Crystal Structure and Optical Properties of $\beta-$ Gallium Oxide thin films

Internship Report

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By

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On work carried out under the guidance of

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-Authors

Abstract

Gallium Oxide (Ga_2O_3) has forgathered significant attention in materials science due to its diverse range of potential applications. The compound which has been familiar to researchers since the late 19^{th} century finds application in a variety of fields such as luminescent phosphors, high-temperature sensors, solar cells, UV optoelctronics etc. Gallium Oxides exhibits 5 polymorphic structures $(\alpha, \beta, \gamma, \delta, \epsilon)$ out of which $\beta - Ga_2O_3$ is widely used due to its high electrical and thermal stability. Hence, the detailed study of crystal structure and optical properties of $\beta - Ga_2O_3$ is carried out in this report. Thin films of $\beta - Ga_2O_3$ fabricated via Radio Frequency (RF) sputtering, is analyzed using X-Ray Diffraction to obtain the crystal structure and lattice parameters. The presence of multiple peaks during XRD analysis indicate polycrystalline nature of the material. Crystallite size and strain exerted on each grain was obtained through Williamson-Hall Plot. Further,we examine the optical properties of these films through UV-Visible Spectroscopy to obtain the Bandgap energy and Urbach Energy

Contents

1 | Introduction

1.1 | Background

Gallium Oxide(Ga_2O_3) has presented itself as a widely studied material in material sciences due to it's wide range of possible applications. The material has been known to us since late 19th Century [3]but much effort has been poured into it's research in the last few decades as it could possibly be the flagbearer of development of transparent semiconducting oxides. Being an ultra wide band-gap semiconductor, it has gotten interest from various fields because of it's applications in electronics, optoelectronics and sensing systems. 5 phases of Gallium Oxide have been studied till now $\alpha, \beta, \gamma, \delta$ and ϵ.[19]Among these polymorphs, β-Ga_2O_3 is the most stable at ambient environments.

1.2 | Our Objective

The objective of this paper is to discuss our findings about the crystal structure and lattice parameters of $\beta - Ga_2O_3$ thin films synthesized using Radio frequency (RF) sputtering, and characterized using X-Ray Diffraction technique. We would further discuss the optical properties of $\beta - Ga_2O_3$ thin films by analysing data from UV-Visible Spectroscopy and calculate the Bandgap Energy and Urbach Energy.

1.3 | Scope of Research

Gallium Oxide has undergone rapid technological maturation over the last decade, pushing it to the forefront of ultra-wide band gap semiconductor technologies. Gallium Oxide is actively used in various fields such as gas sensing. 0_2 and CO have been detected at high temperatures (between 100°and 500°). Along with this selective Hydrocarbons such as acetone and nitromethane are also sensed. Ga_2O_3 is used in Solar Blind Photodetectors. Ga_2O_3 due to its high critical field strength along with large bandgap also finds application is high power devices such as FET and MESFET. Major challenges faced by Gallium Oxide is:

- Low thermal conductivity
- Lack of p-type
- Wafer size limitations
- Multiple polytypes

Active research is going on to overcome these limitations as Gallium Oxide could easily replace other wide-bandgap semiconductors.

2 | Literature Review

2.1 | Crystal Structure of Ga_2O_3

5 phases of Gallium Oxide have been studied till now $\alpha, \beta, \gamma, \delta$ and ϵ.[19]Among these polymorphs, β-Ga_2O_3 is the most stable at ambient environments.
$\beta - Ga_2O_3$ has a monoclinic structure. The unit cell of β-Ga_2O_3 contains 2 crystallographically different Ga cations and 3 O anions. Half of the Ga cations are in distorted tetrahedral coordination(Ga_1), and the other half are in distorted octahedral coordination(Ga_2). O anions are packed in a distorted cubic structure with 2 threefold coordinated types(O_1 and O_2) and one fourfold coordinated type(O_3). Therefore, different bonding environment can be found in β-Ga_2O_3. The low crystallographic symmetry of the monoclinic phase leads to anisotropy of the physical, opical and electricial properties. The (010), and $(\bar{2}01)$ planes are the most commonly used crystal surfaces of device application and thin film growth. β-Ga_2O_3 is a large band-gap semiconductor with direct and indirect bandgaps of 4.9eV and 4.85eV respectively.

2.2 | Thin Film Growth

Thin film materials ranging from microns to nanometres, have exceptional properties compared to the bulk counterpart of the same material. This change in properties because of a change in the dimensions could be because of the things like a larger surface-to-volume ratio and quantum confinement effects. Thin film deposition techniques can be broadly divided into 2 categories, Physical Vapor Deposition and Chemical Vapor Deposition. Some common deposition techniques are as follows:

- **Metal Organic Chemical Vapor Deposition**

Gallium Oxide thin films can be deposited using Gallium Trishexafluoroacetylacetonate [$Ga(HFAc)_3$][2] precursor in the presence of Oxygen or a trimethylgallium (TMGa) and oxygen (O_2) mixture at a temperature of 650°C[9]. Preparation using CVD techniques allows the film to be grown at a relatively low temperature, and have good uniformity.

- **Oxidation of Metal Containing Surfaces**

Oxidation of metal containing surfaces can be used to prepare metal oxide thin films. Gallium Oxide thin films can be prepared by oxidising CoGa[4] or GaAs[16, 8]. Initial oxidation does not result in crystalline films, however, we get the crystalline structure after annealing at temperatures above 700 K.

- **Sol-Gel Method**

Sol Gel method is a versatile technique used to deposit solid thin films using liquid solution. Gallium Oxide thin films can be prepared by using a mix of precursor solutions of gallium isopropoxide, cerium isopropoxide, tungsten ethoxide, antimony butoxide and zinc acetyleacetonate hydrate[12]. These solutions are mixed till homogeneity is achieved after which they can be deposited using the spin coating technique.

- **Pulsed Laser Deposition**

Pulsed laser deposition is a physical vapor deposition technique that uses a high-power pulsed laser beam to ablate material from a target, creating a plasma plume that deposits a thin film on a substrate. Gallium Oxide thin films can be deposited using PLD by using a KrF excimer laser ($\lambda = 248$ nm) as an ablation source, and a ceramic Ga_2O_3 target while keeping the temperature of the substrate between 400-1000°C.[21]

- **Rf Magnetron Sputtering**

Rf Magnetron Sputtering is a physical vapor deposition technique where a high voltage power source is used to create a plasma and sputter the atoms of the desired target. Gallium Oxide thin films can be deposited by using a gallium oxide target using Argon as the sputtering gas.[14]

2.3 | Thin Film Characterization

Once a film is deposited, several techniques are utilized to investigate the crystal structure and quality of the films. Common characterisation techniques are:

- **X-Ray Photoelectron Spectroscopy (XPS)**

X-Ray Photoelectron Spectroscopy (XPS) is a surface-sensitive technique for thin film characterization, providing insights into elemental composition, chemical states, and surface properties. It enables analysis of the outermost layers, typically within 10 nanometers. XPS is crucial for understanding material behavior in applications such as coatings, electronics, and batteries. It allows in situ monitoring of changes during processes like deposition or electrochemical cycling, optimizing manufacturing and understanding material performance under operational conditions.[10]

- **Scanning Electron Microscopy (SEM)**

Scanning Electron Microscopy (SEM) provides high-resolution images that reveal surface morphology and composition. SEM operates by scanning a focused electron beam across the sample, generating signals that convey information about the topography and internal structure of the films. It can achieve resolutions better than 1 nanometer, making it suitable for analyzing grain size, thickness, and shape, which are critical for applications in electronics, optics, and energy devices.[21]

- **High Resolution Transmission Electron Microscopy (HR-TEM)**

High-Resolution Transmission Electron Microscopy (HRTEM) is a powerful technique for thin film characterization, allowing for the examination of structural and morphological details at near-atomic resolution. HRTEM enables the visualization of crystallographic structures, grain boundaries, and defects within thin films, providing insights into their composition and properties. Additionally, it can be used to study dynamic transformations and reactions in real time, making it invaluable for understanding the effects of various treatments on thin film materials. [21]

- **Scanning Tunneling Microscopy(STM)**

Scanning Tunneling Microscopy (STM) is a powerful technique for thin film characterization at the atomic scale. It measures tunneling current between a sharp conductive tip and the sample surface, providing high-resolution images of surface topography and atomic structures. STM is effective for analyzing grain size, defects, and electronic states, and is particularly useful for studying self-assembled molecules. However, it is limited to conductive materials, restricting its application to certain thin film types.[21]

- **Rutherford Backscattering Spectroscopy (RBS)**

Rutherford Backscattering Spectroscopy (RBS) is a valuable technique for thin film characterization, primarily used to determine elemental composition and thickness. By bombarding a sample with high-energy helium ions, RBS measures the energy of backscattered ions, which provides quantitative depth profiles up to 1 micrometer. This non-destructive method is particularly effective for analyzing heavy elements on light substrates and does not require reference standards, making it ideal for semiconductor and optical coating applications.[21]

- **X-Ray Diffraction(XRD)**

In XRD, the variation of intensity of diffracted X-Rays is studied while varying the incident angle slightly over a large range typically 10-80°. The peaks of intensity signify the presence of constructive interference, using which we can gain insight about a series of properties[7] like the crystal structure, degree of crystallinity, film thickness and composition, residual stress and strain, microstructure and texture, preferred orientation, and the phase of the crystal.[1, 5]

2.4 | Optical Properties

Optical properties of metal oxides refer to how these materials interact with light, which includes their behavior in terms of absorption, reflection, transmission, and emission of light. The study of these properties helps in designing materials for specific applications, such as sensors, displays, and energy conversion devices, by tailoring their optical characteristics through material composition and structure.
Optical properties of $\beta - Ga_2O_3$ thin films include:

- Bandgap Energy
- Urbach Energy
- Refractive index
- Dispersion energy
- Defect concentration

Various spectroscopy methods employed to calculate them are

- **Photoluminescence Spectroscopy**

Photoluminescence spectroscopy involves illuminating a sample with light of a specific wavelength, usually from a laser or a lamp to excite electrons within the material from their ground state to higher energy levels. When these excited electrons relax back to lower energy levels they emit photons. This energy distribution is then analyzed in order to determine properties of the material, including defect species, defect concentrations, possible stimulated emission, etc [6].

- **Ellipsometry**

The earliest ellipsometry measurements were used to determine the optical functions such as refractive index and absorption coefficient (α) for several materials. But lately, this method has been largely used to measure the thickness of thin films.

A polarized incident light beam is reflected off a smooth sample surface at a large oblique angle and then re-polarizing the light beam prior to measuring its intensity. Since the process of reflecting light off a smooth sample surface generally changes linearly polarized light into elliptically polarized light, this technique has been called 'ellipsometry'. [17]

- **Raman Spectroscopy**

Raman spectroscopy is a powerful analytical technique used to study vibrational, rotational, and other low-frequency modes in a material. When a sample is illuminated with a monochromatic light source , most of the incident photons undergo elastic scattering (Rayleigh scattering). However, a small fraction of the incident photons undergo inelastic scattering, known as Raman scattering. In this process, the scattered photons lose or gain energy due to interactions with the vibrational modes of the molecules or crystals in the sample

3 | Experimental Methods

3.1 | RF Sputtering

Rf Sputtering is a physical vapor deposition technique where a high voltage power source is used to create a plasma and sputter the atoms of the desired target. Gallium Oxide thin films can be deposited by using a gallium oxide target using Argon as the sputtering gas.[14]

This is the method that we utilised for the deposition of our films, a Ga_2O_3 target was sputtered using a mix of Argon and Oxygen in a 9:1 ratio as the sputtering gas at a pressure of 12 mTorr. We used 3 different substrates; glass, quartz and silicon whose temperatures were in the range of 400°C to 700°C at intervals of 100°C. The sputtering time was kept constant at 2:30 hours for all the samples.

3.2 | X-Ray Diffraction

In XRD, the variation of intensity of diffracted X-Rays is studied while varying the incident angle slightly over a large range typically 10-80°. The peaks of intensity signify the presence of constructive interference, using which we can gain insight about a series of properties[7] like the crystal structure, degree of crystallinity, film thickness and composition, residual stress and strain, microstructure and texture, preferred orientation, and the phase of the crystal[1].

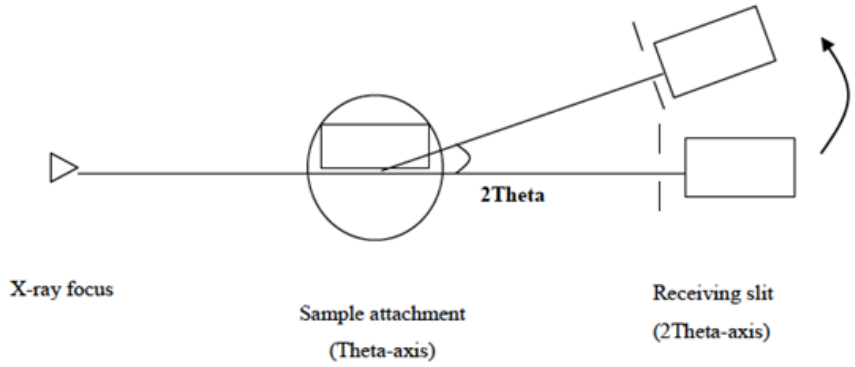

Figure 3.1: The instrument's goniometer uses the following geometry for the collection of data.

We performed XRD by a Rigaku Ultima IV X-Ray Diffractometer using the Bragg-Brentano focusing geometry in the $\theta - 2\theta$ scanning mode in a 2θ range of 10-70° for the samples synthesised at a substrate temperature of 400°C and 500°C and 10-80° for the samples synthesied at 600°C and 700°C. The substrate used was Silicon. The step size was set to 0.02°. A Cu-K_α anode was utilized for the production of X-rays(40kV/40mA) with a wavelength of $\lambda = 1.5406\mathring{A}$.

3.3 | UV-Visible Spectroscopy

UV-Visible Spectroscopy is an analytical technique that is used to measure the absorbance, reflectance or transmittance of waves falling in the visible and ultra-violet range of the electromagnetic spectrum. This technique is commonly used to study the optical properties of materials including semiconductors like Gallium Oxide.[22] Using data from a UV-Vis Spectroscope, we can calculate properties like the bandgap energy and the urbach energy of any given material.

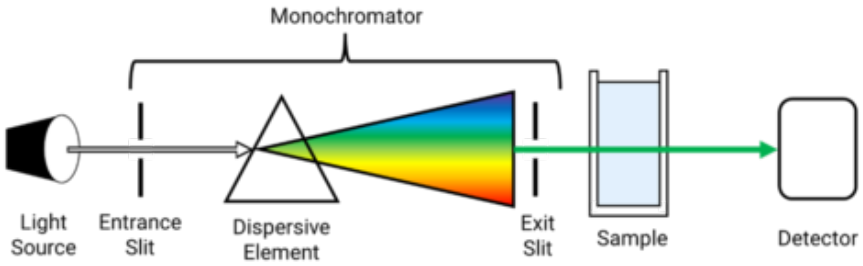

Figure 3.2: Schematics of the inner workings of a UV-Visible Spectrophotometer.

Spectrophotometry was done over a wavelength range of 200nm to 1100nm to study the optical transmission spectra of the thin films deposited on quartz at a substrate temperature of 500°C, 600°C and 700°C.

4 | Results

4.1 | Crystal Structure

Analysis of intensity data from the x-ray diffraction shows us a prominent sharp peak of intensity across all the samples at a 2θ value of 29.2° which corresponds to the (004) plane, with multiple smaller peaks around 2θ values of 35.8°, 39.2° and 43°. Presence of multiple peaks hints towards the polycrystalline nature of the films deposited.

Lattice Parameters

We calculated the lattice parameters a, b, and c with the help of interplanar spacing d_{hkl} of three crystallographic plane (100), (004) and $(\bar{1}13)$ corresponding to the peaks with higher intensity using the formula:

$$\boxed{d_{hkl} = \frac{1}{\sqrt{\frac{h^2}{a^2} + \frac{k^2}{b^2} + \frac{l^2}{c^2}}}} \tag{4.1}$$

The lattice parameters of Gallium Oxide are reported in the table ahead where our findings of the β polymorph are listed along with the parameters of other polymorph referenced from already available literature. β polymorph was found to have a monoclinic structure with the lengths of the unit cell a,b and c in all 3 directions being $a \neq b \neq c$.

Table 4.1: Polymorphs of Gallium Oxide (Ga_2O_3)

Polymorph	Structure	Lattice Parameters	Reference
α	Rhombohedral	$a = 4.9825\mathring{A}$ $c = 13.433\mathring{A}$	M. Marezio[13]
β	Monoclinic	$a = 5.78\mathring{A}$ $b = 3.10\mathring{A}$ $c = 12.21\mathring{A}$	Our Findings
γ	Defective Spinal	$a = 8.30\mathring{A}$	C Otero Areán[15]
δ	Cubic	$a = 10.00\mathring{A}$	R. Roy[18]
ϵ	Hexagonal	$a = 5.120\mathring{A}$ $b = 8.792\mathring{A}$ $c = 9.410\mathring{A}$	S. Yoshioka[20]

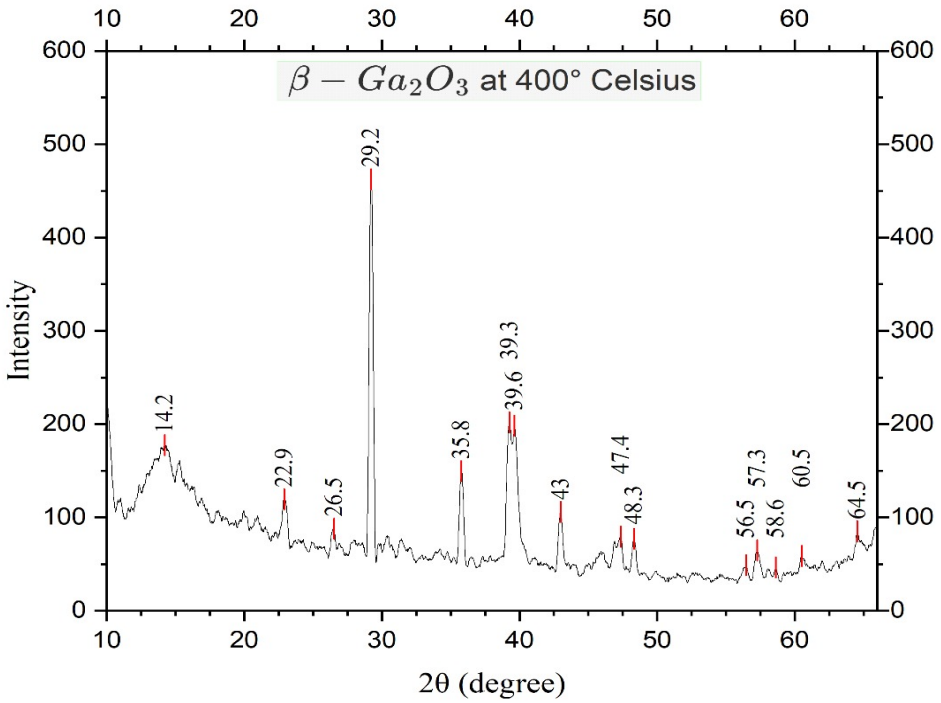

Figure 4.1: Plot of Intensity as a function of 2θ angle of 400° samples.

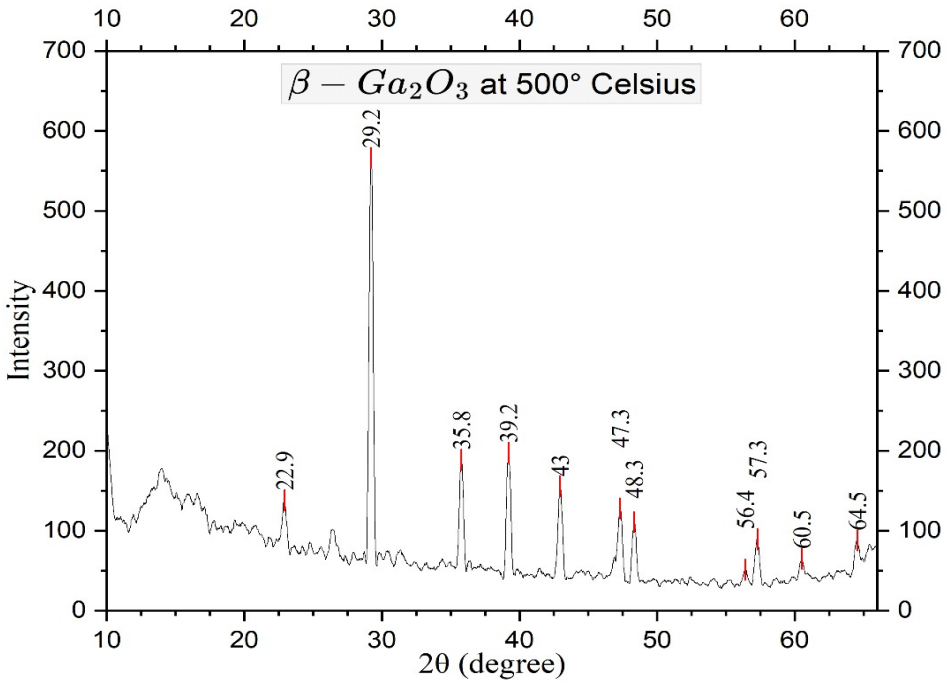

Figure 4.2: Plot of Intensity as a function of 2θ angle of 500° samples.

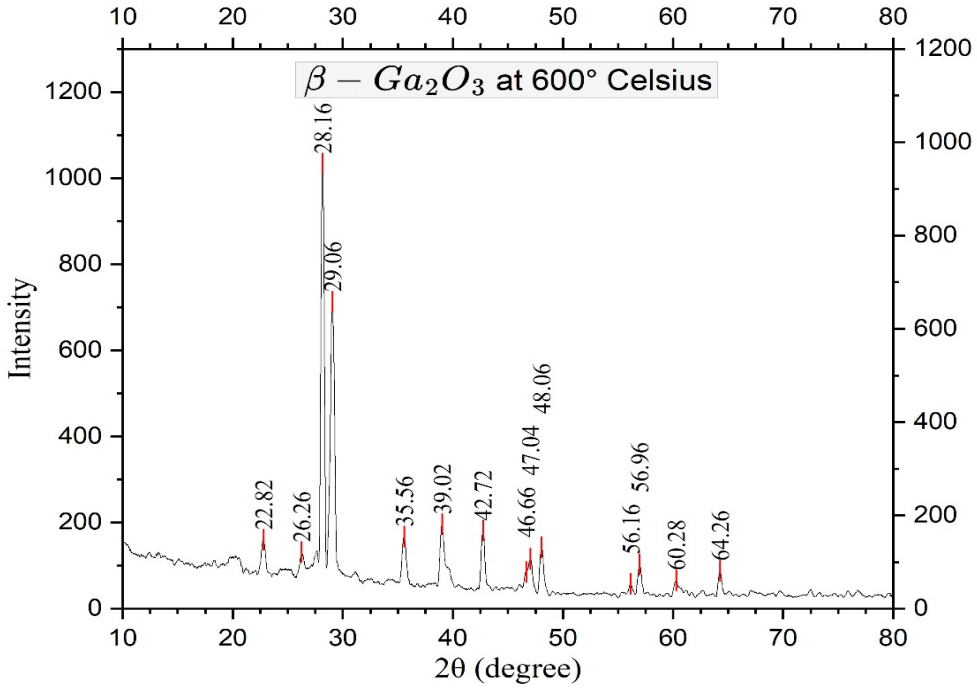

Figure 4.3: Plot of Intensity as a function of 2θ angle of 600° samples.

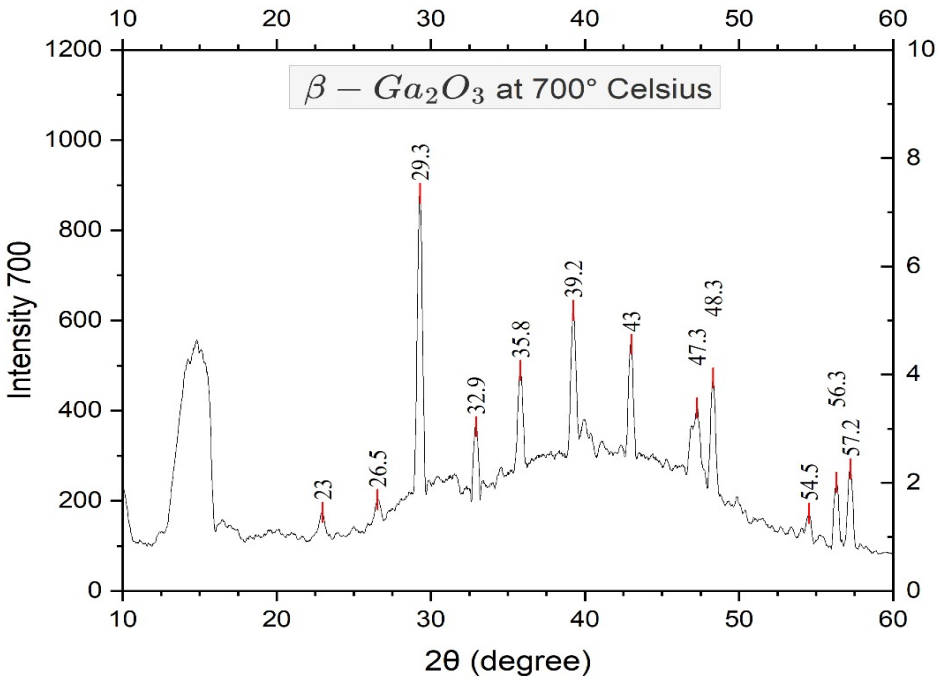

Figure 4.4: Plot of Intensity as a function of 2θ angle of 700° samples.

Crystallite Size

Solids consisting of several tiny crystallites(grains) arranged in different orientations are known as polycrystalline solids. Presence of multiple peaks in the XRD plot suggests that the films deposited are of polycrystalline nature, We calculated the crystalline size in all of our samples using the Debye-Scherrer equation:

$$\boxed{\tau = \frac{K\lambda}{\beta \cos(\theta)}} \tag{4.2}$$

where

- τ is the mean size of the crystalline domains
- K is a dimensionless shape factor, here $K = 0.9$
- λ is the X-Ray radiation wavelength
- β is the full width at half maximum(FWHM)
- θ is the bragg angle

The crystallite sizes were roughly in the range of 22 to 40 nm.

Strain using Williamson Hall Plot

The amount of deformation observed in a polycrystalline structure is refered to as strain. Strain is developed in the nanocrystals due to the point defect, grain boundary, triple junction and stacking faults. Microstrain is calculated using Williamson-Hall method using the equation

$$\boxed{\beta \cos\theta = \frac{K\lambda}{D} + 4\epsilon \sin\theta} \tag{4.3}$$

where

- ϵ is the strain
- β is the full width at half maximum(FWHM)
- K is a dimensionless shape factor, here $K = 0.9$
- λ is the X-Ray radiation wavelength
- D is the crystallite size
- θ is the bragg angle

W-H plot for $\beta - Ga_2O_3$ characterised at various temperatures is plotted with $(4\sin\theta)$ along the x-axis and $(\beta\cos\theta)$ along y-axis. Slop of this plot indicate the strain at different temperatures

Table 4.2: Crystallite size and microstrain obtained through W-H Plot

Material	Temp(°C)	Strain
β-Ga_2O_3	400	0.0026
β-Ga_2O_3	500	0.0056
β-Ga_2O_3	600	0.0039
β-Ga_2O_3	700	0.0006

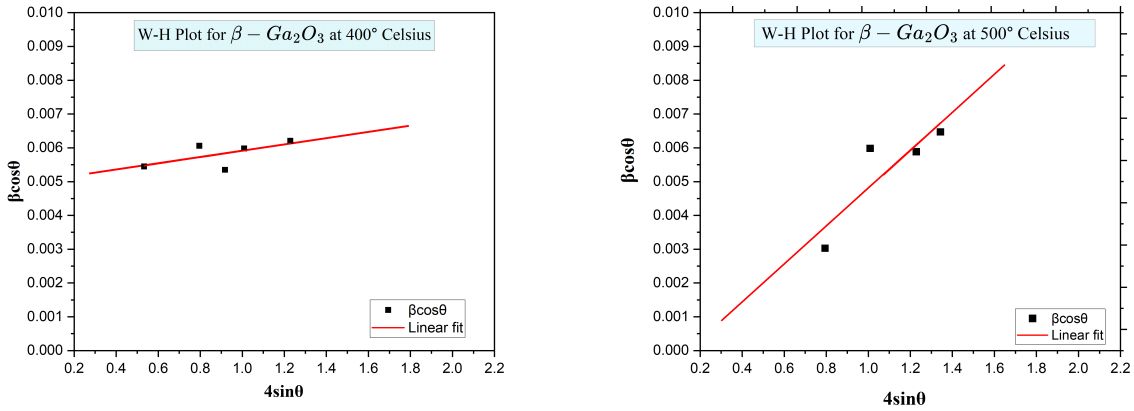

Figure 4.5: Williamson Hall Plots of $\beta - Ga_2O_3$ grown at a substrate temperature of 400°C and 500°C.

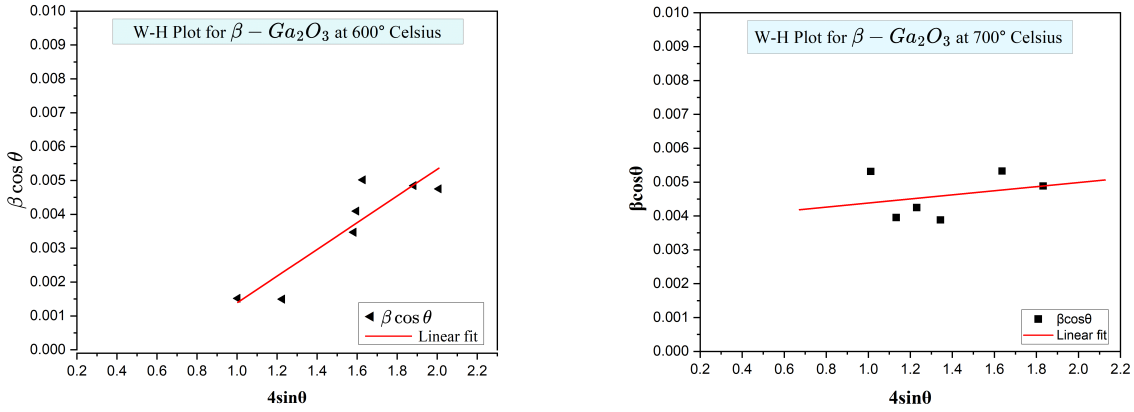

Figure 4.6: Williamson Hall Plots of $\beta - Ga_2O_3$ grown at a substrate temperature of 600°C and 700°C.

4.2 | Bandgap and Urbach Energy

Bandgap Energy

Band gap energy is a key concept in solid-state physics, particularly in materials science and semiconductor technology. It refers to the energy difference between valence band and the conduction band. The bandgap is almost neglible in conductors and too high in insulators for the excitation of

electrons to be feasible. It has an intermediate value in case of a semiconductor material.
The bandgap can be calculated using a Tauc Plot, where the bandgap is equal to intercept on the x-axis of the slope of the linear portion of the graph. The slope of the tauc plot is derived from the tauc equation :

$$\boxed{(\alpha h\nu)^{\frac{1}{n}} = A(h\nu - E_g)} \tag{4.4}$$

where:

- α is the absorption coefficient,
- h is Planck's constant,
- ν is the frequency of light,
- E_g is the bandgap energy,
- A is a constant that depends on the transition probability,
- n is an exponent that depends on the type of electronic transition:
  - $n = \frac{1}{2}$ for direct allowed transitions, (for $\beta - Ga_2O_3$) [11]
  - $n = \frac{3}{2}$ for direct forbidden transitions,
  - $n = 2$ for indirect allowed transitions,
  - $n = 3$ for indirect forbidden transitions.

Table 4.3: Bandgap energy and Urbach energy of our samples

Material	Substrate	Temp(°C)	Bandgap(eV)	Urbach Energy(eV)
β-Ga_2O_3	Quartz	500	4.43	2.29
β-Ga_2O_3	Quartz	600	4.43	2.53
β-Ga_2O_3	Quartz	700	4.5	2.09

Slightly higher bandgap energy for thin films characterised at higher temperature may be due to the amorphous nature or excess oxygen present in the films.

Urbach Energy

Urbach energy is the energy difference between the valence band edge and the exponential decay tail of the absorption coefficient in the band gap region of a material. Urbach energy provides valuable information about the electronic structure and quality of semiconductor and insulator materials, focusing on the gradual absorption tail beyond the sharp band gap edge.

The absorption coefficient (α) can be calculated from the absorbance (A) using the formula:

$$\boxed{\alpha = \frac{2.303 \times A}{d}} \tag{4.5}$$

where:

- A is the absorbance,
- d is the sample thickness.

Next, we plot $\ln(\alpha)$ versus photon energy ($h\nu$):

$$\boxed{\ln(\alpha) = \ln(\alpha_0) + \frac{h\nu}{E_u}} \tag{4.6}$$

where:

- α_0 is a constant,
- E_u is the Urbach energy,
- h is Planck's constant,
- ν is the frequency of light.

To calculate the Urbach energy (E_u), use the slope of the linear fit in the Urbach edge region:

$$\boxed{E_u = \frac{1}{\text{slope}}} \tag{4.7}$$

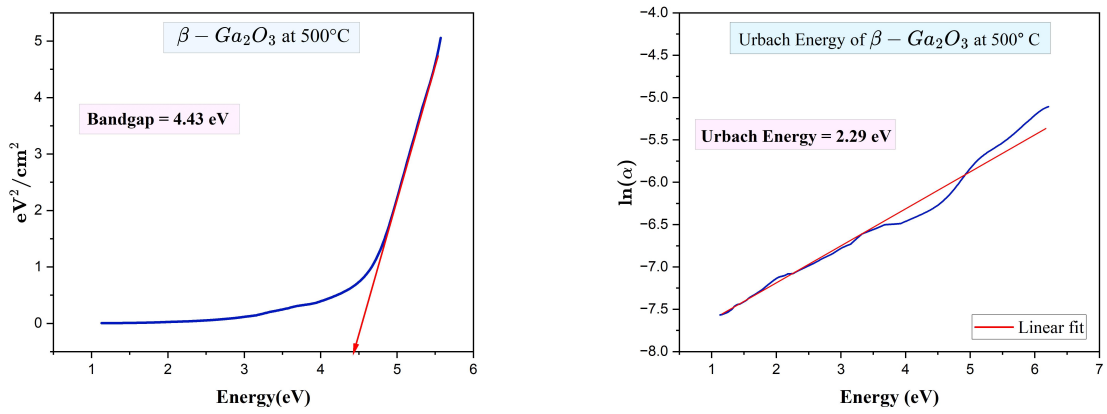

Figure 4.7: Tauc-Plot analysis and Urbach Energy of $\beta - Ga_2O_3$ grown at a substrate temperature of 500°C.

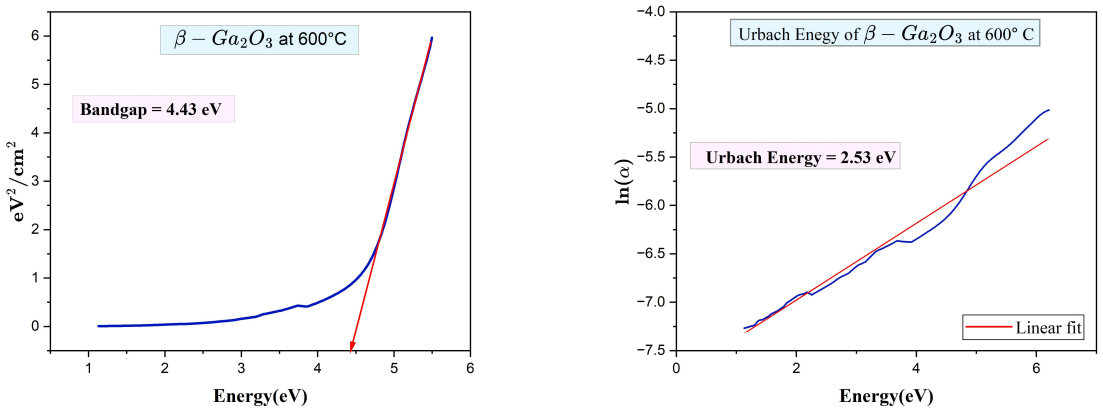

Figure 4.8: Tauc-Plot analysis and Urbach Energy of $\beta - Ga_2O_3$ grown at a substrate temperature of 600°C.

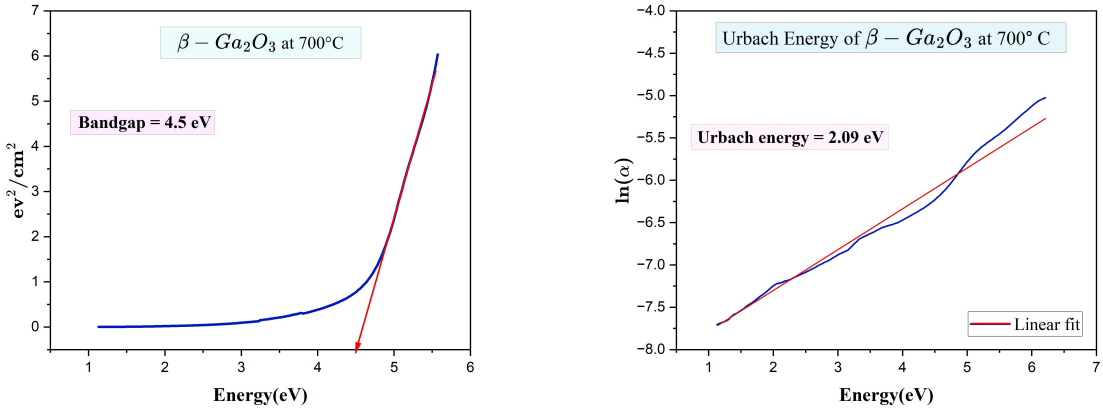

Figure 4.9: Tauc-Plot analysis and Urbach Energy of $\beta - Ga_2O_3$ grown at a substrate temperature of 700°C.

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