΩ was used. Electrometer amplifier was used to
FIG. 2.9 EXPERIMENTAL SET UP FOR I-V MEASUREMENT
measure the potential drop across the film. It measures very small direct current and low DC potential from high impedance source. It has got voltage ranges 10 mV to 10 V of both polarity. Input resistances $10^6$, $10^8$, $10^{10}$ and $10^{12}$ ohms are selectable by the switch provided. For voltage measurement, there is one open position which provides input impedance of $10^{14}$ ohms. We have used this position.

Current was normally measured using Phillips digital VA-$\Omega$-meter (PM 2522). Five current ranges are provided, viz., 0.2, 2, 20, 200 and 2000 ma range. For samples in which the current flowing was very small and could not be measured by the above multimeter, we used Toshniwal polyflex galvanometer, whose sensitivity is $5 \times 10^{-9}$ amperes. Before using it, we calibrated it.

As shown in Figure 2.8 the variation in potential drop across the experimental cell was done by using a potentiometric arrangement. Gradually the applied potential was increased and the corresponding current was recorded. To minimise the polarisation of the specimen, a tap key was used so that the circuit could only be completed at the time of taking observations. After taking room temperature observations (I-V) readings were obtained at different temperatures. Measurements were taken when the substrate attained the desired steady temperature. The temperature was measured using Chromel-Alumel thermocouple.
During all the measurements the rotary pump connected to the measurement chamber, containing the sample, was kept running to avoid the effects due to moisture and other atmospheric effects.

2.7 DIELECTRIC STUDIES

The capacitance or complex admittance of a dielectric specimen may be measured in a variety of ways by appropriate AC bridges. A complete survey of AC bridges and their particular fields of usefulness has been given by Hague (1946). The generally preferred method in a wide range of frequencies is based on the null-balance principle.

2.7.1 AC Bridges

The commonest type of AC bridge circuit is the four arm impedance bridge - a generalisation of the Wheatstone's bridge which is a particular type of impedance bridge where all four arms are pure resistances. The basis of calculation is the application of Kirchhoff's law. The bridge is balanced when the voltage appearing at the two points, across which the detector is connected, becomes equal both in magnitude and in phase. Thus for the circuit shown in Figure 2.10, we have two balance conditions which must be satisfied simultaneously. They are

\[ Z_1 Z_3 = Z_2 Z_4 \quad \text{for magnitude balance} \]
\[ \phi_1 + \phi_3 = \phi_2 + \phi_4 \quad \text{for phase angle balance} \]
Figure 2.10: Impedance Bridge Circuit

Figure 2.11: Schering Bridge Circuit

Figure 2.12: Transformer Bridge Circuit
Thus the unknown may be measured in terms of known impedance of the other three branches. Generally two arms are kept fixed and hence at the balance point, the unknown \( Z \) can be expressed as

\[
Z = A + jB
\]

one of the adjustable elements appears in \( A \) but not in \( B \), the other appears in \( B \) but not in \( A \).

Since the impedance containing each arm of an AC bridge may be a combination of resistances, inductances and capacitances, a great variety of bridge types is possible.

2.7.2 Schering Bridge

The Schering bridge shown in Figure 2.11 is extensively used for measuring the capacitance and the dielectric loss of condensers. For a fixed ratio \( R_2/R_1 \) of the resistance arms of the Schering bridge, the unknown capacitance is directly proportional to the standard capacitance \( C_3 \), thereby permitting \( C_3 \) condenser dial to be calibrated directly in terms of the unknown capacitance irrespective of the losses. At the same time, the resistance/conductance of the unknown condenser is determined by the value of capacitance \( C_2 \) required to achieve balance. Hence \( C_2 \) condenser dial can be directly calibrated in terms of resistance/conductance of the sample.
In the Schering bridge either the generator or the detector must be connected through a transformer with specially designed internal shielding. The shielding in the transformer forms part of a complete system of shielding which is an important feature of any properly designed Schering bridge.

2.7.3 Transformer Ratio Arm Bridge

If the bridge coupling transformer is provided with a centre tap on one of its windings, it is possible to eliminate two of the ratio arms of the Schering bridge. A variety of transformer bridges are described in the literature and reference may be made to Young (1946), Clark and Vanderlyn (1949), Cole and Gross (1949), Wilhelm (1952) etc. A schematic diagram of a simple transformer bridge is shown in the Figure 2.12. The bridge has two arms of the conventional type and the transformer contains the other two arms. The capacitance and conductance of the dielectric specimen may be measured with this bridge by using the full substitution method. In this method, the circuit is balanced by means of $C_N$ and $R_N$. The resulting expressions for the capacitance and resistance are

$$\begin{align*}
\frac{1}{R_x} &= \frac{1}{R_N} - \frac{1}{R_N} \\
C_x &= C_N^1 - C_N
\end{align*}$$

\[ (2.3) \]

The primes indicate the balance setting without the dielectric specimen.
2.7.4 Hewlett Packard Multi Frequency LCR Meter 4276A

In the present investigation we have used this bridge for the measurement in the range 100 Hz to 100 kHz. The equipment which is a recent addition, is an impedance measuring instrument, based on micro-processors and utilizes guarded four pair configuration measurement technique. It measures the value of the component and/or device under test at the frequency test signal level and do bias level at which they will be operated in the actual circuit. The instrument provides multiparameter measurements and gives a wide selection of 11 parameters for more accurate evaluation of electronic materials or components with high measurement speed for most needed combined parameters. They are:

(i) Inductance L
(ii) Capacitance C
(iii) Resistance R
(iv) Impedance Z
(v) Dissipation D
(vi) Quality factor Q
(vii) Equivalent series resistance ESR
(viii) Conductance G
(ix) Phase angle θ
(x) Variations in L, C, R, Z
(xi) Percentage variations in L, C, R, Z

Operation of push buttons (refer Figure 2.13) and other characteristics are detailed below.

(1) The instrument is properly earthed and a warm up time of half-an-hour is given to it.
FIG 213  HEWLETT PACKARD 4274 A MULTI FREQUENCY LCR METER
(2) Device is connected in the unknown arm at the appropriate terminals using appropriate test fixtures. We have used Tweezer type test fixture (MO 16034 B) which is used for three terminal configuration measurement. The device is properly earthed. One end of the lead is disconnected from the instrument.

(3) Self-test button is pushed. This functionally verifies proper operation of both the analog and digital circuitry, but it does not check instrument's calibration accuracy.

(4) Button marked ZERO is pushed to compensate for the errors due to self or mutual inductance, stray capacitance and/or residual inductance in the test leads or test fixtures used. The normalization range is as detailed below:

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Range</th>
</tr>
</thead>
<tbody>
<tr>
<td>Inductance</td>
<td>upto 2000 mH</td>
</tr>
<tr>
<td>Capacitance</td>
<td>upto 20 μF</td>
</tr>
<tr>
<td>Resistance</td>
<td>upto 0.5 Ω</td>
</tr>
</tbody>
</table>

Now the disconnected lead is again connected.

(5) Ranging and circuit mode selection is normally accomplished automatically. However, both range and circuit mode may be selected manually. There are three push buttons for circuit mode. One when the specimen is measured in terms of its series components and the other when it is measured in terms of its parallel components. Third push button is 'auto', which we have used. This selects the appropriate range by itself.
(6) Proper frequency is chosen by frequency step buttons. The instrument covers the wide frequency range of 100 Hz to 100 KHz in 11 spot frequencies in 1-2-4-steps as shown:

- 100, 120, 200, 400 Hz
- 1K, 2K, 4K Hz
- 10K, 20K, 40K Hz
- and 100K Hz

(7) The voltage or current values are then monitored on the 3 digit display for accurately setting the actual conditions under which the device under test will operate. Test signal level can be adjusted from 1 mV to 5 volt rms. In the present work, we have used 2 mV signal.

(8) Measurement ranges for the various components are as follows:

- L : 100 nH to 1000 H
- C : 1.0 pF to 1.0 F
- R, Z, ESR : 100 mΩ to 10 MΩ
- D : 0.0001 to 9.999

(9) Appropriate button is pushed for the quantity to be measured. Two six digit displays with annunciators allow 4½ digit or 5½ digit (high resolution mode) with a basic accuracy of 0.1%. The high resolution mode is very convenient for measuring film capacities with a maximum dielectric loss resolution of .00001 and capacity resolution of .0001 pF.
2.7.5 Wayne Kerr RF Bridge

The photograph 2.14 shows Wayne Kerr universal RF bridge B 602 with source and detector which we have used for the measurement in the range 100 kHz to 10 MHz. The bridge is based on the transformer ratio-arm principle. The equipment is designed to have a continuously variable precision voltage dividers by using a magnetic field in a single turn loop. One of the consequences of this is, that there is no need of variable standards. Fixed standards for capacitance, resistance and inductance with phase reversing windings on the transformers, permits aperiodic measurements in all four quadrants of the complex plane. When the test condenser is measured in terms of its parallel components, the bridge measures capacitance, conductance, resistance and inductance with an accuracy of 1% at frequencies between 100 kHz and 3 MHz. From 3 to 5 MHz the accuracy is 2% and from 5 to 10 MHz, a 5% accuracy is claimed. The capacitance can be measured upto 1000 microfarad and resistance upto 100 M ohms. The arrangement of the bridge is such that on the five upper ranges (for example 1 pF, 10 pF, 100 pF, 1 nF and 10 nF for capacitance) the unknown is measured in terms of its parallel components, while on the lower three ranges the measurement is in terms of the series components. Either form can be readily converted into the other. Discriminations in linear scales are 0.1% of full range capability. One division of these scales represent 1% of full range capability. A complete analysis of the bridge is given in the
FIG. 2.14 EXPERIMENTAL SET UP FOR HIGH FREQUENCY DIELECTRIC MEASUREMENT BY WAYNE KERR BRIDGE
Wayne Kerr monograph No.1 on the transformer ratio arm bridge.

We have used Wayne Kerr Model SR 263 L as source and detector for the bridge. It covers the frequency range 100 KHz to 100 MHz in nine bands. Separate attenuators are provided for source output and detector input. Tuning of source and detector are ganged and null detection is by visual observation.

For operation of the instrument, the first requisite is to make the appropriate ground and neutral connections according to the type of measurements required. For unbalanced impedances ground terminal of the instrument is connected to the ground of the specimen under test. The bridge is trimmed. The test condenser is connected to appropriate range. Desired frequency is chosen and the balancing dials are operated to obtain null position. If necessary, source output and sensitivity of the detector may be increased. Capacitance \( C_p \) is noted from the unity scale dials provided for them. Unity scale indications of conductance dial are divided by unity scale indications of capacitance dial, the result is multiplied by \( 10^{7/2\pi n} \) where \( n \) is the frequency chosen. This gives dissipation factor \( \tan \delta \). This multiplication is not done for frequency 1.592 MHz. The experiment was performed at different temperatures, the heating arrangement has already been described.
2.8 THICKNESS MEASUREMENT

2.8.1 Tolansky Technique

Thicknesses of most of the films were measured by the method of multiple beam interferometry (Tolansky, 1948). We have used Tolansky technique which utilizes 'Fizeau's fringes of equal thickness'. In this technique an optical wedge is produced between the film surface and an optical flat (called Fizeau plate), and this is illuminated with a parallel monochromatic light at normal incidence. The interference fringes are formed which contours the points of equal air gap thickness and are separated by a distance of half wavelength. There is a linear correspondence between the vertical dimensions of the surface and the excursions of the interference fringes. Conventional Metallurgical microscopes can be used for the observation and measurement of these fringes (Maissel and Glash, 1970).

We have used Metallurgical microscope with a calibrated eye piece manufactured by Andhra Scientific Co. For monochromatic source, we have used mercury lamp with a green filter (5461 Å). The schematic diagram of the apparatus is shown in Figure 2.15 and actual photograph of the experimental set up is shown in photograph 2.16.

The stage interferometer designed by us is shown in Figure 2.17. The arrangement is very simple; it consists of a
FIG. 2.15 MEASUREMENT OF FILM THICKNESS USING METALLURGICAL MICROSCOPE.
FIG 2:17 SET UP FOR THICKNESS MEASUREMENT.
base plate of 5 cm diameter. The base plate contains a central table of adjustable height and three finely threaded long screws to hold an upper flat ring plate at any desired height. The flat ring plate is provided with the space to hold the Fizeau plate. The Fizeau plate is kept in this space and is firmly clipped from the sides. The ring plate is supported by the long screws and is sandwiched between springs and adjustment screw. This screw adjustment controls the strike and dip of the Fizeau plate with respect to the film surface. Fixing of plate on the ring has its own advantages as now it is not possible to slide the flat relative to the film thereby preventing the scratches on the film.

For the measurement of thickness of the film, the film is scratched without affecting the substrate, so that a step is created equal in height to the film thickness. Scratched film surface is silvered to be approximately 100% reflecting by vacuum evaporation technique. Similarly, one surface of the Fizeau plate is also silvered to achieve 80% reflection. This value of 80% reflection for the glass surface is a practical value, being relatively easy to achieve and not critical (Boeker and Benjamin, 1962). The coating of the two glass plates with the same reflecting material causes the phase change suffered by the two interfering beams on the reflection from the two surfaces to be the same, thereby making the fringe shift at the step edge due to step height only. Good quality fringes occur only if the two silvered surfaces are very close
together. The film thickness is given by the expression

\[ d = \frac{p \cdot \lambda}{2} \]  

.. (2.4)

where \( p \) is the fringe displacement and \( \lambda \) is wavelength of the light used.

2.8.2 Thickness by Quartz Crystal Monitor

By measuring change in frequency of the quartz crystal wafer due to film deposition the thickness of the film deposited on the wafer can be calculated by the expression already given in Section (1.4.1 b)

\[ \Delta_f = -C_f \cdot t_q \cdot C_F \]  

.. (2.5)

By measuring the distance of the substrate \( R_S \) and the distance of the quartz wafer \( R_Q \) from the evaporation source film thickness deposited at the substrate can be calculated. If the film thickness at the substrate is \( t_s \) and that at the wafer is \( t_q \), then from the inverse square relation

\[ t_s = t_q \cdot \frac{R_Q^2}{R_S^2} = \frac{\Delta f}{C_f \cdot C_F} \cdot \frac{R_Q^2}{R_S^2} \]  

.. (2.6)

For a 5 MHz crystal, \( C_f = 5.63 \times 10^7 \text{ cm}^2/\text{g} \) and density of the cerium dioxide is \( 8 \text{ g/cm}^3 \). Hence film thickness can be calculated.

Alternatively, we can calibrate the shift in terms of
aluminium deposits. If frequency shift \( x \) corresponds to 1 cm deposit of aluminium, then

\[
x = c_f \cdot t \cdot \frac{C_{Al}}{C_F}
\]

\[
t_s = \frac{x}{\Delta f} \cdot \frac{C_{Al}}{C_F} \cdot \frac{R_p^2}{R_s^2}
\]

Thickness of films used for dielectric measurements in the range 100 Hz to 100 KHz were measured by this method.