ELABORATION AND CHARACTERIZATION OF ZNO THIN FILMS STRUCTURAL AND OPTICAL STUDY

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Abstract. The Nano-topographical structure of the solid surface is known as a necessary parameter in the physicochemical characterization and wetting properties. In this study, the physicochemical properties are evaluated by calculating the surface energy and by measuring the contact angle. The structural proprieties were determined using XRD. The optical proprieties were studied using the UV-visible technics. Substrates used in this study are the zinc oxide thin films deposited on the glass by sputtering under different powers (150, 200 and 250 watt). The Nano-topographic properties were examined using the atomic force microscopy (AFM) in order to calculate the roughness of different substrates. As results, the images obtained by atomic force microscopy showed that the growth of the power causes the growth of the roughness. XRD diagram assessment revealed that the deposited films have a preferential crystallographic direction according to the (002) plane while maintaining the initial orientation. The optical characterization showed that the bandwidth of these films is in the order of 3.28 eV. It is interesting to mention that the increase in RF power has slightly increased the energy of gap.

1. Introduction

The nanotechnology, or generally the nanosciences including microelectronics, and biomedical are the fields of application of zinc oxide (ZnO) [1,2]. ZnO is a photocatalyst semiconductor material widely studied and used because of the advantages that present as availability, low cost, chemical
stability, abundance and nontoxicity [3-5]. Zno is known by its characteristics as the band gap that is about 3.37 eV, the binding energy that is 60 meV, an emission that is close to the UV and a thermal stability that is high [6]. The lifetime and the recombination rate of the Zno electrons allow it to be an attractive material for photocatalytic applications.

The properties of nanostructured ZnO are related to the size and shape of the nanostructure, its purity, and the technique and parameters of the elaboration [7-10]. There are several methods to elaborate ZnO, and we can quote the chemical vapor deposition, thermal evaporation, epitaxy, laser ablation, sol-gel... for example solvothermics is used in paper [11], hydrothermal reaction in the work [12], microwave radiation in the study [13], combination of ultrasound and microwave in paper [14] and sol-gel method in [15]. The Zno elaboration techniques and the associated conditions are not the only ones that have an impact on its properties. The works of [16-19] have shown that the photocatalytic activity in the visible region of the solar spectrum can be improved by adding other chemical elements such as Co, Cu, Fe, Cr, Ag, and Sm.

In this work, we have elaborated ZnO thin layers by sputtering technique. The characterization is done using X-ray diffraction (XRD) for structural study, while the atomic force microscopy (AFM) is used for microstructure observation, UV-visible spectroscopy and contact angle measurement technique are applied for optical property study and surface energy calculation, respectively.

2. Experimental

2.1. Elaboration of thin layers of zinc oxide by RF sputtering under different powers

ZnO thin films were prepared by RF sputtering in an ALCATEL SC451 deposition system equipped with an ALCATEL ARF 601 RF generator operating at 13.56 MHz. These films were deposited using a high purity metal target Zn with a diameter of 10 cm in a mixture of argon gas oxygen Ar / O2 with 30% oxygen. Before the deposition, the glass substrates were ultrasonically cleaned with acetone and isopropyl alcohol to remove any impurity and rinsed with distilled water. Then, the substrates were mounted at a distance of 10 cm above the Zn target (5 N: purity, diameter: 10 cm). The vacuum chamber was evacuated to get a final pressure of 1.5.106 bar. The target was pre-repassed for 10 minutes, and the substrates were not intentionally heated during deposition. These parameters were obtained from a previous optimization of the deposition conditions. The sample was cut into 1cmx1cm coupons cleaned and rinsed with distilled water.

2.2. Characterization of ZnO thin films

2.2.1. X-ray diffraction. The X-ray diffraction is a non-destructive structural analysis technique that characterizes the crystalline nature of the deposits and controls their crystallographic quality. The
information obtained by this analysis includes the crystalline orientation of a deposit made on a substrate or obtained in powder form.

The principle is based on the diffraction of monochromatic x-rays by the atomic planes of the crystals of the studied material. Diffraction occurs only in crystallized materials and when the Bragg relationship is verified:

\[ 2d_{hkl} \sin \theta = n\lambda \]  

Here, \( d_{hkl} \) is the interplanar distance, being the distance separating the index planes (hkl). \( \theta \) is the angle of incidence of X-rays on the surface of the studied material. \( n \) is the order of refraction and \( \lambda \) is the wavelength of the X-ray beam.

To obtain the value of the different interplanar distances, it is sufficient to determine the values of \( \theta \), for which the material diffracts. X-ray diffraction experiments provide a list of diffraction peaks. Each peak corresponds to a family of reticular index plane (hkl). The identification of the phase and its mesh parameters are then carried out by a comparison with existing material databases (JCPDS sheets [20]). The calculation of the size of the crystals is done using Scherrer's method [21] based on the enlargement of the diffraction peaks due to the size of the crystallites:

\[ D = \frac{k\lambda}{\beta \cos \theta_{hkl}} \]  

\( D \) is the Mean diameter of crystallites in the direction perpendicular to the planes (hkl). \( K \) is the Form factor, which is equal to 0.9 for crystallites of isotropic or quasi-isotropic form, \( \lambda \) is the wavelength of the X-rays. \( \beta \) represents the half-height width observed in the diffraction diagram and \( \theta_{hkl} \) is the Diffraction angle of the (hkl) ray.

In this work, X-ray diffraction analyses were performed using a Philips X'Pert MPD diffractometer connected to a microcomputer. The X-ray beam is generated by a copper anti-cathode of wavelength \( \lambda = 1.54056 \) Å, using the radiation Kα in the angle range 2θ between 20° and 60°.

2.2.2. **UV Spectrophotometry - VIS – NIR.**

Spectrophotometry provides information on the optical properties of the sample to be analysed, such as light transmission and absorption, and optical gap estimation. It can also inform us, in some cases, about the thickness of the sample and even go back to its optical constants [22].

The principle of this technique is based on the interaction of the light emitted by the sample to be analyzed. A portion of the incident beam will be absorbed or transmitted by the sample. When a substance absorbs light in the ultraviolet and visible range, the absorbed energy causes disturbances in the electronic structure of atoms, ions or molecules. One or more electrons absorb this energy to jump from a low energy level to a higher one.
In this work, the experimental spectra of the samples are measured using a UV - VIS - NIR spectrophotometer of the Shimadzu UV-3101PC type.

2.2.3. Atomic force microscopy.

Atomic force microscopy (AFM) is a probe scanning imaging technique that maps the interactions between a fine tip and a supported surface or objects. Many parameters (shape and peak nature, range of interaction forces, etc.) influence the resolution. Although point atomic defects can be seen, the technique is widely used at lower resolution. The range of acquisition modes and observable samples are very rich as well.

In this work, we used the Nanosurf Easyscan 2 AFM in order to investigate the topography of the samples.

2.2.4. Surface energy. The nature of the surface of a material is related to the chemical properties of the molecules present on the surface. A surface is only rarely of the same nature as the constituent present in the volume. The surface energy of a solid is related to the intermolecular interactions that characterize the attractive and repellent aspect of a surface. The contact angle technique is one of the techniques commonly used to measure surface energy. The contact angle is defined as the angle between the tangent to the liquid-vapour interface and the tangent to the solid interface on the contact line between the three phases. The equation that connects the contact angle and the solid-vapour interface, the solid-liquid interface energy and the liquid-vapour energy is called Young's equation given by [23].

\[ \gamma_{LV} \cos \theta_Y = \gamma_{SV} - \gamma_{SL} \]  

\( \gamma_{LV} \), \( \gamma_{SV} \) and \( \gamma_{SL} \) represent the liquid-vapor, solid-vapor, and solid-liquid interfacial tensions, respectively, and \( \theta_Y \) is the contact angle.

The free energy of a surface cannot be measured directly. Thus, the use of test liquids is essential. The liquids must be deposited on the solid surface. Van OSS and CHAUDHURY exploited acid-base theory and proposed a model for calculating surface energy [24]. This model takes into account molecular interactions via electron donor / acceptor processes that involve the complementary properties of liquid and solid. The surface tension of the solid is decomposed into two contributions. The first is that resulting from the forces of Lifshitz-van der Waals, noted \( \gamma_{SW} \), and the second noted \( \gamma_{AB} \) is derived from acid-base interactions with \( \gamma_{AB} = 2(\gamma^s_{s} \gamma^s \gamma^s_{l})^{1/2} \). The components of surface energy are determined by measuring the contact angle using three probe liquids (one non-polar and two polar) with known concentrations.
\[ \gamma_L (\cos \theta_Y + 1) = 2(\gamma_{LW}^L \gamma_{SW}^L)^{1/2} + 2(\gamma_S^+ \gamma_L^-)^{1/2} + 2(\gamma_S^- \gamma_L^+)^{1/2} \] (4)

\( \theta_Y \) is the contact angle measured, \( \gamma_{LW}^L \) is the der Waals Van free energy component, \( \gamma^+ \) is the electron acceptor component, \( \gamma^- \) component is the electron donor and the indexes (S) and (L) denote solid surface and the liquid phase, respectively.

To measure the contact angle, we used the GBX Digidrop Goniometer. The chosen liquids cover a wide range of surface tensions. Some are apolar (diiodomethane and 1-bromonaphthalene) and others polar (water, formamide and dimethylsulfoxide (DMSO)). Water and formamide are the most polar.

3. Results and discussion

3.1. X-ray diffraction

As shown in Figure 1, the X-ray diffraction patterns of the samples reveal only one peak, which appears at an angle close to 34.1°. This applies to all three deposition powers. This peak corresponds to the plane (002) of the hexagonal Wurtzite mesh of ZnO. Therefore, the layer grows perpendicular to the plane of the substrate, following a preferential crystallographic direction. This shows that the thin layers of ZnO have a preferred growth orientation in the direction (002). In addition, it can be seen that no other diffraction peaks appear with the increase in deposition power. The initial preferential orientation is therefore not changed when the power is increased. Crystallite size ranges from 4.3 nm to 6.2 nm. The thin layer produced under 200 watts shows a maximal size.

![Figure 1. ZnO X-ray diffraction spectra prepared under different powers (150, 200, 250 Watt), with 30% oxygen](image-url)
Figure 2 shows the evolution of crystallite size as a function of deposition power. It is noted that crystallite size varies when RF power varies. The obtained ZnO thin films have an average crystallite size ranging from 4.3 nm to 6.2 nm. The thin layer produced under 200 watts shows a maximal size.

3.2. Optical properties

We performed measurements of the UV-Visible transmittance to determine the optical gap of ZnO thin films. The optical transmission was measured as a function of the incident wavelength, ranging from 300 nm to 2000 nm.
Figure 3 shows the optical transmission of ZnO thin films. It can be seen that the samples have an average transmission of around 90% in the visible range. The abrupt drop in transmission for wavelengths below 380 nm corresponds to the absorption in ZnO due to the transition between the valence band and the conduction band. The determination of the optical gap is based on the model proposed by Tauc [25], where $E_g$ is related to the absorption coefficient $\alpha$ by:

$$\alpha \cdot h \nu = A(h \nu - E_g)$$

(5)

$A$ is a constant reflecting the degree of disorder of the amorphous solid structure, $E_g$ is the optical gap expressed in eV, $h \nu$ is the photonic energy in eV.

By plotting $(\alpha h \nu)^2$ against $h \nu$, the value of the optical gap $E_g$ can be extrapolated to $(\alpha h \nu)^2 = 0$, as shown in the example presented in Figure 4.

Figure 3. Optical transmission spectra of ZnO films deposited under different powers.
The bandwidth of ZnO thin films obtained at 150, 200 and 250 is 3.24, 3.23 and 3.21 eV respectively. This shows that the value of the optical gap has been slightly increased with the increase in the power of the deposition.

3.3. Morphological properties

The surface of thin films of zinc oxide with different topographies of the samples used in this study is characterized by atomic force microscopy (AFM). The images obtained from the three samples are presented in Figure 5.

The variation in the deposition power of zinc oxide gives different topographies. Using advanced image analysis by the Esayscan 2 software of the Nanosurf Flex AFM device, the surface properties of the different substrates can be described by a set of roughness parameters. Selected numbers of calculated roughness parameters for the samples are listed in Table .1 and table .2. The RMS roughness (Ra and Sa) and the absolute height (Ry and Sy) increase with the increase in power.
Figure 6 shows this increase, the surface of the glass without ZnO deposition has the lowest roughness values compared to the surfaces of ZnO films. In addition, Figure 6 shows the influence of the power of the deposition of ZnO thin films on roughness parameters. The topography is well modified and the roughness values increase with the increasing power.

This means that the power of Zinc oxide deposition by RF sputtering is a paramount parameter that directly influences the structure and topography of the surface of the substrates. Thus, the grain size of the samples increases with increasing power. This gives different topographies and roughness values.

**Figure 5.** 2D and 3D AFM images of the thin layers of zinc oxide produced at different powers.

| Substrat | Ra (nm) | Rq (nm) | Ry (nm) | Rp (nm) | Rv (nm) | Rm (nm) |
|----------|--------|--------|--------|--------|--------|--------|
| ZnO 150  | 1.3277 | 1.6936 | 9.4731 | 5.0682 | -4.4049| 0.039171|
| ZnO 200  | 2.5322 | 3.1752 | 15.087 | 8.267  | -6.8197| 0.17348 |
| ZnO 250  | 5.0135 | 2.6446 | 1.3859 | 7.0664 | -6.7923| -0.90649|
Table 2. Surface roughness of ZnO films

| Substrat | Sa (nm$^2$) | Sq (nm$^2$) | Sy (nm$^2$) | Sp (nm$^2$) | Sv (nm$^2$) | Sm (nm$^2$) |
|----------|-------------|-------------|-------------|-------------|-------------|-------------|
| ZnO 150  | 2.3629      | 3.07587     | 29.388      | 20.652      | -8.736      | 0.019989    |
| ZnO 200  | 4.8403      | 6.9602      | 84.317      | 63.275      | -21.041     | 0.019989    |
| ZnO 250  | 7.6511      | 10.985      | 121.09      | 81.928      | -39.159     | 0.01998     |

Figure 6. Comparison between Ra and Sa roughness of glass and ZnO films.

3.4. ZnO thin films surface energy

The contact angle measurements were carried out using a goniometer (GBX instruments, France) according to the sessile drop sampling method.

We used diiodomethane and 1-bromonaphthalene as liquids. They are apolar, and water, formamide and dimethylsulfoxide (DMSO) are polar, knowing that water and formamide are the most polar. Water is used as a reference. Van Oss, Chaudhury and Good chose $\gamma_{\text{water}}^+ = \gamma_{\text{water}}^- = 25.5$ mN/m values [26]. The different values of the components of the liquids that we used are collated in Table 3.
Table 3. The values of the surface tensions of the liquids used and their components [26].

| Liquid       | Surface energy component (mJ / m²) | \( \gamma_L \) | \( \gamma_{lw} \) | \( \gamma_{AB} \) | \( \gamma_{D} \) | \( \gamma_{L} \) |
|--------------|-------------------------------------|----------------|----------------|----------------|----------------|----------------|
| Water        |                                    | 72.8           | 21.8           | 51.0           | 25.5           | 25.5           |
| Formamide    |                                    | 58.0           | 39.0           | 19.0           | 2.28           | 39.6           |
| Diiodomethane|                                    | 50.8           | 50.8           | ≈ 0            | ≈ 0            | ≈ 0            |

The hydrophobicity of one or more given materials can be defined as a function of the variation of the free energy of interaction between two fragments of this material immersed in water (w). The free energy comprises a polar component (AB) and a non-polar component (LW). The variation of the total free energy is given by [27]:

\[
\Delta G_{iwL} = -2\left[\left(\gamma_{lw}^L\right)^{1/2} - \left(\gamma_{lw}^L\right)^{1/2}\right) + 2\left(\gamma_{lw}^L\gamma_{lw}^L\right)^{1/2} - \left(\gamma_{lw}^L\gamma_{lw}^L\right)^{1/2}\right]
\]

When the value of \( \Delta G_{iwL} \) is negative (the free energy of interaction between molecules is attractive), it means that the solid surface has less affinity for water than between it. Then, it has a hydrophobic character. However, the solid surface is hydrophilic when this value is positive (\( \Delta G_{iwL} > 0 \)).

Table 4 shows the different components of the surface energy of ZnO thin films calculated by acid-base theory using the surface tension components of liquids given by VCG.

Table 4. Contact angle values and surface energy components.
The surface energy value varies between 45.21 mJ/m² and 40.7 mJ/m² for ZnO films. For the glass surface the surface energy value is 50.1 mJ/m². The water contact angle values on the substrates of the ZnO film are greater than 65° (θ water, ZnO 200 w = 78.4°) and on the glass surface θ water = 34.2°. As explained by Van Oss, the free energy of interaction (ΔG_{iw}) is negative for the substrates of the ZnO film, the degree of hydrophobicity varies between ΔG_{iw} = -36.61 mJ/m² and ΔG_{iw} = -44.97 mJ/m². The value of the free interaction energy of the glass surface is positive ΔG_{iw} = 22.48 mJ/m².

The difference between the surface of the glass and that of the ZnO film is observed in the acid-base interaction γ^{AB} (electron acceptor and donor) also called polar. The value of the acid-base component γ^{AB} = 3.0 mJ/m² calculated for the surface of the 200 watt ZnO film is low in front of the value of this component for the glass surface (γ^{AB} = 18.5 mJ/m²). On the other hand, the basic component of the glass surface is very important before that of the ZnO film surface (γ^{glass} = 45.3 mJ/m², γ^{ZnO 150 w} = 12.2 mJ/m²). This means that this difference is regarding the electron donor/acceptor interaction between the glass surface and the ZnO film. The dispersive component of the surface energy for the different substrates is very important in front of the acid-base component. The values range from γ^{D} = 39.04 mJ/m² and γ^{D} = 43 mJ/m² for ZnO film surfaces, and γ^{D} = 32.7 mJ/m² for glass surface.
Figure 7. Characteristic of the surface energy LW and AB of the different samples obtained at different powers (150w, 200w and 250w).
The characteristics presented in Figures 7 and 8 summarize the obtained results. The difference between the surface of the glass and the surface of the ZnO film is well observed. The acid-base components of glass have 36.2% of the surface energy. On the other hand, the acid-base component of ZnO films has only 4.89%, 7.08% and 1.71% respectively for ZnO 150w, ZnO 200w and ZnO 250w of the surface energy. In addition, the glass surface has a very important base component value in front of the values of this component of the ZnO thin film surface. It can also be seen that the acid component of the various samples is practically negligible compared to the base component. It can be said that ZnO films have reduced the electron accepting character of the glass surface. The results also showed that the glass surface is governed by short-range forces because it has a very high acid-base (polar) surface energy value. However, ZnO thin films are governed by long-range forces because they have quite high LW surface energy values confirming the results obtained in the works [27,28].
Figure 9 shows the result obtained from the hydrophobicity of the different samples. The degree of hydrophobicity expressed in terms of free energy of interaction $\Delta G_{iw}$ increases with the increase in the deposition power of ZnO thin films. The results also reveal that these films are hydrophobic. The surface of the glass has a positive value $\Delta G_{iw}$ indicating that this surface is hydrophilic. The coating of the ZnO also is modified the hydrophobic/hydrophilic character of the glass surface.

4. Conclusion

The thin films of zinc oxide produced by RF sputtering under different powers have been characterized by different techniques. The diversity of the results has showed the influence of the processing conditions on the optical, topographical, structural and physico-chemical properties.

From a physico-chemical point of view, the comparison between the surface of the glass and the surface of the ZnO films has confirmed that the surface of the glass is hydrophilic and the surface of the ZnO films is hydrophobic. Moreover, the surface of the glass is governed by the supported edge forces (AB) and the surface of the ZnO films is governed by the long-range forces (LW).
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