Chapter III

Characterization techniques

3.1 Introduction:

In this chapter, we discuss the details of various analytical techniques used in the present study for thin film characterization. The techniques involve stylus profilometry for thickness measurements and X-ray diffraction for examining crystallinity, crystallite size and orientation. Dynamic Force Microscope (DFM) and Atomic Force Microscope (AFM) were used for surface topography and surface roughness determination. UV-Vis-NIR spectrophotometer was used to obtain spectral transmission curves from which the optical constants such as refractive index and optical band gap were extracted. The low frequency dielectric characteristics and tunable properties of BST5 were carried out using a LCZ meter and an impedance analyzer using Metal-Insulator-Metal (MIM) structures. The microwave dielectric characteristics of BST5 thin films were determined using a vector network analyzer (VNA). A brief introduction to photolithography and lift-off process is also given.
3.2 Material characterization:

3.2.1 Thickness measurement:

The thickness of all the samples were measured using stylus profiler (also known as profilometer) (Model XP-1, Ambios Technology, USA). Surface profilometry is a direct, simple and fast measurement technique for determining the physical thickness of thin films. The only requirement is the existence of a step as shown in figure 3.1. In this study, the step was made by covering a part of the substrate using a thin stainless steel shadow mask during deposition. This method involves loading a stylus (generally a diamond stylus) slightly in contact with the film surface and gently dragging it across the step as shown in figure 3.1. The vertical deflection measures the change in step height (film thickness) and the trace is recorded with high accuracy. Film thickness was also estimated from the spectral transmittance curves as described later.

![Image of stylus profilometry](image)

**Figure 3.1:** Illustration of stylus profilometry to measure film thickness.

3.2.2 X-Ray Diffraction:

X-ray diffraction is a well-known technique for characterizing the crystal structure of materials. X-rays have a wavelength of the order of Angstroms (10^{-10} m), which is comparable to the inter-atomic spacing in crystalline solids. Therefore, a diffraction pattern can be observed when a beam of x-ray is directed on a crystalline material (where the atoms are arranged periodically). This diffraction pattern is directly related to the crystal structure of the material under observation. The diffraction of x-rays by a crystalline material is illustrated in figure 3.2.

In the present work, two types of X-ray diffractometers with different x-ray source were used to characterize the samples. One was equipped with Co Kα (\(\lambda=1.7889\) Å) radiation in a wide angled powder X-ray diffractometer (INEL Model CPS120) and a position sensitive detector while the other one was a conventional X-ray diffractometer operating in the Bragg-Brentano geometry with Cu Kα (\(\lambda=1.54056\) Å) radiation (Philips...
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PW 1830 diffractometer). Calibration using a Si standard was done to account for the instrumental line broadening and the value was approximately 0.05° for the former and 0.10 for the later. The patterns were compared with standard patterns (JCPDS) and the phases and degree of crystallinity was determined. A slow scanning rate of 0.5°/min was used to extract data for the measurement of crystallite size. These results gave important information regarding the variation in the films’ orientation, lattice constants, crystallite size, strain etc. as a function of processing conditions.

3.2.2.1 Calculation of crystallite sizes from x-ray diffraction patterns:

Bragg’s Law:

In figure 3.2 below, consider rays 1 and 1a in the incident beam. They strike atoms K and P in the first plane of atoms and are scattered in all directions. Only in the directions 1’ and 1a’, the scattered beams are completely in phase (because QK – PR = PK Cos θ - PK Cos θ = 0) and so capable of reinforcing one another.

![Figure 3.2: Diffraction of X-rays by periodically arranged atoms in a crystalline material.](image)

This relationship is true of all the planes separately, and we have to find the condition for reinforcement of rays scattered by atoms in different planes.

Let us take rays 1 and 2 scattered by atoms K and L, and the path difference for rays 1K1’ and 2L2’ is

\[ ML + LN = d \sin \theta + d \sin \theta \] (3.1)

If this path difference is equal to a whole number \( n \) of wavelengths,
\[ n\lambda = 2d \sin \theta \quad (3.2) \]

where, \( \theta \) is the diffraction angle, \( \lambda \) is wavelength of the X-ray and \( n \) is the order of reflection, which may take any integral value consistent with \( \sin \theta \) but not exceeding unity.

This relation was first formulated by W. L. Bragg and is known as the Bragg law\(^2\).

\[ \frac{n\lambda}{2d} = \sin \theta < 1 \quad (3.3) \]

i.e., \( n\lambda \) must be less than 2d. For \( n = 1 \) the condition for diffraction at any observable angle \( 2\theta \) is \( \lambda < 2d \).

For most sets of crystal planes \( d \) is of the order of 3 Å or less, which means that \( \lambda \) cannot exceed about 6 Å.

Scherrer’s formula is used to estimate the crystallite size, \( t \) given by,

\[ t = \frac{0.93\lambda}{\beta \cos \theta_\beta} \quad (3.4) \]

where, \( \beta \) is angular width, in terms of \( 2\theta \) and is measured in radian at an intensity equal to half the maximum intensity and is called full width at half maximum (FWHM) (shown in figure 3.3 below)

\[ \beta = \frac{1}{2} (2\theta_1 - 2\theta_2) \quad (3.5) \]

**Figure 3.3:** Graphs show the FWHM.
3.2.2.2 Calculation of misfit strain:

The misfit strain $u_m$, can be calculated using the formula,$^{3,4}$

$$u_m = \frac{a_s - a_f}{a_s}$$  \hspace{1cm} (3.6)

where, $a_s$ is the substrate lattice parameter or that of the lower lying layer and $a_f$ is the lattice parameter of the deposited film.

3.2.2.3 Calculation of residual strain:

The residual strain, can be calculated using the formula,$^5$

$$u_b = \frac{d_b - d_f}{d_b}$$  \hspace{1cm} (3.7)

where, $d_b$ is the inter-planar spacing of the bulk BST5 and $d_f$ is that in the film.

3.2.3 Scanning Probe Microscopy:

Scanning probe microscopy (SPM) covers several related technologies for imaging and measuring surfaces on a fine scale, down to the level of molecules and groups of atoms. SPM technologies share the concept of scanning an extremely sharp tip (3-50 nm radius of curvature) across the sample surface. The tip is mounted on a flexible cantilever, allowing the tip to follow the surface profile.

When the tip moves in proximity to the investigated object, forces of interaction between the tip and the surface influence the movement of the cantilever$^6$. These movements are detected by selective sensors. Various interactions can be studied depending on the mechanics of the probe. Our interest is in the atomic force microscope (AFM). The atomic force microscope (AFM) probes the surface of a sample with a sharp tip (often less than 100A$^9$ in diameter). The tip is located at the free end of a cantilever that is 100 to 200μm long. Forces between the tip and the sample surface cause the cantilever to bend, or deflect. A detector measures cantilever deflections as the tip is scanned over the sample, or the sample is scanned under the tip. The measured cantilever deflection is mapped as the surface topography$^7$. AFM can be used for insulator and semiconductor as well as electrical conductors$^8$. The schematic diagram of AFM is shown in figure 3.4.
Several forces contribute to the deflection of an AFM cantilever. The force most commonly associated with atomic force microscopy is an inter-atomic force called the Van der Waals force depending upon the distance between tip and sample as shown in figure 3.5.

**Figure 3.4:** Schematic diagram of atomic force microscopy (AFM).9

**Figure 3.5:** Nature of forces in the Contact and Non-contact mode of AFM operation.
Two distance regimes are labeled in figure 3.5 the contact regime and the non-contact regime. In the contact regime, the cantilever is held less than a few angstroms above the sample surface and the interatomic force between the cantilever and sample is repulsive\textsuperscript{10}. In the non-contact regime, the cantilever is held about tens to hundreds of angstroms away from its attractive regime (largely a result of the long-range Van der Waals interactions).

### 3.2.3.1 Non-contact AFM:

Non-contact AFM (NC-AFM, otherwise known as dynamic force microscope, DFM) is one of the modes of operation in AFM, where the cantilever is vibrated near the surface of the sample. The spacing between the tip and sample for DFM is of the order of tens to hundreds of angstroms. This spacing is indicated in the Van der Waal force curve in figure 3.5 as the non-contact regime.

DFM is desirable because it provides a means for measuring sample topography with little or no contact between the tip and sample. Similar to contact-AFM, DFM can also be used to measure the topography of the insulator and semiconductors as well as electrical conductors. The total force between the tip and sample is very low, generally $10^{-12}$ N.

Since the force between tip and sample in the non-contact regime is low, it is more difficult to measure this region than in the contact regime. In addition, the cantilever used in DFM is stiffer than those used for contact AFM because soft cantilevers can be pulled easily onto the sample surface. Hence, both these factors make the DFM signal difficult to measure. Thus, a sensitive, AC detection scheme is used in this operation.

In the non-contact mode, the system vibrates a stiff cantilever near its resonant frequency (typically from 100 to 400 kHz). Then, it detects the changes in the resonant frequency or vibration amplitude as the tip comes near the sample surface. The sensitivity of this detection scheme provides sub-angstrom vertical resolution in the image, as with contact AFM.

The relationship between the resonant frequency of the cantilever and variations in sample topography can be explained as follows. The resonant frequency of a cantilever varies as the square root of its spring constant. In addition, the spring constant of the
cantilever varies with the force gradient experienced by the cantilever. Finally, the force gradient, which is the derivative of the force Vs distance curve changes with the tip-to-sample separation. Thus, change in the resonant frequency of the cantilever can be used as a measure of change in the force gradient, which changes with the tip-to-sample spacing, or sample topography.

DFM does not suffer from the tip or sample degradation effects that are sometimes observed after taking numerous scans with contact AFM. As mentioned above, DFM is preferable to contact AFM for measuring soft samples. In the case of rigid samples, contact and non-contact AFM for images may look the same. In the present study Seiko SPA-400 scanning probe microscope was used in the DFM mode to obtain the surface morphology of BST5 thin film samples.

3.3 Optical Characterization:

Optical properties of the BST5 films were studied using a JASCO V-570 UV-VIS-NIR double beam Spectrophotometer. The optical studies of thin films are mainly concerned with physical phenomena like transmission, reflection, absorption and also the interaction of photon energy with matter and consequent changes in electronic states\(^\text{11}\). The important parameters that can be obtained from the measured spectral transmittance data include optical band gap, refractive index and the thickness. In the present study the data were collected in the range of 190 to 2500 nm, with air as reference.
The Ultra Violet -Visible-Near Infrared (UV-VIS-NIR) spectrophotometer uses two light sources, a deuterium (D$_2$) lamp for ultraviolet region and a halogen lamp for visible and NIR region. The light from the source lamp gets reflected from mirror 1 and beam passes through slit 1 and hits a diffraction grating. The grating can be rotated allowing for a specific wavelength to be selected. At any specific orientation of the grating, only monochromatic (single wavelength) beam successfully passes through slit 2. A filter is used to remove unwanted higher order diffracted beam. The light beam hits a second mirror before it gets split by a half mirror (half of the light is reflected, the other half gets transmitted). One of the beams is allowed to pass through a reference sample (air in the present case), the other passes through the film coated substrate. The intensities of the light beams are then measured at the end as shown in figure 3.6. The photometer (not shown) computes the ratio of the sample signal to reference signal ($I/I_0$) to obtain the transmittance.

If light is incident on a film of refractive index, $n$, coated onto a substrate of refractive index $s$, then at the air-film, the film-substrate and substrate-air interfaces, part of the incident intensity is reflected and part of it is transmitted. From fundamental considerations of optics it follows that since the reflected and transmitted beams originate
from a single coherent source, the beams will exhibit interference effects. The schematic diagram of the optics is shown in figure 3.7 and the typical interference spectrum of the transmitted beams is shown in figure 3.8.

Figure 3.7: Interference from a thin film on a thick transparent substrate, s>>d.

Figure 3.8: A typical spectral transmittance curve drawn with the envelope for the calculation of optical constants.
The condition for constructive interference in such a case is given by,

\[2nd = m\lambda \]  

(3.8)

where \(n\) is the refractive index of the film at a wavelength \(\lambda\) and \(m\) is the order of interference. \(d\) is the thickness of the film that can be calculated from the derived values of refractive indices.

In general the transmission \(T\), is given by the expression,

\[T = \frac{Ax}{B - Cx \cos \phi + Dx^2} \]  

(3.9)

where,

\[A = 16n^2s\]
\[B = (n+1)^3(n+s^2)\]
\[C = 2(n^2-1)(n^2-s^2)\]
\[D = (n-1)^3(n-s^2)\]
\[\phi = 4\pi nd/\lambda\]
\[x = \exp(-\alpha d)\]

For such a system at the points of constructive and destructive interference, the transmittance \(T_M\) and \(T_m\), respectively are given by

\[T_M = \frac{Ax}{B - Cx + Dx^2} \]  

(3.10)

and

\[T_m = \frac{Ax}{B + Cx + Dx^2} \]  

(3.11)

For simplicity it can be assumed that the transmission is a continuously varying function of wavelength which can be approximated by drawing an envelope across the spectrum, connecting all the maxima and all the minima.

From this it can be shown in the region of weak absorption that

\[n = \left[ N + \left( N^2 - n_s^2 \right)^{\frac{1}{2}} \right]^{\frac{1}{2}} \]  

(3.12)

where,
\[ N = 2n_s \frac{T_M - T_m}{T_M T_m} + \frac{n_s^2 + 1}{2} \]

On adding the reciprocals of the equations 3.10 and 3.11 yields,

\[ \frac{2T_M T_m}{T_M + T_m} = \frac{Ax}{B + Dx^2} \quad (3.13) \]

and solving for \( x \) we get,

\[ x = \frac{F - F[F^2 - (n^2 - 1)(n^2 - s^2)]^{\frac{1}{2}}}{(n-1)^3(n-s^2)} \quad (3.14) \]

where

\[ F = \frac{8n^2s}{T_i} \]

and

\[ T_i = \frac{2T_M T_m}{T_M + T_m} \]

\( T_M \) is the maximum of transmission on the envelope at a wavelength \( \lambda \), and \( T_m \) is the minimum in transmission on the envelope at the same wavelength. The \( T_M \) and \( T_m \) at each wavelength can therefore be read off from the envelope and the refractive index, consequentially can be derived at each wavelength.

From the condition for constructive interference it can be seen that for two successive maxima occurring at wavelengths \( \lambda_1 \) and \( \lambda_2 \), the equation becomes,

\[ 2n_1d = m_1\lambda_1 \quad (3.15) \]

and

\[ 2n_2d = m_2\lambda_2 \quad (3.16) \]

Also

\[ \left| m_1 - m_2 \right| = 1 \quad (3.17) \]

So,

\[ d = \frac{\lambda_1\lambda_2}{2(n_1\lambda_2 - n_2\lambda_1)} \quad (3.18) \]

In the region of strong absorption, from Beer-Lambert’s law given by,

\[ I = I_0 \exp(-\alpha d) \quad (3.19) \]
where,

\[ I_o = \text{incident intensity} = 1 \]

\[ I = \text{intensity at a given wavelength}, \lambda \]

\[ d \] is the thickness of the film and \( \alpha \) the absorption coefficient in \( \text{cm}^{-1} \).

Since \( d \) is known from previous calculations and \( I \) is a measured quantity (i.e. the transmission at a wavelength \( \lambda \)) the absorption coefficient \( \alpha \) can be calculated. Knowing \( \alpha \), from the expression for the so-called “Tauc gap” the fundamental absorption edge of the material can be determined.

The expression for the Tauc gap is given by\(^{14}\),

\[ \alpha \nu = \text{constant} \times (\hbar \nu - E_g)^2 \]  

(3.20)

x-intercept of the extrapolation of the linear region in a plot of \((\alpha \nu)^{1/2}\) vs \(\hbar \nu\) will give the value of the band gap \(E_g\). The error associated with the measurement of \(k\), \(n\) and \(d\) is \(\pm 0.005\), \(\pm 0.02\) and \(\pm 10\, \text{nm}\) respectively.

The thickness measured using this technique was comparable with the thickness measured using the stylus profiler.

3.4 Electrical Characterization:

None of the material analysis techniques discussed above require specific sample preparation or any complex test structures. However, electrical characterization of the BST5 thin films requires special test structures and sample preparation. The electrical characterization includes measurement of dielectric constant, dielectric loss and tunability at low frequency (40Hz to 1MHz) and at microwave frequencies (2 GHz to 10 GHz). The test structures used for electric characterization are M-I-M capacitor structure and Co-planar wave guide (CPW) lines. In the present study, these structures were fabricated using a single step photolithography and lift-off process. The top silver (Ag)/ gold (Au) multilayer metallization was done using a dual cathode RF magnetron sputtering system (MAGSPUT-1G2-RF-HOT-UPG, Torr, USA). Silver was chosen because of its high conductivity and the thin layer of gold acts as a capping layer because of its oxidation resistance.
3.4.1 Photolithography and lift-off process:

Photolithography is a process of transferring patterns of geometric shapes on a mask to a thin layer of photosensitive material covering the surface of a substrate (wafer) or a film coated substrate. The photosensitive material is known as “photoresist”. The resist pattern defined by the lithographic process is not a permanent element of the final device, but only a replica of device features\(^\text{15}\). There are two types of photoresists, they are positive photoresist and negative photoresist\(^\text{16}\). In the present work, an “i” line \((\lambda=365\text{nm})\) positive photoresist (OIR620-10M, Fuji films) was used. The steps involved in the photolithographic process are wafer cleaning; dehydration bake, photoresist spin coating; soft baking; mask alignment and exposure and development.

**Cleaning:** In order to remove the traces of dust, organic and ionic contamination from the surface, the thin film coated substrates were ultrasonically cleaned for 5 minutes each in acetone, isopropyl alcohol and de-ionized (DI) water. The substrates were then rinsed under free flowing DI water for 30 seconds and desiccated using dry \(\text{N}_2\).

**Dehydration bake:** The cleaned substrates were baked at 110\(^\circ\)C for 15 minutes to dry off moisture that may be present on the wafer surface.

**Photoresist spin coating:** The photoresist is coated on the surface of thin film deposited substrate using a spin-coating machine (WS-400B-6NPP, Laurell). The substrate is placed on a vacuum chuck which can rotate along its own axis while holding the substrate. A small quantity of the photoresist is dispensed in the centre of the spinning substrate and the rotation causes the resist to be spread across the surface of the wafer with excess being thrown off. The uniformity and thickness of the photoresist depends on the spinning process. Initial spinning speed of 5rpm (rotations per minute) and then at 2500 rpm for 30 seconds guaranteed uniform spreading and thickness of 1.2 ± 0.05 \(\mu\text{m}\). The spin coating process is shown in figure 3.9.

**Soft bake:** The photoresist coated substrates were gently heated at 105\(^\circ\)C for 15 min in a hotplate to evaporate the resist solvent and to partially solidify the resist. Soft bake time and temperature also influence resist thickness, adhesion, exposure and development rate. The thickness of the soft baked resist was about 1.0 ± 0.05 \(\mu\text{m}\) after soft bake process.
Exposure: The soft baked photoresist is exposed to UV light through a photomask containing the geometric patterns. The photomask is created by a photographic process and developed onto a glass substrate. Alignment of the mask is critical and is achieved by adjusting x-y positions as well as by rotation (θ) as shown in figure 3.10.

Once the mask is accurately aligned with the pattern on the substrate's surface, the photoresist is exposed through the pattern on the mask with a high intensity ultraviolet light. The alignment and UV light exposure for 10 seconds was done using a contact aligner (MJB4 mask aligner, Suss). The photograph of the aligner is shown in figure 3.11.
Developing: During the exposure process, the resist undergoes a chemical reaction, depending on the chemical composition of resist. The action of light on a positive resist as in our case causes it to polymerize in places where it has been exposed to UV light. Thus the polymerized part can be removed during developing, which is the last steps in the photolithographic process. In our case the exposed resist was developed in a water based developer solution for 60 seconds, thoroughly rinsed in running DI water and dried in a hot plate.

The top electrode consisting of multilayer of silver (Ag, 400nm) and gold (Au, 100nm) was deposited on the substrate such that both the exposed and un-exposed regions are covered completely.

In the lift-off process, the metalized substrates were put in an ultrasonic acetone bath for about 10 minutes to remove the unwanted photoresist and the metal layer.

Various electrical measurements were carried out using these top electrode patterned BST5 thin films.

3.4.2 Low frequency dielectric measurements:

The frequency and voltage dependent dielectric properties of BST5 films grown on Pt/Si substrates were characterized at low frequencies (≤ 1MHz) using the MIM
structures. The top electrode of 300μm² area of the MIM structure was fabricated using the photolithographic process as described in the previous section. The measurements were carried out from 40 Hz to 1 MHz using an Agilent 4294A impedance analyzer (shown in figure 3.12).

![Photograph of the Agilent 4294A impedance analyzer.](image)

**Figure 3.12:** Photograph of the Agilent 4294A impedance analyzer.

The schematic cross section and top view of the M-I-M structure fabricated is shown in figure 3.13.

![Cross-sectional view of the MIM structure.](image)

**Figure 3.13:** Cross-sectional view of the MIM structure.

The top and bottom electrodes of the MIM (Au/Ag/BST5/Pt) structures were connected to the impedance analyzer through two tungsten probes connected to their respective micropositioners in the probe station (LMS 2709, J microtechnology), which in turn is connected with the impedance analyzer allowing a straightforward calculation of dielectric permittivity by the parallel plate capacitor equations.
An impedance analyzer measures the current response of the circuit by applying a sinusoidal voltage signal and calculates the impedance and phase angle. The impedance is converted to capacitance while the phase angle to $\tan \delta$ assuming an equivalent circuit of ideal capacitors and resistors to mimic a real capacitor that allows the flow of alternating current. Capacitance and $\tan \delta$ can be measured over a range of DC bias superimposed on the sinusoidal voltage signal. The collected $C-V$ data allows calculation of the permittivity, dielectric loss, and tunability of the BST film at discrete DC bias voltages. The tunability measurements using impedance analyzer in the present study was carried out at 100 kHz and 1 MHz.

Dielectric frequency dispersion was measured using the same set-up that is used for C-V characterization. The Agilent 4294A impedance analyzer can operate between 40 Hz and 110 MHz but the MIM capacitors are only impedance matched to the measurement circuit over a range of 40 Hz to 1 MHz of frequency. Thus for frequency higher than 1 MHz the calculated capacitance and $\tan \delta$ values were dominated by measurement artifacts.

This set of electrical characterization experiments allows a comprehensive understanding of the dielectric properties of BST5 thin films as they relate to tunable varactors in frequency-agile circuits. Capacitance versus DC bias yields a quantitative estimate of tunability, loss tangent and the ability to tolerate high fields while measurement of the frequency dispersion gives a qualitative assessment of the mobility and concentration of excess charges.

The dielectric properties were calculated using the following equations\(^{17}\).

Relative permittivity,

$$\varepsilon_r = \frac{C \cdot d}{A \cdot \varepsilon_0} \quad (3.21)$$

Quality Factor,

$$Q = \frac{1}{\tan \delta} \quad (3.22)$$

Relative tunability (%)

$$\text{Relative tunability (\%)} = \frac{\varepsilon_{\text{max}} - \varepsilon_{\text{min}}}{\varepsilon_{\text{max}}} \times 100 \quad (3.23)$$

and Figure of merit,

$$FOM = \text{Relative tunability} \times Q \quad (3.24)$$

Here, $\varepsilon_0$ is the permittivity of free space, $A$ and $d$ are the capacitor area and film thickness respectively and $Q$ is the measured quality factor of the capacitor under test.
3.4.3 Microwave characterization:

3.4.3.1 Modified cavity perturbation technique

Microwave dielectric measurement of permittivity and loss tangent of the BST5 thin films deposited on fused silica substrates were carried out at an X band spot frequency of 12.15 GHz using the modified cavity perturbation technique\textsuperscript{18,19}. The experimental measurements were performed using a TE\textsubscript{10n} mode WR-90 rectangular cavity of length 11.6 cm, width 2.3 cm and height 1.07 cm connected through a coaxial to wave-guide adapter to Port 1 of the Agilent 8722ES Vector Network Analyzer (VNA) after performing the one port calibration at the adapter surface. The photograph of the VNA connected to the rectangular cavity is shown in figure 3.14.

![Figure 3.14: Photograph of the rectangular cavity connected to the VNA.](image)

A 1 mm thick bare fused silica substrate with 25 mm length and 5 mm width was thoroughly cleaned and placed inside the cavity through a slot drilled at the center of the cavity. The position of the slot is taken such that the sample surface would be tangential to the maximum electric field (E\textsubscript{max}) position always. First the resonance frequency and quality factor of the empty cavity with the bare substrate is recorded. The bare substrate is then coated with BST5 thin film and this coated substrate is again inserted into the cavity and the corresponding resonance frequency and quality factor are determined. The
superimposed response of the empty cavity, cavity with bare substrate and cavity with film coated substrate are given in figure 3.15.

![Figure 3.15](image)

**Figure 3.15:** The response of the empty cavity, cavity with bare substrate and cavity with film coated substrate superimposed in the same scale.

The main assumption of this method is that the dimensions of the sample are small compared to the wavelength, which is always true in the case of thin films. If the sample surface lies across the cavity and tangential to the electric field then the dielectric constant is given by

\[ \varepsilon' = K \frac{L}{\tau} \frac{\Delta f}{f_0} + 1 \]  

where \( L \) is the cavity length, \( \tau \) is the film thickness, \( \Delta f \) is the frequency shift, \( f_0 \) and \( f_0 \) are the resonance frequencies of the cavity with bare substrate and film coated substrate respectively. The coefficient, \( K \), is a measure of the area of the sample with respect to the cross sectional area of the cavity. It is equal to unity when the whole cross section is covered by the film and is less than unity when the cross section is partially filled with the film. Similarly dielectric loss is given by

\[ \varepsilon'' = K \frac{L}{2\tau} \left[ \frac{1}{Q_{Ls}} - \frac{1}{Q_{L0}} \right] \]  

where \( Q_{Ls} \) and \( Q_{L0} \) denote the unloaded Q factor of the cavity with and without the film coated substrate. The uncoated substrate, in this method therefore, is considered as part of the cavity giving a net resonant frequency, \( f_0 \), provided the substrate is of a low dielectric
constant material. The cavity with the film-coated substrate inside is taken as the perturbed cavity with a resonance frequency $f_0$, $Q_{L0}$ and $Q_{Ls}$ are the corresponding unloaded quality factors of the cavity. It was assumed that the shift in resonance frequency and quality factor from $(f_0, Q_{L0})$ to $(f_{Ls}, Q_{Ls})$ is only due to the properties of the film since the substrate is considered as part of the cavity\textsuperscript{18,19}. It is a reasonable assumption when dielectric constant of the substrate is low as for fused silica ($\varepsilon = 3.8$) in the present case. Hence, the above equations can be applied directly to find out the dielectric constant and loss tangent of the thin film deposited on low dielectric constant substrates. The dielectric constant of BST5 films deposited on single crystal substrates cannot be measured using any of the resonant perturbation techniques because of their high value of dielectric constant.

3.4.3.2 Split post dielectric resonator technique:

This is a non-destructive and accurate technique for measuring the complex permittivity of dielectric substrates and thin films deposited on them at a spot frequency\textsuperscript{21}. It is another resonant structure but without a metallic cavity. It consists of two sapphire dielectric post resonators separated by a distance in which the electric field is uniform\textsuperscript{22}. The gap between the resonators can be used for inserting low dielectric constant substrates of 1 inch square area before and after coating the films. The rest of the principle remains the same as that of the modified cavity perturbation technique. The geometry of split post dielectric resonator used in our measurements is shown in figure 3.16.

The SPDR typically operates in the TE$_{01n}$ mode that has only an azimuthal electric field component, so that the electric field remains continuous on the dielectric interfaces\textsuperscript{23}. This makes the system insensitive to the presence of air gaps perpendicular to the z-axis of the fixture. The real part of permittivity $\varepsilon_r$ of the sample is found on the basis of the measurements of the resonant frequencies and thickness of the sample as an iterative solution.
Figure 3.16: A picture of the split dielectric resonator measurement set up with its schematic diagram.\textsuperscript{21}

\[
\varepsilon' = 1 + \frac{(f_0 - f_s)}{hf_s K_s(\varepsilon', h)}
\]

(3.27)

Here, \(h\) is the sample thickness, \(f_0\) and \(f_s\) are the resonance frequency of the SPDR with the substrate and the film coated substrate respectively. \(K_s\) is a function of the sample’s dielectric constant \(\varepsilon_r\) and thickness \(h\). Since \(K_s\) is a slowly varying function of \(\varepsilon_r\) and \(h\), the iterations using the equation 3.27 converges rapidly.

The loss tangent is computed using the equation,

\[
\tan \delta = \frac{Q^{-1} - Q_{\text{DR}}^{-1} - Q_{\varepsilon}^{-1}}{p_{es}}
\]

(3.28)

where, \(Q\) is the unloaded \(Q\) factor of the SPDR containing the dielectric sample and \(p_{es}\) is the electrical energy-filling factor of the sample. \(Q_e\) is the \(Q\) factor depending on the metal losses of the SPDR containing the dielectric sample and \(Q_{\text{DR}}\) is the \(Q\)-factor depending on the dielectric losses in the dielectric resonators.

3.4.3.3 On-wafer test and analysis:

For the measurement of the scattering parameter of any test device, the device has to be connected to a vector network analyzer. Before the advent of coplanar probes, finding the RF behavior of a device was a complicated process. The wafer had to be diced and an individual die had to be mounted onto a test fixture\textsuperscript{24}. Only then could the device performance be known. Fixturing involved attaching the die to a PCB, wire bonding to
the bond pads, connecting RF cables to the fixture, and measuring. Discriminating between the device and the fixture response had become central problem for high volume screening. One of the obvious advantages of the on-wafer prober at microwave frequencies is that one can get instant measurements on wafers. Thus on-wafer characterization became inevitable in microwave measurements of unpackaged devices. A typical on-wafer microwave measurement setup is shown in figure 3.17 and it consists of a Vector Network Analyzer (VNA), a probe station, on-wafer probes, RF cables and a calibration substrate. The quality of RF measurements depends on the VNA, the reliability of the RF cable and fixture, connectors and the calibration quality. A brief description of the measurement setup is given below.

![Figure 3.17: Photograph of the on-wafer measurement setup.](image)

**Network Analyzer:** Network analyzers are widely used to measure the four elements in a 2 port scattering matrix \((S_{11}, S_{12}, S_{21}, \text{ and } S_{22})\). Basically, a network analyzer can separate and measure the four waves independently; two forward waves, \(a_1\) and \(a_2\) and two reverse traveling waves, \(b_1\) and \(b_2\). The scattering parameters can then be obtained by a combination of these four waves. In our experiments, Agilent Technologies 8722ES VNA was used for the S parameter measurements. It can operate over a frequency range of 50 MHz to 40 GHz.
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**Probe station:** In a probe station, the wafer is held on to a vacuum chuck. The probes are fixed to micro-positioners (KRN-09S) magnetically attached to the probe station. The micro-positioners, enable fine movement up to 0.5” in the x, y plane and up to 0.5” lift in the z direction with 40 turns per inch (tpi) precision movement. RF probes with 250 μm pitch with Ground- Signal- Ground (GSG) configuration (GGB Industries, USA) were used for the measurements.

**Calibration substrate:** An accurate and easily usable calibration substrate with a well defined calibration coefficient and a detailed instruction set to allow accurate calibration of the measurement system (VNA+cabling+probes) is essential for the on-wafer measurements. Typical elements for calibrating a microwave measurement system consist of open, short, matched loads and a thru. These four elements have electrical characteristics that are very different from one another so that each element contributes an important part to the overall calibration process. In the present study, the calibration substrate (model no. Cs-5, GGB industries, USA) that contains high precision elements for calibrating out the errors and losses in an on wafer probing arrangement involving a microwave network analyzer, its associated cabling, and the probes are used.

3.4.3.4 **Coplanar wave guide:**

The Co-Planar Waveguide (CPW) is a type of planar transmission line used in microwave integrated circuits (MIC) as well as in monolithic microwave integrated circuits (MMIC). The unique feature of this transmission line is that it is uniplanar in construction i.e., all the conductors are on the same side of the substrates which, simplifies the manufacturing and allows fast and inexpensive characterization using on-wafer techniques. The basic structure of the CPW is illustrated in figure 3.18.
Figure 3.18: A typical coplanar waveguide (a) cross-sectional view of the structural dimensions, (b) electromagnetic field distribution. The solid lines represent electric fields and the dashed lines represent the magnetic field lines.

This arrangement is assumed to be symmetrical with the strip width, \( w \), and the longitudinal gap, \( s \). The side conductors are ultimately grounded theoretically at infinity. The CPW has some advantages compared to the other transmission lines such as microstriplines. They are simple to fabricate and have reduced dispersion (for small dimensions) as well as radiation losses. They have a reduced cross talk between the lines with higher directivity and a low dependence on substrate thickness. However, the current density on the CPW signal line is not uniform across its surface. The current is concentrated more on the edges of the CPW signal line when compared to that of a microstripline of the same dimensions, which leads to a higher insertion loss in CPW geometry. The biggest advantage of the CPW over microstripline is that the CPW line width is independent of its line impedance. Therefore, one can use wider line which in turn helps in reducing the high frequency conductor loss. It is also possible to achieve a wider range of impedance values in a CPW line when compared to that of other transmission lines like microstriplines.

3.4.3.5 Calibration comparison techniques:

This is a broad band technique that can be used for the extraction of dielectric properties of thin films on a substrate. This technique can also be employed to extract the voltage dependent dielectric properties. The calibration comparison technique makes use of two identical coplanar waveguides (CPW) patterned on the surface of the film and the bare substrate. Two CPW test structures of a 100-micron gap and a 200 micron width were patterned simultaneously, one on the test film and the other on the bare substrate by
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a lift–off process as shown in figure 3.19. The scattering parameters of these CPW transmission lines were measured using a VNA and a probe station (LMS-2709) mounted with the GSG probes of 250-micron pitch.

![Figure 3.19: (a) A picture of the CPW transmission lines fabricated for on-wafer measurement, mounted on the probe station and (b) CPW lines on BST5 thin film deposited on fused silica.](image)

The method used in this study compares the propagation characteristics of the transmission lines fabricated on the bare substrate (reference sample) and the substrate coated with the thin film of BST (test sample). This technique has been successfully used in the past for the characterization of low k as well as high k dielectric thin films\(^27\). The resistance and inductance of the reference and test CPW lines are assumed to be same. Also the loss tangent of the low–loss microwave substrate is assumed to be negligible. The ratio of the propagation constants of the CPW lines in both the cases is given as:

\[
\frac{\gamma_{\text{test}}}{\gamma_{\text{ref}}} = \sqrt{\frac{(R_{\text{test}} + j\omega L_{\text{test}})(G_{\text{test}} + j\omega C_{\text{test}})}{(R_{\text{ref}} + j\omega L_{\text{ref}})(G_{\text{ref}} + j\omega C_{\text{ref}})}}
\]  

(3.29)

This can also be written as:

\[
\frac{(\alpha_{\text{test}} + j\beta_{\text{test}})}{(\alpha_{\text{ref}} + j\beta_{\text{ref}})} = \sqrt{\frac{G_{\text{test}} + j\omega C_{\text{test}}}{j\omega C_{\text{ref}}}}
\]  

(3.30)

where, \( \alpha \) and \( \beta \) are the frequency dependent attenuation and the phase constant respectively. The R, L, C, and G are the resistance, inductance, capacitance and conductance per unit length of the CPW transmission line and they all are frequency
dependent parameters. The conductance per unit length of the test sample can be expressed as:

\[ G_{\text{test}} = \omega \left( C_{\text{test}} \tan \delta_{\text{eff}} \right) \]  \hspace{1cm} (3.31)

where, \( \tan \delta_{\text{eff}} \) is the effective loss tangent of the CPW structure. Substituting \( G_{\text{test}} \) from equation 3.29, and \( C_{\text{test}} = C_{\text{film}} + C_{\text{ref}} \) in equation 3.28, and comparing the real and imaginary parts of the left and right hand sides and solving them, we get the capacitance of the film (\( C_{\text{film}} \)). The dielectric constant of the film \( \varepsilon_{\text{film}} \) is determined from \( C_{\text{film}} \) using the conformal mapping technique\(^{28}\). In the limit of the dielectric film thickness \( t << s \), where \( s \) is the spacing between the centre conductor and the ground line,

\[ \varepsilon_{\text{film}} = \left( s \cdot C_{\text{film}} \right) \left( 2 \varepsilon_0 t \right) - \varepsilon_{\text{substrate}} \]  \hspace{1cm} (3.32)

and the loss tangent of the film is given as:

\[ \tan \delta_{\text{film}} \approx \tan \delta_{\text{eff}} \cdot \left( 1 + C_{\text{ref}} / C_{\text{film}} \right) \]  \hspace{1cm} (3.33)

The frequency dependent attenuation, \( \alpha \) and phase constant, \( \beta \) for the test and reference lines are calculated from the measured magnitude and phase of the \( S_{21} \).

3.4.3.6 Tunability measurements- using CPW structures:

The calibration comparison method can also be extended to measure the dielectric properties of the films under an applied electric field (tunability measurements)\(^{29}\). For the microwave tunability measurements, a DC bias voltage was applied to the CPW lines through the high voltage bias tees connected to the VNA. DC blocking capacitors were used at both the ports of the VNA to give additional protection to it during these measurements. The magnitude and phase of the \( S_{21} \) of the CPW lines patterned on the BST5 thin films are measured under a bias voltage of 100 V. This voltage was able to produce a field of around 10 KV/cm only in the test structures employed. Higher fields were not achieved in this geometry due to the limitation in the bias tees employed.
3.5 Summary:

In summary, an introduction to various characterization techniques such as surface profilometer, X-ray diffraction, UV-Vis-NIR spectrophotometer, Atomic Force microscope used in the present were discussed. The test structures used for measuring the low-frequency dielectric properties using LCZ meter and impedance analyzer and high-frequency (microwave) dielectric properties using Vector Network Analyzer (VNA) were also discussed. An introduction to photolithography and lift-off process and the steps involved in realizing test structures and devices were given.

The next chapter deals with the deposition of amorphous BST5 thin films and the effect of \textit{ex situ} annealing on the structural, microstructural, optical and dielectric properties.
References:

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