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Physical properties of indium zinc oxide and aluminium zinc oxide thin films deposited by radio-frequency magnetron sputtering

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Abstract

Indium-doped zinc oxide (IZO) and aluminium-doped zinc oxide (AZO) thin films were grown by radio-frequency (RF) magnetron sputtering onto optical glass substrates and their structural, morphological, and optical properties were discussed in terms of varying the sputtering power as 40 W, 60 W, 80 W, and 100 W. No heating substrate or any post-thermal treatment was performed. The structural features were analyzed by grazing incidence X-ray diffraction and revealed the amorphous phase for IZO samples, while the AZO thin films inherited the Wurtzite structure of zinc oxide. The morphological properties were investigated by atomic force microscopy (AFM), in tapping mode and scanning electron microscopy (SEM). The AFM images showed relatively uniform and smooth surfaces for all prepared structures. The optical transmission spectra proved the excellent theoretical values of transparency for metallic oxides in the visible region of the electromagnetic spectrum, i.e. ~60% for IZO and ~70% for AZO. The obtained results showed that even without any thermal treatment the structural, morphological, and optical properties of IZO and AZO thin films prepared by RF magnetron sputtering are similar with those for samples subjected to medium or high temperatures.

1. Introduction

Zinc oxide (ZnO) is an A\textsubscript{II}B\textsubscript{VI} binary compound which exhibits specific semiconducting features, such as n-type conductivity, large exciton binding energy (60 meV) and wide direct band gap (3.35–3.37 eV) at room temperature, and Wurtzite thermodynamically stable phase [1–6]. Moreover, its intense study is also due to the low toxicity and relatively cheap cost material [7] and excellent piezoelectricity and pyroelectricity properties [8, 9], manifested also at nanometric scale[10–13]. Notwithstanding, the pristine ZnO has rather high electrical resistivity because the free charge carriers’ concentration is small [14]. This is the reason why frequently materials from group III, such as aluminium, gallium, and indium, are used as doping elements to improve both its electrical and optical behaviour [15, 16]. Indium-doped zinc oxide (IZO) and aluminium-doped zinc oxide (AZO) are two of the most used metallic oxides for electronic and optoelectronic applications, such as anti-reflection coatings for transparent electrodes [17–21], gas sensors [22, 23], light-emitting diodes [24], photocatalysts [25], piezoelectric transducers [26], varistors, spintronic devices, lasers [27], and photovoltaica structures [28]. Their versatility is strongly related to a large variety of deposition methods; e.g. Lee and collaborators studied the effects of annealing temperature on the optical band-gap of IZO thin films, obtained by radio-frequency (RF) magnetron sputtering [29], Ma and collaborators proposed one-step spinning coating method [2], while Yung-Chen Cheng obtained AZO films by atomic layer deposition [30].
Despite that the atomic radius of indium ions (In$^{3+}$, 0.80 Å) is larger than that of zinc ions (Zn$^{2+}$, 0.74 Å), the mixture of these two leads to specific optoelectronic properties and make the IZO compound very sensitive to the cationic coordination [31, 32]. In the case of AZO, the atomic radius of aluminium ions (Al$^{3+}$, 0.53 Å) is smaller than that of Zn$^{2+}$, so ZnO lattice will diminish [11, 33]. Besides, the substitution of Zn$^{2+}$ with Al$^{3+}$ forms shallow donor levels which improve the electrical conductivity of AZO material.

This paper explains the inter-relation between the structural, morphological, and optical characteristics of IZO and AZO thin films, obtained by RF magnetron sputtering. The samples were fabricated without heating the substrate or any post-thermal treatments. This approach is motivated by the fact that the electrochromic devices and polymer photovoltaic structures are sensitive to high temperatures [34, 35].

2. Experimental procedures

Indium zinc oxide (IZO) and aluminium zinc oxide (AZO) thin films were deposited onto optical glass, by RF magnetron sputtering. The equipment used for the deposition was from Tectra Company, accommodating one sputtering gun and the used targets were commercially available from Kurt J. Lesker Company (AZO) and FHR Company (IZO), respectively, and were used without further purification. Their purity was 99.99% and the ratio of the compounds was In$_2$O$_3$:ZnO 90:10, wt% and Al$_2$O$_3$:ZnO 2:98, wt%, respectively. The working gas was Argon 5.0 and the substrate-to-target distance was 11 cm. All glass substrates were subsequently cleaned in acetone and distilled water for 15 min, into an ultrasonic bath, prior to the deposition process. Working pressure, deposition time and the distance between target and samples were maintained constant, while the sputtering power was varied as 40 W, 60 W, 80 W, and 100 W. All fabrication parameters are summarized in Table 1.

![Figure 1. GIXRD patterns of fabricated IZO films by RF magnetron sputtering.](image)

### Table 1. Fabrication parameters of IZO and AZO thin films, obtained by RF magnetron sputtering.

| Sample | Sputtering Power (W) | Working Pressure (Pa) | Deposition Time (min) | Target diameter (inch) |
|--------|----------------------|-----------------------|-----------------------|------------------------|
| IZO1   | 40                   | 0.53                  | 30                    | 1                      |
| IZO2   | 60                   |                       |                       |                        |
| IZO3   | 80                   |                       |                       |                        |
| IZO4   | 100                  |                       |                       |                        |
| AZO1   | 40                   | 0.53                  | 30                    | 2                      |
| AZO2   | 60                   |                       |                       |                        |
| AZO3   | 80                   |                       |                       |                        |
| AZO4   | 100                  |                       |                       |                        |

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The X-ray diffraction patterns were taken in grazing incidence geometry (GIXRD) at an angle of 1.5°, in the ranges of 2θ = 15° – 70°, using a Bruker D8 Discover diffractometer operating at 40 KV and 40 mA (CuKα source with λ = 1.5406 Å), at room temperature. This type of investigation is preferred to be used for the characterization of thin films because the path of incident X-ray photons through the samples is increased.
Moreover, in this way the signal of the substrate is strongly reduced. The grain size ($D_g$), the micro-strain ($\langle \varepsilon^2 \rangle^{1/2}$) and the lattice constants ($c$, $a$) were determined by analysing the largest diffraction peak fitted by Voigt profile.

The atomic force microscopy (AFM) analysis was performed in tapping mode, using an A.P.E Research Company equipment, at room temperature, and the scanning electron microscopy investigations with a JEOL 7600F Field-Emission Scanning Electron Microscope (FE-SEM) equipped with an energy dispersive X-ray (EDX) analyzer. The EDX spectroscopy was employed to qualitatively and quantitatively confirm the elemental composition of the samples. The optical transmission spectra were acquired in ranges of 300 nm–2500 nm, at room temperature, too, using a Perkin Elmer Lambda 750 spectrometer. When theory holds, the thickness of samples was determined by building the interference envelope functions, otherwise by X-ray reflectometry measurements (XRR) using same equipment as for GIXRD.

**Figure 2.** The recorded profiles of (222) peak in Bragg-Brentano theta-theta geometry and the curve obtained by fitting with Voigt profiles, for IZO fabricated films. The labels (a), (b), (c), and (d), respectively, are assigned to IZO1, IZO2, IZO3, and IZO4 samples.

**Table 2.** Structural parameters of prepared IZO films, evaluated from the recorded profiles of (222) peak in Bragg-Brentano theta-theta geometry.

| Sample  | $D_g^{(222)}$ (nm) | $\langle \varepsilon^2 \rangle^{1/2}$ | $a$ (Å) |
|---------|-------------------|--------------------------------------|---------|
| IZO1    | 2.3               | 4.36·10^{-2}                         | 9.65    |
| IZO2    | 2.36              | 4.26·10^{-2}                         | 9.70    |
| IZO3    | 2.5               | 4.02·10^{-2}                         | 9.71    |
| IZO4    | 2.25              | 4.05·10^{-2}                         | 9.67    |
3. Results and discussion

3.1. Results for IZO thin films

The GIXRD patterns of fabricated IZO thin films are shown in figure 1, and the recorded profile of (222) peak in Bragg-Brentano theta-theta geometry for samples IZO1, IZO2, IZO3, and IZO4 is presented in figure 2.

As one can easily notice, the grown IZO films are amorphous, despite that the thickness increases. Because the substrate was intentionally not heated, the crystalline structure of samples is very poor. This behaviour was expected because IZO is thermally stable at temperatures up to 400 °C–500 °C [36, 37]. The large peak around 34° can be interpreted as an overlap between the (222) characteristic cubic plane from In2O3 and (002) from ZnO. The structural parameters of fabricated samples were calculated and are presented in table 2; the obtained values are similar.

The morphology of the surface of prepared IZO samples investigated by AFM is shown in figure 3. One may observe that the surface of all fabricated samples is uniform and smooth, no cluster being formed during the deposition process. This remark is validated by the very small values of root mean square roughness (RMS). Also, Skewness (Skew) and Kurtosis (Kurt) parameters were determined and were summarized in table 3.

Table 3. Calculation of specific parameters of topography (root mean square roughness - RMS, Skewness and Kurtosis) for IZO fabricated films by RF magnetron sputtering. The thickness of samples is also mentioned.

| Sample | RMS (nm) | Skew | Kurt | Thickness (nm) |
|--------|----------|------|------|----------------|
| IZO1   | 1.12     | 0.87 | 1.32 | 286            |
| IZO2   | 1.19     | 0.90 | 1.95 | 587            |
| IZO3   | 1.08     | 0.89 | 1.62 | 781            |
| IZO4   | 1.02     | 0.77 | 1.03 | 959            |

The RMS is an overall measure of the texture of the surface and it is calculated by the formula:

$$\text{RMS} = \sqrt{\frac{1}{l} \sum_{0}^{l} Z^2(x)} \ dx$$

where $l$ is the length of scanned area, $Z(x)$ is a function assigned to the height of the surface relative to the best fitting plane, and $x$ is the position [38]. Skewness and Kurtosis parameters

![Figure 3. AFM analysis of fabricated IZO films, obtained in tapping mode. For all samples the scanned area was 10 μm × 10 μm.](image)
indicate the symmetry of the analyzed surface and are evaluated using the below expressions.

\[
\text{Skew} = \frac{1}{RMS^3} \int \int (Z(x, y))^3 \, dx \, dy
\]

\[
\text{Kurt} = \frac{1}{RMS^4} \int \int (Z(x, y))^4 \, dx \, dy
\]

**Figure 4.** Scanning electron microscope (SEM) closed-up micrographs showing cross-sectional views of the glass substrates coated with IZO films sputtered at an RF plasma power of (a) 40 W, (b) 60 W, (c) 80 W, and (d) 100 W. The inset of (d): corresponding low magnification section view of the IZO-coated glass substrate. (e) EDX analysis of the IZO films.

**Figure 5.** Transmission spectra of obtained IZO samples, acquired in the ranges of 300–2500 nm, at room temperature, together with the spectrum for glass substrate.
For grown IZO films, despite the working power variation, no significant changes of RMS are observed. Moreover, because the values of Skewness parameter are larger than 0, the surface of samples is dominated by peaks, while the smaller values of 3 of Kurtosis parameter indicate that no extreme peaks or valleys are present.

The cross-section SEM micrographs of fabricated IZO thin films are presented in figure 4, together with their corresponding EDX spectra. The EDX patterns indicate the presence of silicon (Si), oxygen (O), indium (In), and zinc (Zn) elements, with neglected impurities concentration, and the elemental calculations described well the coexistence of both, the supporting glass and IZO film. Noteworthy, excluding the contribution coming from the glass support (SiO$_2$), the elemental analysis table suggests also an excellent stoichiometry of the IZO film, as the calculated values for the elemental components were obtained in the expected atomic ratios for ZnO and In$_2$O$_3$, respectively.

The optical transmission spectra of prepared IZO films are presented in figure 5. Against their thickness, IZO films have relatively good optical transmission, larger than 60% in the visible range. Because IZO is a direct band gap semiconductor, the values of forbidden energy band were determined using the well-known formula: $a h \nu = A(\nu - E_g)^{1/2}$, in which $A$ is a constant, $h \nu$ is the energy of the incident photons, and $E_g$ is the value of the band gap. The calculated values of $E_g$ by optical spectroscopy are reported in table 4.

The obtained values of $E_g$ are smaller than those reported in literature, e.g. 3.75–3.79 eV [30, 39] for IZO, and we admit that the differences between prepared samples are just noticeable. Nevertheless, a decrease of band gap with the increase of thickness can be observed. This behaviour was also noticed for some $A_2B_6V_4$ compounds [40, 41], and is associated either with the reduction of strain in the films or with the enlargement of size of the grains [42] according with quantum confinement process. For our samples, by correlating the GIXRD results, we concluded that both assumptions are appropriate.

### 3.2. Results for AZO thin films

The GIXRD patterns of fabricated AZO thin films are shown in figure 6, while the recorded profiles of (002) peak of AZO1, AZO2, AZO3, and AZO4 samples are presented in figure 7. Same equipment and parameters were used as for IZO layers analysis.
The fabricated AZO layers are polycrystalline and they inherited the Wurtzite structure of zinc oxide \( \text{ZnO} \).

As can be easily observed, the grains are preferentially located on \((002)\) direction and, also, the \((103)\) peak becomes more pronounced by increasing the thickness. Quantitative data of crystalline structure have been measured in Bragg-Brentano theta-theta geometry for the highest diffraction peak \((002)\) to avoid the additional bordering of peaks from GIXRD. For each fit of samples, the residuals between experimental information and theoretical analysis are presented at the bottom of the graphs.

In table 5 are shown the calculated values for grain size \( D_{\text{eq}} \), micro-strain \( \langle \varepsilon^2 \rangle^{1/2} \) and lattice constants. All samples have small crystallite size and AZO2, AZO3 and AZO4 have similar values for mean-square strain and grain size. Due to the low deposition power used for AZO1, the sputtered atoms had more time to achieve an equilibrium position in crystal structure and so are forming larger crystallites and structural defects along \((002)\) orientation. At 100 W power deposition is observed a low decrease in the accumulated strain with slightly larger

**Table 5.** Structural parameters of prepared AZO films, evaluated from the recorded profiles of \((002)\) peak in Bragg-Brentano theta-theta geometry.

| Sample | \( D_{\text{eq}} \) (nm) | \( \langle \varepsilon^2 \rangle^{1/2} \) \( (\varepsilon^2)_{1/2} \) | \( a (\text{Å}) \) | \( c (\text{Å}) \) |
|--------|-----------------|-----------------|-----------------|-----------------|
| AZO1   | 11.25           | 8.45 \( \times \) \( 10^{-3} \) | 3.24            | 5.30            |
| AZO2   | 6.34            | 1.49 \( \times \) \( 10^{-2} \) | 3.21            | 5.25            |
| AZO3   | 4.38            | 2.15 \( \times \) \( 10^{-2} \) | 3.21            | 5.25            |
| AZO4   | 4.96            | 1.91 \( \times \) \( 10^{-2} \) | 3.23            | 5.27            |

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grain size mostly because the film has reached a thickness for which the crystalline structure is more stable. Similar results were reported by A. Pruna and collaborators [16]. No Al₂O₃ phase was identified and this can be associated with the substitution of Zn atoms by Al ones or with the segregation of Al and the formation of Al-O bonds [43, 44].

The topography of the surface of prepared AZO thin films was analysed by AFM, in tapping mode, and the results are presented in figure 8. As one can notice, the surface of AZO1 contains clusters of less than 1 μm dimension, most likely formed during the deposition process due to the low sputtering power that lead to small values of kinetic energy when the atoms reached the substrate. Despite these clusters, the overall RMS for scanned area is 1.06 nm. Moreover, by increasing the thickness a slightly increase of roughness was observed. The calculated RMS, Skewness and Kurtosis parameters for AZO grown samples are summarized in table 6.

Because the values of Kurtosis parameter for AZO3 and AZO4 samples are larger than 3, this indicates extreme differences between valleys and peaks. This observation is validated by the increase of Skewness parameter, showing that from a surface dominated by peaks, for AZO1 and AZO2 films, an easy passage to surfaces containing a mixture of valleys and hills, for AZO3 and AZO4 samples, appeared. We assume this behaviour is strongly related to the increase of thickness of fabricated AZO thin layers.

The cross-section SEM micrographs of fabricated AZO films, together with their corresponding EDX pattern are shown in figure 9.

The EDX analysis of AZO films indicates the only presence of constitutive elements, i.e. oxygen (O), silicon (Si), zinc (Zn), and aluminium (Al). Similarly as for IZO samples, the quantitative elemental analysis of AZO films demonstrates a very good compositional quality of the AZO-coated substrate in terms of atomic ratios.
associated with $\text{ZnO}$ and $\text{Al}_2\text{O}_3$. As a general remark, the obtained EDX spectra include $\text{In}$ and $\text{Al}$ peaks demonstrating that the elements have been successfully accommodated into the $\text{ZnO}$ matrix during the sputtering process.

The optical transmission spectra of AZO fabricated thin films are shown in figure 10, altogether with that of optical glass substrate.

All obtained samples have good transparency, larger than 70% in the investigated spectral domain, i.e. 300 nm—2500 nm. As expected, the aluminium doping $\text{ZnO}$ does have neglected effects on the transparency of
grown AZO films, especially in the visible region of electromagnetic spectrum, but reduces the optical band gap [43]. AZO samples are direct energy band gap semiconductors, so the optical band gap values were calculated as for IZO films (see table 7).

The AZO thin films inherited the crystalline structure and high transparency of ZnO in the visible range and the replacement of zinc atoms with aluminium ones drove to a reduction of forbidden energy gap due to a shift of Fermi surfaces to conduction band [45]. As a general observation, we may notice small differences for the values of the optical band gap of prepared samples.

4. Conclusions

Metallic oxides based on ZnO, i.e. IZO and AZO, were grown by RF magnetron sputtering onto optical glass substrates. All fabrication parameters were maintained constant but the sputtering power was varied as 40 W, 60 W, 80 W, and 100 W. Also, no thermal treatments were performed during or after the samples’ preparation. The GIXRD analysis indicated the amorphous phase for IZO samples and the Wurtzite crystalline structure for AZO ones. Due to their amorphous phase, their calculated values of grain size and micro-strain are similar. For AZO thin films this observation is valid only for those structures obtained at 60 W, 80 W, and 100 W. The AFM images carried out in tapping mode revealed a relatively smooth and uniform surface for all prepared samples, with average values of root mean square roughness of 1.10 nm for IZO and 1.97 nm for AZO. The EDX analysis demonstrated that both indium and aluminium were embedded into the ZnO matrix, and the concentration of impurities can be neglected.

The transmission spectra acquired in the ranges of 300–2500 nm proved that these kinds of materials are suitable for optoelectronic applications due to their high transparency in the visible and near-infrared spectral domains, e.g. ~60% for IZO and ~70% for AZO. As a general conclusion, this study pointed out that the fabricated IZO and AZO layers have good structural, morphological and optical properties, many times similar with those subjected to thermal treatments, but of course finding new cheaper procedures of specific thermal treatments could further improve the properties of these films very useful in transparent electronics and optoelectronic devices.

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