CHAPTER III
EXPERIMENTAL TECHNIQUES AND PROCEDURES

This chapter deals with the techniques, employed in the present investigation for the preparation and characterisation of Bi-SiO cermet films. Different deposition techniques used for the film preparation in brief and the technique employed in the present studies are discussed. The experimental methods used for the study of composition, thickness, structure, electrical, dielectric and optical properties of these films are also outlined.
3.1. Introduction

The properties of a thin film depend strongly on the structure of the film which in turn is influenced by the processes that occur during the film formation. Hence, the properties of a thin film may be quite different from that of the bulk, particularly when the thickness of the film is very small. Though each deposition technique has its own advantages, the properties of the films prepared under each deposition technique are much influenced by the deposition parameters like rate of evaporation, source to substrate distance, the pressure in the chamber, the substrate temperature etc.

The present study was undertaken to prepare Bi-SiO cermet films and examine their behaviour for a possible application as resistors in microelectronic circuits. As far as possible, the deposition technique chosen must be simple, easy to operate and at the same time preferably at a lower cost with which good quality films can be obtained.

Several special evaporation techniques have been developed for depositing the materials whose
constituents have different vapour pressures. The literature survey [1-6], revealed that both evaporation and sputtering techniques had been employed for the preparation of various cermet systems such as Cr-SiO, Au-SiO\(_2\) and Ta-Al\(_2\)O\(_3\) etc. Some of the techniques used for the preparation of metal-dielectric composites are discussed below.

3 2. **Evaporation Techniques**

3 2.1. *Thermal evaporation*

The simplest method for deposition of a film is by thermal evaporation of the source material in good vacuum. This is also known as resistive heating. In this method the evaporant material is placed in a filament or boat which is heated resistively and the vapour is allowed to condense onto a substrate. Cermets of Cr-SiO, Mn-SiO and Mn-MgF\(_2\) have been prepared by this technique [7-12]. However, due to the different vapour pressures of the constituent elements, it may not be possible to prepare stoichiometric films.
3.2.2 Flash evaporation

In this technique, small quantities of the constituents in powder form are evaporated from a single source. The material taken in the form of fine powder is dispensed onto a heater source (Tantalum or Molybdenum boat), from a vibratory chute in vacuum. This method is applicable for the evaporation of alloys, metal dielectric mixtures and compounds [13-16]. The source temperature must be kept sufficiently at a high temperature such that the instantaneous evaporation of the constituents can occur. If the source temperature is very high, spattering of the material may take place and if it is too low, films with uniform composition throughout cannot be achieved. Hence, in order to avoid spattering of the material, as well as to obtain uniform films, the source temperature must be neither too high nor too low. In this technique, the constituents can be either physically mixed depending on the composition required or a pellet is prepared for a desired composition and ground into fine powder and fed into the hopper. The optimisation of the source temperature should be done accurately since, the evaporant may stick on or accumulate on the chute and
complete evaporation of the material may not take place and this may lead to nonuniformity in film composition.

3.2.3. Electronbeam evaporation

In this evaporation technique, the material is vapourised by an energetic electron beam. The electron source is generally a heated tungsten filament. These high energy electrons are accelerated, deflected and focused through a controlled field onto the evaporating surface. The electron energies are typically of the order of 3 to 10 kW. When the beam impinges on the material, most of the kinetic energy will be converted into heat and the material becomes a molten drop and evaporates. Since, relatively high evaporation rates can be achieved with this technique, this method is suitable for large scale productions.

Because of the high power density at the point of beam impact, spattering of the material may take place. Metal-dielectric mixtures of Cr-SiO (19) have been deposited by employing this technique.
3.2.4. Sputtering technique

Sputtering is a process where the material is dislodged and ejected from the surface of the material due to the momentum exchange associated with the surface bombardment by high energetic particles. The ejected atoms from the surface of the target (the cathode) are allowed to condense onto a substrate which is kept in between anode and cathode or onto the anode itself to form a film. Normally, the chamber is evacuated to a pressure of the order of $5 \times 10^{-4}$ torr. The bombarding species are generally ions of heavy inert gas. In this process, the composition of the sputtered films tends to be the same as that of the target. As the films are prepared comparatively in poor vacuum, it is difficult to obtain pure and stable films. The main difficulty is with the availability of high quality target materials. However, the cermet films of Bi-$SiO_2$ [20], Au-$SiO_2$ [21] and Ta-$Al_2O_3$ [22] have been deposited using this technique.

3.2.5. Co-evaporation technique

In this technique, the two sources are kept
In the same vacuum system and the material is evaporated simultaneously by resistive heating. This is also known as two source or three temperature or Gunther technique [23]. The main advantage of this technique, is that the simultaneous evaporation of the two materials onto the same substrate overcomes the problem of fractionation and the decomposition encountered in the direct evaporation. This technique is found to be very much applicable for the materials which form neither compounds nor single systems. The experimental setup is very simple and can be easily handled. The loss of material in the film formation is avoided and the thickness and rate of deposition can be easily controlled. To avoid cross contamination, the sources are to be separated by shields, which shadow one source from the vapour of the other, without blocking passage to the substrate. Different cermet systems, such as Ag-SiO [24], Cr-SiO [25] and some compounds like InSb [26] CdTe [27] have been prepared using this technique.

3.3 Substrate cleaning

The quality of the surface is the most
important property of the substrate. The nature of the substrate has a significant influence on the growth of the films and this in turn has a profound effect on the properties of the films. The preparation of the films with reproducible properties would necessitate the substrate surfaces that are chemically inert and thermally stable. Hence, in order to obtain uniform films with good adhesion and stability, well cleaned smooth surfaces are essential.

There are several methods to remove the contaminations that are present on the substrate surface. Before any substrate can be used, it must be adequately cleaned. The cleaning procedures to be used depend on the nature of the substrate material and the nature of the contaminants present. Normally, the contaminations will be those from manufacturing procedure of the substrate, air borne dust, finger prints and some oily contaminants.

The organic contaminants can easily be removed with surface active emulsifying washing solutions whereas inorganic contaminants generally require a direct mechanical attack for their removal especially when they are in the particle form. Hence,
the substrate surfaces must be made clean enough that the contaminants do not interfere with the adherence of the film. There are several methods for substrate cleaning. Of all the methods, ultrasonic cleaning is considered to be the best method for cleaning the substrate surfaces.

Corning 7059 glass substrates, NaCl crystals and Copper discs were used as substrate materials in the present study. They were cleaned using the following procedures.

In the case of glass substrates, they were first wiped with tissue paper until no visible contamination was seen. Then they were immersed in hot chromic acid for two to three hours and washed with running tap water. In order to remove greasy or oil contaminants, the substrates were scrubbed vigorously with teapol, a liquid cleanser and a detergent. The substrates were rinsed in cold tap water and then in double distilled water. The chemically cleaned surfaces were then subjected to ultrasonic cleaning where ultrasonic waves with high accelerating forces separate the contaminating particles adhering to the surface of the substrate. They were then mounted vertically in a vapour
degreasing chamber, containing pure isopropyl alcohol which was heated. The substrates were kept in the chamber until they attained the vapour temperature and then they were allowed to dry.

The NaCl substrates were cleaved with a clean sharp razor blade. The abrupt cleavage steps and other defects on the substrates were removed by polishing on a moist cloth followed by a quick rinse in water and methanol. The substrates are then dried by blowing hot air.

The copper substrate discs were immersed in concentrated nitric acid for two to three minutes and rinsed thoroughly in running tap water, followed by deionised water. They were then cleaned ultrasonically. They were allowed to dry and kept in vapour degreasing chamber. These substrate materials were used for compositional analysis.

3.4. Masks

Different masks were prepared from cleaved mica for obtaining film patterns that were necessary for various studies. The design of several masks used in
the present investigation are shown in fig 1. Masks (i) and (ii) were used for depositing thick electrodes for the contacting purpose, used in resistivity measurements. The mask 'a' was used to deposit bottom electrode on the substrate and mask 'b' was used to deposit top electrode for dielectric measurements. Pattern 'c' represents the final form of metal-cermet-metal configuration. The shaded portion represents the effective area of the sandwich and the area enclosed by dotted line represents the film. All the masks were immersed in concentrated HNO₃ and then rinsed in tap water, followed by a rinse in double distilled water. Finally they were allowed to dry.

3.5 Film preparation

In the present study the co-evaporation technique was employed for the preparation of Bi-SiO cermet films. The films prepared by this technique were found to be of uniform composition and pinhole free.

The experimental setup used for the preparation of Bi-SiO cermet films is shown in fig 2. The two sources (made of tantalum) were positioned such
Fig 1 Mask configuration
Fig 2 Experimental setup of co-evaporation technique
that the substrate area was exposed uniformly to both
the vapour streams and the two sources were tilted such
that both were aimed at the same substrate surface,
under the same angle of incidence. Here, the two
sources were separated by 6 cm and the distance between
the source to substrate was 15 cm. A special type of
mask was designed, fixed to the substrate holder and was
kept exactly in between the two sources to isolate the
individual vapours initially. The mask was provided
with a gap of 7.5 cm x 3 cm towards the substrate so as
to enable the intermixing of the two vapours to form a
uniform cermet film. A shutter was fixed to the base
plate which was mechanically operated for a fixed time.
The schematic diagram is shown in fig. 3. The thickness
of the samples was monitored by a quartz crystal. To
obtain different compositions of Bi-SiO, the correspond-
ing atomic percentages of Bi and SiO were calculated
and weighed accurately by Stanton monopan electronic
micro balance and were transferred into the tantalum
boats. The main requirement of a vacuum coating
system is the production of necessary vacuum in the
chamber in which the evaporation is to be carried out.
A Hind High Vac coating system model V5-4 was used for
the deposition of experimental films. A 12" diameter
Fig 3  Schematic diagram of co-evaporation system.
stainless steel belljar with two glass windows was mounted on the stainless steel base plate which was fixed on the top flange of a high vacuum baffle valve. The base plate was provided with 12 bored holes for the feed-throughs. The primary evacuation from 760 torr to $10^{-1}$ torr in the belljar could be done with a two-stage rotary pump. This was provided with a gas ballast valve for pumping out condensable vapours. The belljar could be evacuated to $2 \times 10^{-6}$ torr by a conventional three-stage water-cooled oil diffusion pump backed by the rotary pump. DC 704 silicon oil was used as the charge of the diffusion pump as it had all the desirable properties, viz., low vapour pressure, high molecular weight and high resistance to oxidation in the operating temperature. The diffusion pump was connected to the vacuum chamber via a baffle valve. A stainless steel liquid nitrogen trap is fixed in between the baffle valve and the diffusion pump in order to reduce the back streaming of the diffusion oil vapours into the working chamber and to get the required high vacuum within a short period of time. To measure the backing and roughing pressures, two pirani gauge heads working on the principle of thermal conductivity of gases were used in the measuring range 100 to $10^{-3}$ torr. The cold
cathode ionisation gauge (a high vacuum penning gauge) measured the vacuum in the chamber in the range $10^{-3}$ to $10^{-6}$ torr.

Two identical tantalum boats of 1cm x 1cm in area and 0.5 cm in height were fixed at a distance of 5 cm above the baseplate. The boats were connected through a thick copper leads to a low tension water-cooled feed-throughs. Two transformers capable of producing 100 amps at 10 volts and 200 amps at 5 volts were used for resistively heating the two sources containing bismuth and silicon monoxide respectively. A circular substrate holder made up of copper was mounted on three legs. A radiant heater was placed above the substrate holder and the power was supplied from a variac. The substrate temperature was monitored by a copper-constantan thermo-couple. All the electrical connections were taken through the teflon feed-throughs fixed to the base plate. The substrates that were kept exactly above the two sources, (making normals to the respective sources of Bi and SiO) consist of a pure film of Bi and a pure film of SiO whereas the substrates kept at the centre of the holder consist of a uniform composite film of Bi-SiO of area 7.5 cm x 2.5 cm.
3.6. Characterisation of the films

The following studies were undertaken to characterise the films in the present study:

1. Thickness measurement
2. Film composition
3. Structure
4. Surface morphology
5. Electrical properties
6. Dielectric properties
7. Optical properties

The techniques employed to investigate these properties are discussed in the following sections.

3.6.1 Film thickness measurement

The thickness of the films determined by multiple beam interferometer and quartz crystal monitor are briefly described below.

3.6.1.a. Multiple beam interferometer

In Tolansky's multiple beam interferometer
technique [28], the basic principle involved is the interference of light reflected from two surfaces that are slightly inclined at each other, thus forming an air wedge. The thin film whose thickness is to be determined, is half coated on the substrate. The entire substrate is over coated with a good reflecting film resulting in a step.

The experimental setup [29] consists of an optical microscope which was used to observe the fringe pattern. Light coming from the mercury vapour lamp is allowed to pass through a green filter. The collimated monochromatic light of green illuminates the fringe pattern formed between the top fizeau plate and the substrate. The fringe pattern produced is viewed through a microscope. The fringe separation \(X\), the step height \(x\) were measured accurately with the help of a micrometer attached to the optical microscope. The film thickness \(t\) in angstroms was evaluated from the relation,

\[
t = \frac{x}{X} \frac{\lambda}{2}
\]

where, \(\lambda\) is the wavelength of green light. The film thickness could be measured to an accuracy of \(\pm 10\ \text{Å}\).
3.6.1.b. Quartz crystal monitor

In this technique an AT cut quartz crystal was used to monitor the film thickness. When this quartz crystal with previously deposited electrodes, receives a deposit of mass 'm' at the same time as the substrate, the frequency of the quartz crystal changes. The rate of change in resonant frequency is proportional to the deposition rate and the total change in frequency during deposition is proportional to the mass of the deposit. Since, the crystal is made part of an oscillator, measurement of the change in oscillator frequency permits a determination of the deposition rate.

3.6.2. Compositional analysis

The composition of thin films has received considerable attention in recent years and this has resulted in rapid improvements in a broad range, especially in the field of analytical techniques. Each technique has its own attributes and disadvantages. The technique employed depends on the sample material, method of exciting the sample and the signal that is
detected. In the present study, the composition of the film was determined by Auger Electron Spectroscopy (AES) and Energy Dispersive Analysis by X-rays (EDAX).

3.6.2.a. Auger Electron Spectroscopy

In AES, the electrons are emitted when the excited atoms release their energy in nonradiative transitions. With the help of AES, the composition of the sample can be determined by measuring the energy distribution of electrons that are emitted with characteristic energies during irradiation with a beam of energetic electrons.

In the present investigation, the compositional analysis was carried out by using Perkin Elmer Model PHI-661 Auger Electron Spectrometer (fig 4). The auger electron spectroscopy system consists of an ultra high vacuum system, an electron gun for specimen excitation and an energy analyser for detection of auger electron peaks. The auger analyser is a cylindrical mirror type with a detection limit of 0.1 at%. The sample was evacuated to $10^{-6}$ torr. The atomic percentages can be calculated from the auger spectra using...
Fig 4 Perkin Elmer PHI-661 Auger Electron Spectrometer
auger peak to peak amplitude and primary electron beam current using the relation,

\[ C_x = \frac{I_x}{S_x} / \frac{I_y}{S_y} \]

Where, 'I_x' is the auger peak to peak height of the element taken from the spectra and 'S_x' is the relative sensitivity factor. The relative sensitivity factors of 'B', 'Si' and 'O' are taken from the standard charts of Auger hand book [30]. The corresponding atomic percentages are displayed and recorded on the chart.

3.6.2 b Energy Dispersive Analysis by X-rays

The EDAX Model 840A, X-ray energy analyser attached to the scanning electron microscope JOEL analytical scanning microscope (JSM-840A / WDS / EDS) was used for compositional analysis of the films.

The glass substrate, containing the experimental film was mounted on a platform of the EDAX analyser with silver conducting paste to avoid charging problem. The characteristic X-ray peaks corresponding to the constituent element present were displayed on the screen. From the position of the peaks observed on the
screen, the elements present were identified. The peak height represents the concentration of the element under observation. This peak height was recorded as number of counts in a time interval of 20 sec in the photomultiplier. The relative counts of the constituents give the film composition. The surface of the film was scanned at three different places and the average counts of the constituent elements in the film were recorded.

3.6.3 Structural analysis

The structure of the films was analysed by X-ray and electron diffraction techniques.

3.6.3 a. X-ray diffraction

The structural properties of the experimental films were analysed by Siemens X-ray diffractometer in the scanning range of 2θ from 0 to 180°.

The basis for X-ray diffraction is the Bragg equation, which describes the condition for constructive interference.
2\alpha \sin \theta = n \lambda

where, '\lambda' is the wavelength of the incident radiation. A CuKα target was used as the X-ray source. The pattern was recorded on a chart at a scan speed of 1 cm/min. From the XRD peaks, the 'd' values were calculated using the Bragg equation. The <hkl> values were identified from the standard charts, and the lattice parameters were calculated using the relation,

\[ a = \sqrt[3]{\frac{h^2 + k^2 + l^2}{d^2}} \]

\[ \frac{1}{d^2} = \frac{4}{3a^2} \left( \frac{h^2 + hk + k^2}{2} \right) + \frac{1}{c^2} \]

The grain size of the sample was estimated from the diffraction pattern using,

\[ L = \frac{K\lambda}{\beta \cos \theta} \]

where, '\beta' is the half intensity width of the peak in radians, 'θ' the peak angle, 'λ' the wavelength and 'K' the constant equal to unity.

3.6.3.b. Electron diffraction studies

Electron diffraction is another technique used to identify the structure of the samples in a
transmission electron microscope, the electrons that form the image must pass through the specimen. Hence, the specimen thickness is limited to less than 1000 Å, to get a good diffraction pattern. For high voltage instruments, this thickness limit can be extended to few thousand angstroms or to few microns. This is generally a destructive analysis and hence the TEM analysis is usually viewed as the last step for the measurements.

For the electron diffraction studies, a Philips CM-300 KV transmission electron microscope was used. The films that were formed on NaCl crystals were dissolved in distilled water. The film that will float on the surface of the water is collected on copper grids, dried and was used for the electron diffraction studies. The diffraction pattern of the specimen is photographed.

3.6.3.c. Surface morphology

The surface morphology of a material is the most important special area of interest in characterising a material. A scanning electron microscope provides a direct means of examining the surface
morphology of a sample at high magnifications, with high resolution.

The surface morphology of the present samples was studied, by using JEOL Scanning Electron Microscope. The films were deposited onto the Corning glass substrates, precoated with gold in order to avoid charging problems if there are any and to improve the resolution of the image. The samples were mounted in the vacuum chamber and the surface of the film was scanned at five different spots. The grain size was also calculated from the micrographs.

3.6.4. **Electrical Properties**

The electrical behaviour of the experimental films was studied from the resistivity and TCR measurements. These investigations made it possible to determine the values of sheet resistance, TCR and the activation energy for conduction.

3.6.4 a. **Measurement of resistivity and TCR**

The resistivity of the films was measured by
standard four probe technique. The sample was placed in
the holder with good electrical contacts. The current
was varied from few micro amperes to one milli amp and was
measured using keithley 180B digital multimeter. The
corresponding voltage was measured by a hp 3466 digital
multimeter.

The sheet resistance $R_s$ was calculated using
the relation,

$$R_s = \frac{\frac{1}{2} (R_1 + R_2)}{\ln 2} \cdot f$$

where, 'f' is the van der pauw correction factor, $R_1$
and $R_2$ are the resistances calculated by passing the
current through AB and measuring the potential diffe-
rence across CD and passing current through AD and
measuring potential difference across BC.

The resistivity $\rho$, of the film was calculated
using the relation

$$\rho = R_s \cdot t \text{ ohm-cm}$$

where 't' is the thickness of the film.

The temperature coefficient of resistance was
measured in the temperature range 303 - 450 K. The heating and cooling rates were maintained at 5 °C/min. The temperature coefficient of resistance ($\alpha$) was calculated using the relation

$$\alpha = \frac{1}{R} \frac{dR}{dT} \; / ^{\circ}\text{C}$$

3.6.4.b. Annealing

Since considerable changes take place during heat treatment, the experimental films were heat treated in a vacuum of $1 \times 10^{-5}$ torr at 100 and 180 °C for four and eight hours. The variation in resistivity was measured with annealing time and temperature.

The activation energy for conduction was determined from the plots of log $\rho$ Vs reciprocal temperature in the temperature range 303-450 K, using the relation

$$\rho = \rho_0 \exp \left( \frac{E_a}{kT} \right)$$

where, $\rho_0$ is the constant of proportionality, $K$ is the Boltzmann constant, $E_a$ is the activation energy and $\rho$ is the resistivity of the sample.
3.6.4.c. **Current-Voltage characteristics**

The ohmic nature of the films was observed from the current-voltage characteristics studied, by HP model 4140 B pA meter, in the voltage range 0 to 90 V.

3.6.4.d. **Stability**

All the experimental films were exposed to air at room temperature for a long time and the resistivity was measured from the day after the evaporation. The stability of the films was determined from this variation of resistivity as a function of time.

3.6.5. **Dielectric Properties**

The dielectric behaviour of the experimental films was studied by capacitance bridge method. A Systronics LCR bridge, type 921 was used for this purpose. The dielectric constant 
$\varepsilon$ was calculated using the relation

$$
\varepsilon = \frac{C_t}{\varepsilon \cdot A}
$$
where, 'C' is the capacitance, 't' is the thickness and 'A' is the area of the film respectively and 'ε₀' is the permittivity of free space (8.85 x 10⁻¹² F/m).

These experimental results were analysed in the light of the theoretical approximations of Maxwell-Garnett.

3.6.6 Optical properties

The optical absorption, transmittance and reflectance measurements were studied using Hitachi U-3400 UV-VIS-NIR double beam spectrophotometer (fig 5) in the wavelength range 0.2 to 2 μm and Pye-Unicam SP3 300 IR spectrophotometer (fig 6) in the wavelength range 2.5 to 25 μm.

The optical absorption coefficient 'α' was calculated from the transmittance Vs wavelength spectrum using the relation,

\[ \ln T = -αt \]

where, 'T' is the film transmittance and 't' is the thickness of the film.
Fig. 5 Hitachi U 3400 UV-VIS-NIR Spectrophotometer
Fig. 6 Pye-Unicam SP3 300 IR Spectrophotometer
The refractive index of the films was calculated from the polarisation spectrum by solving the simultaneous equations of Fresnel and Snell. The formula for calculating the refractive index from 'S' polarisation is

\[
\eta_n = \frac{\left(1 - R_s^2\right)^2}{2 \sin^2 \psi_i - 1 + 2(1 - \sin^2 \psi_i) \left(1 + \sqrt{1 - \left(\frac{1 - R_s^2}{1 + R_s}\right)}\right)}
\]

\[
\frac{1 - R_s}{1 + R_s}
\]

Similarly, the formula for calculating the refractive index from 'P' polarisation spectrum is

\[
\eta_n = \sqrt{1 + 4 \sin^2 \psi_i \left(1 - \sin^2 \psi_i\right) \left(1 - \frac{1}{\sqrt{R_p}}\right)^2}
\]

\[
\frac{2 \left(1 - \sin^2 \psi_i\right) \left(1 - \sqrt{R_p}\right)}{1 + \sqrt{R_p}}
\]

where, 'n' is the refractive index of the sample material, 'R_s' and 'R_p' are the photometric values of S and P representing polarisation components of the reflected beam obtained when the polarisation plane is parallel and perpendicular to the incident plane respectively, \(\psi_i\) is the incident angle.
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