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## **INFLUENCE OF SUBSTRATE TEMPERATURE ON RF SPUTTERED $MgIn_2O_4$ FILMS**

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### **ABSTRACT**

Optical access in the visible spectrum and large-area electrical contact are two key components of many modern technologies, and both are made possible by transparent conducting oxides (TCO). The characteristics of an oxide may be modified by adding either native or substitutional dopants, allowing for the achievement of both high transparency and the desirable electrical conductivity ( $>10^3 \text{ } \Omega^{-1}\text{cm}^{-1}$ ). The deposition of transparent conducting oxides (TCO) has gained popularity due to its potential use in LEDs, OLEDs, and flat panel displays. The electrical and optical characteristics of the TCO materials employed in their manufacture determine the efficiency and performance of these devices. Increasing the number of charge carriers or the mobility of carriers is one way to improve the electrical characteristics of these substances. Doping the substance increases the concentration of carriers. This is because ionized impurities enhance carrier scattering and free carriers absorb more light, reducing carrier mobility.

**KEYWORDS:** Substrate Temperature, RF Sputtered,  $MgIn_2O_4$  FILMS, transparent conducting oxides

### **INTRODUCTION**

The only approach to boost conductivity in TCO materials without negatively impacting transparency and device performance is to boost mobility. extending the relaxation period by extending the time between consecutive scattering events of the free electrons or decreasing the effective mass of the electron might enhance electron mobility. Transparency in the visible spectrum and high electrical conductivity in spinel oxide structures have been shown in several scientific research.  $MgIn_2O_4$ ,  $CdIn_2O_4$ ,  $Cd_2SnO_4$ ,  $CdGa_2O_4$ ,  $ZnGa_2O_4$ ,  $Zn_2In_2O_5$ ,  $Zn_2SnO_4$ , etc. are only a few of the recently found transparent and electroconductive oxides.  $MgIn_2O_4$  stands out due to its great transparency and strong

electrical conductivity. As a result, this chapter discusses the measures taken to optimize the deposition conditions in order to acquire acceptable electrical and optical characteristics while creating high-quality  $MgIn_2O_4$  thin films by the RF sputtering process. Structure, surface morphology, composition, as well as optical and electrical characteristics all contribute to MIO films' device performance. These material characteristics also call for a thorough examination of processing methods.

### **PREPARATION OF $MgIn_2O_4$ THIN FILMS**

The RF magnetron sputtering method was used to create the MIO thin films on glass substrates. To achieve uniform, pin hole free, well adhered, clear films, the film

preparation parameters of RF magnetron sputtering, such as target to substrate distance, argon flow, were tuned. To get films with sufficient characteristics for use in devices, factors like substrate temperature and RF power are adjusted. Therefore, MIO films were deposited at room temperature, 1000C, 1500C, 2000C, and 2500C to examine the impact of substrate temperature on the film characteristics. In 50W increments, the RF power was increased to a maximum of 200W. Structure, surface morphology, composition, thermal, optical, and electrical characterization findings of MIO films as a function of substrate temperature are discussed in this chapter.

Using a PANalytical X'Pert PRO multifunctional diffractometer with CuK radiation ( $\lambda=1.5418$ ), we have done in-depth structural analyses as a function of substrate temperature. High-resolution transmission electron microscopy (HRTEM) was used to verify the as-prepared MIO films' atomic-level structural characteristics, and SAED patterns were also produced. The films' surfaces were analyzed for morphology using a JEOL JSM 35CF scanning electron microscopy (SEM) and an atomic force microscope (AFM). The Hitachi - 3400 twin beam UV-Vis-NIR spectrophotometer and indigenously integrated photoacoustic spectrometer were used to investigate the films' optical characteristics. The photoacoustic spectrometer was used to investigate the films' thermal characteristics. Ecopia HMS-3000 Hall effect measuring device was then used to conduct electrical characterizations of the samples. The data

is evaluated methodically and compared to previous studies on MIO films.

## **THICKNESS OF THE SPUTTERED $MgIn_2O_4$ FILMS**

It is important to quantify film thickness since it has such a profound effect on the films' physical qualities. Several sputtering parameters, including RF power, substrate temperature, and substrate to target distance, may be adjusted to modify the layer thickness. This research uses the thickness distribution function of the sputtering source, which is protected by a permalloy shield, to determine the optimal distance between the target and the substrate, which is kept constant at 6 centimeters throughout. The substrate temperature and RF power were adjusted to deposit the films. Using a Mitutoyo SJ-301 surface roughness tester, we were able to determine that the thickness of the deposited MIO sheets is anywhere between 0.42 and 0.65 m.

### **1. Effect of Substrate temperature on Thickness**

The layer thickness changes as a function of substrate temperature for a range of RF sputtering powers. For films coated at various substrate temperatures, the film thickness decreases as the substrate temperature rises. The energy of the particle expelled from the target is increased when the substrate is heated from RT to 2500C.

As a consequence, the substrate temperature has increased, slowing the pace at which the add atoms have cooled. It leads to a thinner film as atoms diffuse into the surface of the film. When raising the substrate temperature from RT to 2500C, the sputtering yield of the target material increases directly to the substrate

with decreased collisions with the sputtering gas—Argon atoms, in the case of films coated at 150 W RF power.

## STRUCTURAL CHARACTERIZATION

Improved device performance relies heavily on the ability to characterize the structural properties of thin films. The preparation parameters for RF magnetron sputtered MIO films determine whether the resulting film will be amorphous or crystalline. The preparation procedure and the deposition parameters influence the degree of crystallinity, structural order, and structural transitions. That's why structural characterisation is so important: it sheds light on how a material really behaves. To determine a material's internal structure, X-ray diffraction is an effective method. The RF sputtered MIO films in the current work are structurally characterized using high resolution transmission electron microscope (HRTEM) images with selected area electron diffraction (SAED).

### 1. X-ray Diffraction Analysis

The XRD results demonstrate that the film placed on the glass substrate and kept at RT is in its amorphous or first-stage of crystallization form. It might be because the MIO species on the substrate are not fully formed at the lower temperature. Extremely high orientation along the (311) plane of the cubic spinel phase is found in the film deposited at a substrate temperature of 1500C, with just a little amount of deviation. At higher substrate temperatures, total film coalescence may occur, followed by perfectly aligned particle formation.

Films of magnesium indium have been deposited at various temperatures,

including room temperature (RT), 100 °C, 150 °C, 200 °C, and 250 °C, to examine the impact of substrate temperature on the crystallinity of the resulting film. The XRD patterns of MgIn<sub>2</sub>O<sub>4</sub> films formed at various substrate temperatures. The (311) peak is more prominent in the prepared polycrystalline films. Other peaks (220, 331, 422, 511, 531) originating from the diffraction planes are also apparent, but at lower intensities. The existence of all these peaks, at whatever preparation temperature, definitively verifies the synthesis of single-phase MgIn<sub>2</sub>O<sub>4</sub> with an inverted spinel structure.

The strength of the peaks found for the 150 0C deposited films is comparatively greater than that for the 200 0C and 250 0C deposited films, which is an intriguing observation. Because Mg and In species move into the tetrahedral and octahedral positions of the spinel structure, thick films are formed with increased crystallinity. Additionally, the diffraction planes' measured 'd' spacing is quite near to the norm.

### 2. Transmission Electron Microscopic Analysis

High resolution transmission electron microscopy (HRTEM) paired with a selected area electron diffraction (SAED) pattern corroborated the microstructure of the MIO film deposited at 150 W RF power and 1500C substrate temperature. The film was detached from the substrate and analyzed using HRTEM. The reflection plane of cubic spinel MIO is reflected well, revealing orderly lattice fringes with an interplanar spacing of roughly 0.2222 nm. There is a great degree of perfection inside individual grains of the film, as seen by the length of the lattice

fringes to the top right side of the micrograph. The plane reflections in the SAED pattern indicate that MIO films are polycrystalline. Excellent quality MIO films may be deposited at a substrate temperature of 1500C, as shown by the HRTEM micrograph and SAED pattern.

## **SURFACE MORPHOLOGICAL STUDIES ON MgIn<sub>2</sub>O<sub>4</sub> FILMS**

Another crucial aspect of TCO research is the examination of the material's surface morphology. The TCOs' surface resistance is proportional to their thickness. Therefore, the deposited films need to be homogeneous. In order to avoid the localized field effect and surface scattering, TCOs also need to have a smooth surface [1]. Analyzing the surface morphology of transparent conducting MIO films is crucial for learning about their viability and applicability. Therefore, a JSM 35CF JEOL SEM and a Nanoscope E-3138J AFM were used to examine the surface morphology of the MIO films. The analytical findings are presented in depth in the sections that follow. Key factors in determining the performance of the films for their suitability to develop various special devices are the host material's structure, optimum stoichiometry, and surface morphological changes, etc., which must be taken into account when preparing thin films for device fabrication. We looked at the MgIn<sub>2</sub>O<sub>4</sub>'s surface morphology at different deposition temperatures using scanning electron microscopy (SEM) and atomic force microscopy (AFM).

### **1. Scanning Electron Microscopic (SEM) Studies**

The surface morphology of the RF magnetron sputtered MIO thin films was

studied using a scanning electron microscope (SEM), which may provide information on topographical characteristics, morphology, phase distribution, crystal structure, and crystal orientation. Films were deposited using 150W of RF radiation at room temperature, 1000C, 1500C, 2000C, and 2500C, and SEM pictures were taken at each temperature to see how the surface morphology changed with each. SEM images of MgIn<sub>2</sub>O<sub>4</sub> films deposited at RT, 100, 150, 200, and 250 0C. The grain size distribution is not homogeneous because of the mixed phase assessment. Surface morphology of MgIn<sub>2</sub>O<sub>4</sub> films is clearly affected by growing circumstances and procedures, as seen in the picture. Microscopically, particles in a MgIn<sub>2</sub>O<sub>4</sub> film formed at room temperature (RT) are all about 96 nm in size. However, the layer produced at 1500C has a higher particle size, on the order of 220 nm. It looks like the sample's surface is made up of grains of tiny pyramidal crystallites that are uniformly distributed over the whole surface. To the extent that SEM resolution permits, the MgIn<sub>2</sub>O<sub>4</sub> films created through RF sputtering seem absolutely flat. Adatoms diffuse more quickly at higher substrate temperatures, speeding up the atomic rate of grain formation. Additionally, at higher temperatures, the films are impacted by thermal gradient, low tension, and shocks, resulting in the creation of an organized settling of crystallites in their preferred positions within the film structure, resulting in a continuous film. The SEM morphological analysis reveals that when the substrate temperature rises, the surface's uniformity, crystallinity, and homogeneity improve.

The XRD findings are corroborated by the observation that the crystalline quality of the films improves with an increase in the substrate temperature.

## 2. Surface Topography by Atomic Force Microscopy (AFM)

The films' morphology was further investigated using atomic force microscopy (AFM). Because of its high resolution; AFM can investigate both individual grains and the spaces between them in three dimensions. The analysis is crucial since the conduction mechanism relies so heavily on the grain and grain border. The relationship between substrate temperature and film surface characteristics has been studied. The growth has a distinct columnar structure, with identically sized and shaped columns throughout. Films deposited at 150 °C have what seems to be a homogeneous top layer with closely packed columns. The specified columns are more widely spaced in the film formed at RT, suggesting a reduced surface density. Typically, increased transmittance is seen in films with bigger column size. Both the RT and 150 °C deposited MgIn<sub>2</sub>O<sub>4</sub> films had RMS roughness values of 12.8 nm and 5.7 nm, respectively. This concludes that films formed at 150 °C are more smoothly prepared than those placed at lower temperatures. This smooth formation may be attributable to the amorphous nature of the films, as shown in AFM pictures of films deposited at ambient temperature. As the substrate temperature rises, the MIO particles in close proximity tend to fuse together, forming a matrix. As the temperature rises, the particles coalesce entirely.

## THERMAL PROPERTIES OF MgIn<sub>2</sub>O<sub>4</sub> FILMS

Transparent conducting oxides used in a wide variety of applications need regular and exact determination of their thermal characteristics, such as thermal diffusivity and thermal conductivity. Heat conduction through a material is mostly dependent on a property called thermal diffusivity. This may be a necessary prerequisite for picking the right material, which in turn ensures the material's dependability and longevity. Due to its dependence on microstructural changes, composition, and processing conditions, thermal diffusivity [3] warrants further investigation. The thermal and optical characteristics of MIO thin films were investigated using photoacoustic spectroscopy. The current article is an effort to investigate the thermal property of the TCOs, despite the fact that the photoacoustic approach has become an essential instrument for the precise assessment of the thermal characteristics of a wide range of materials, notably semiconductors [4, 5].

For MIO thin films formed at substrate temperatures of 100, 150, and 200 °C with 150 W RF power, The existence of thermo elastic bending is shown by the presence of a curvature, as seen in the image. The thermo elastic bending is caused by the much decreased thermal diffusivity of the MIO thin films, which is only possible because the sample thickness is so small. Temperature-elastic deformation also is sensitive to crystallinity [6]. The thermal diffusivity for the MIO films formed at variable substrate temperatures with varied RF power was obtained by fitting the curve to the higher modulation frequency

area, since the Rosencwaig-Gersho theory is valid for the thermally thick regime.

Through the use of photoacoustic spectroscopy, the thermal diffusivity of MIO thin films coated at RT, 100, 150, 200, and 250 °C and with RF power of 150 W was determined with an average accuracy of 0.00321011 m<sup>2</sup>/s. Given that thermal diffusivity rises with rising substrate temperature, the results of photoacoustic depth profiling. The crystallinity of MIO thin films is thought to have a role in the observed rise in thermal diffusivity with increasing substrate temperature [7]. There may be less lattice distortion as the substrate temperature rises and the films become more crystallized [8]. As a consequence, the thermal diffusivity of the samples improves due to a reduction in phonon scattering.

## **OPTICAL PROPERTIES OF MgIn<sub>2</sub>O<sub>4</sub> FILMS**

Electromagnetic waves are absorbed and emitted by thin layers. Films' unique properties often become apparent in their radiation interactions. Information received from studying films often fluctuates since the underlying foundation for the interaction shifts with the wavelength of the light used in the experiment. The interaction also changes depending on the film's morphology. As a result, the structural and optical characteristics of MIO films prepared using RF magnetron sputtering were investigated.

### **Optical Transmittance**

Transparent conducting oxide films need in-depth research on their optical characteristics for practical device focused applications. Transmittance-improving

coatings (TCOs) should have excellent transmittance, as implied by their name. It is widely known that the amorphous content, presence of impurities, voids, dislocations, and lattice trapping centers greatly affect the optical characteristics of thin films. The films must be free of these faults in order to be used in TCO devices. If a film has a high transmittance, it must be devoid of the aforementioned flaws. Using a spectrometer, researchers looked at how much light MIO films let through. The effect of substrate temperature on the optical transmittance of films produced with different amounts of RF power was investigated.

Due to its remarkable material properties, magnesium indium oxide (MgIn<sub>2</sub>O<sub>4</sub>) is now of tremendous interest for optoelectronic applications. MgIn<sub>2</sub>O<sub>4</sub> films produced on glass/quartz substrates showed temperature-dependent changes in optical characteristics between 300 and 1500 nm. The transmittance value is shown to improve with longer wavelengths. The MgIn<sub>2</sub>O<sub>4</sub> films deposited at 150 °C had a visual transparency of around 75% and an IR transmission of 83%. This transmittance value is in good agreement with that of MgIn<sub>2</sub>O<sub>4</sub> films produced using r.f. magnetron sputtering [9, 11] and the pulsed laser deposition method [12]. MgIn<sub>2</sub>O<sub>4</sub> films made at room temperature (RT), 100 degrees Celsius (C), two hundred degrees Celsius (C), and two hundred fifty degrees Celsius (C) have lower transmittance values (about 60 to 70 percent). The scattering at grain borders and the size of the grains are responsible for the dramatic changes in transmittance that occur as a function of the deposition

temperature. In terms of grain size, the surface examination shows that MgIn<sub>2</sub>O<sub>4</sub> films formed at 150 °C are superior. In addition, the basic light absorption drastically reduces the film's transparency in the UV area.

High uniformity of RF magnetron sputtered MIO films is shown by the absence of interference fringes in transmittance spectra of MIO films formed at the desired substrate temperature. An increase in crystallization from an amorphous state [13] may be responsible for the reduction in transmittance values when the substrate temperature is raised.

## CONCLUSION

Thin films of MgIn<sub>2</sub>O<sub>4</sub> have their thermal characteristics analyzed using an in-house developed photoacoustic spectrometer. The films' very low thermal diffusivity argues for their supposed inertness to heat and cold. The films' thermal diffusivity changed as the substrate temperature changed. Thus, by altering the substrate temperature, the films' thermal characteristics may be adjusted. The UV-Vis-NIR transmittance spectra of the produced oxide thin films were examined to determine their optical properties. MgIn<sub>2</sub>O<sub>4</sub> thin films' optical bandgap was calculated using their transmittance curve. Optical constants  $n$  and  $k$  were also computed, with results that were consistent with those published in the literature. MgIn<sub>2</sub>O<sub>4</sub> thin films were measured to have an optical band gap of 3.76 eV and a maximum optical transmittance of 70%. The Hall effect measuring device and a linear four probe configuration were used to take the electrical measurements. The optimized MgIn<sub>2</sub>O<sub>4</sub> thin films were found to have a resistivity of 6.45  $\times 10^{-1}$  cm, a

mobility of 6.9 cm<sup>2</sup>/Vs, and a carrier concentration of 1.40  $\times 10^{18}$  cm<sup>-3</sup>. Finally, the ethanol sensing characterization was performed on MgIn<sub>2</sub>O<sub>4</sub> thin films produced under optimum deposition circumstances. Due to their high conductivity and strong chemical inertness, the MgIn<sub>2</sub>O<sub>4</sub> thin films displayed a moderate reaction to the ethanol vapor, although not particularly high. The films' response to increasing ethanol content was linear. The sensor's reaction time to the ethanol vapor was quite short, and it recovered quickly after being exposed to the vapor.

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