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Preparation and Characterization of ZnO Nanocrystalline Layers

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Abstract

This work consists on the study of the optical properties of zinc oxide layers. These Layers are elaborated under various conditions by cathode sputtering on glass substrates and by thermal oxidations of Zn layers deposited on different types of substrates by vacuum evaporation. The oxidation treatments of Zn are made in oxygen atmosphere at temperatures between 400˚C and 450˚C for different times. The analyses by diffraction of X-rays, Optical Microscopy and Scanning electron microscopy enabled us to understand that the zinc oxide films deposited by cathode sputtering on a glass substrate and having a thickness of at least 240 nm and those prepared by thermal oxidation at 450˚C during 2 hours of layers of Zn on an alumina substrate are homogeneous and consist of grains of size 30 nm. The optical transmission measurements show that the gap is around 3.02 eV for layers obtained by oxidation and 3.3 eV for those deposited by sputtering. These samples have a good optical transparency in the visible.

Keywords

Oxide, Gas, Adsorption, Conductance, Properties

1. Introduction

Zinc oxide has interesting properties that qualifies it among semiconductor materials the most used in the field of gas detectors [1].

In order to obtain materials with good stability in the environment, metal oxides appear to be most appropriate for many applications. In the field of gas detection, several studies [2] showed that zinc oxide is very promising.

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It is a semiconductor with a direct large gap and generally, crystallizes in würtzite structure [3]. However, the size, the orientation of grains and the surface quality depend on the conditions in which the material was prepared and the different treatments which could be made. Indeed, surface states of semiconductor materials generate superficial electronic properties which are often significantly affected by their interactions with foreign elements [4].

2. Experimental Conditions

In the aim to obtain various qualities of zinc oxide layers, in order to study their stability and to propose them for practical uses, particularly in the gas detection, we used two techniques: the sputtering of zinc oxide and thermal oxidation of zinc layers prepared by vacuum evaporation.

In the technique of sputtering, the layers were deposited on glass substrates by using a target of zinc and injecting oxygen in argon atmosphere. Oxygen will interact with the zinc to form molecules of ZnO.

Other layers of zinc oxide were obtained by thermal oxidation of Zn layers, in oxygen atmosphere, at temperatures 420˚C - 450˚C during different periods of 2 to 4 hours. The layers of Zn were prepared by vacuum evaporation on glass and alumina substrates.

3. Characterization of ZnO Layers

Analysis by X-rays diffraction, optical microscopy and scanning electron microscopy, were carried out in order to identify the phases formed, the crystalline structure, size and superficial aspect of ZnO layers elaborated. **Figure 1** and **Figure 2** present spectra of X-rays diffraction obtained on layers prepared under different conditions. They show a preferred orientation of grains for all layers, according to the plans (002).

The samples prepared by thermal oxidation of zinc layers gave spectra (**Figure 1**) characteristics of ZnO but with the presence of lines specific of O_2 and Zn. The presence of Zn proves that, under the conditions used, the oxidation reaction is partial.

In the case of layers obtained by sputtering, the spectra of X-rays diffraction (**Figure 2**) are characteristic of the compound.

The micrographs (**Figure 3** and **Figure 4**) of optical microscopy show that layers prepared under different conditions have different aspects.

Layers obtained by thermal oxidation on alumina substrates (**Figure 3(a)**) are more homogeneous than those prepared using the same technique on glass substrates (**Figure 3(b)**). According to **Figure 4**, layers obtained by sputtering are relatively homogeneous and this aspect seems to improve with the thickness.

We observe that there is a granulometry for the various aspects of layers obtained by thermal oxidation on the glass substrate (**Figure 4**).

The observations by scanning electron microscopy show that the layers obtained by thermal oxidation on alumina substrates (**Figure 5**) are more homogeneous than those obtained on glass substrates (**Figure 6**). We

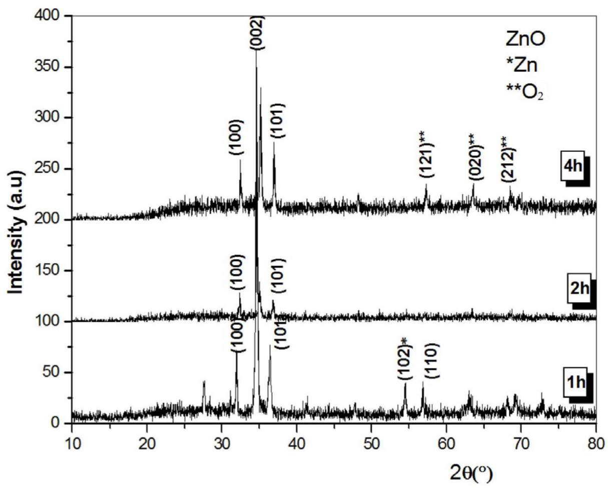


Figure 1. X-rays diffraction of zinc oxide layers obtained by thermal oxidations at 450˚C during (1h-2h-4h) on glass substrates.

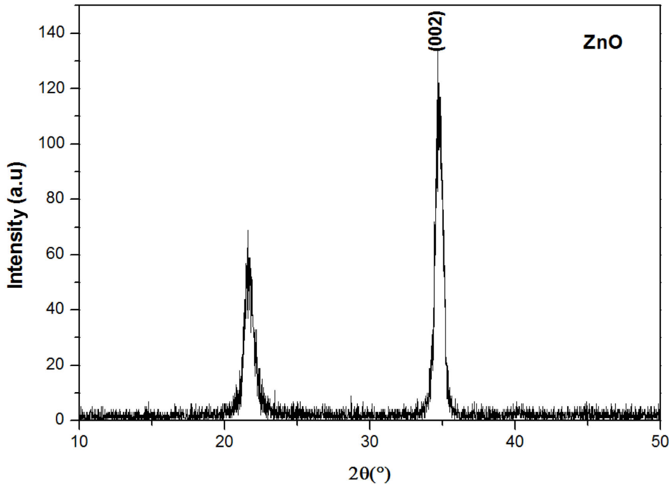
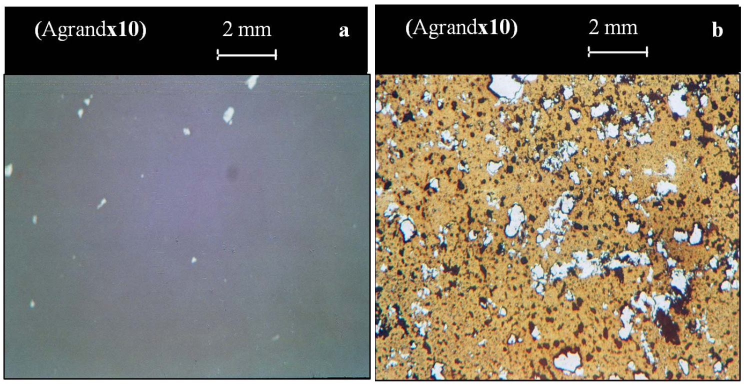
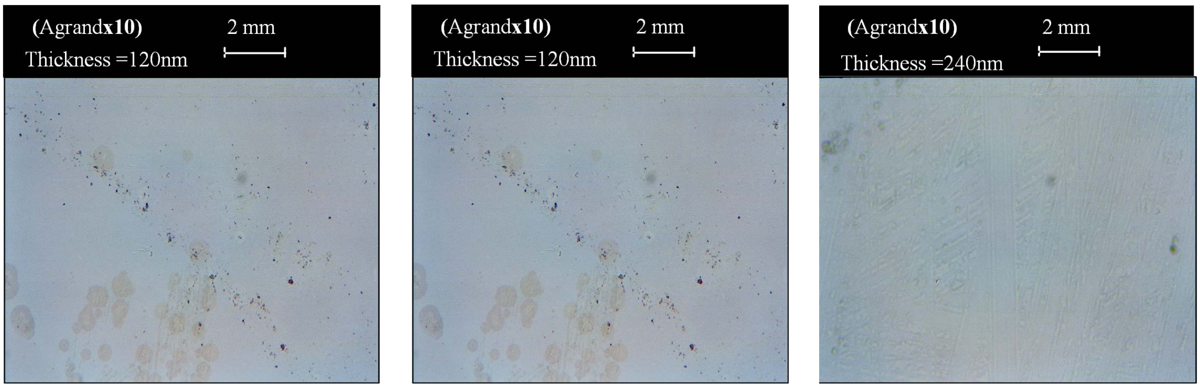


Figure 2. X-ray diffraction of zinc oxide layer, obtained by sputtering on glass substrate.



(a) (b)

Figure 3. Optical microscopy of zinc oxide layers obtained by thermal oxidation during 2 hours at: (a) 420˚C on an alumina substrate; and (b) 450˚C on glass substrate.



(a) (b) (c)

Figure 4. Optical Microscopy of zinc oxide layers with different thicknesses, obtained by sputtering on a glass substrates.

note the existence of grains with different forms and aspects for the layers produced by thermal oxidation on glass substrates.

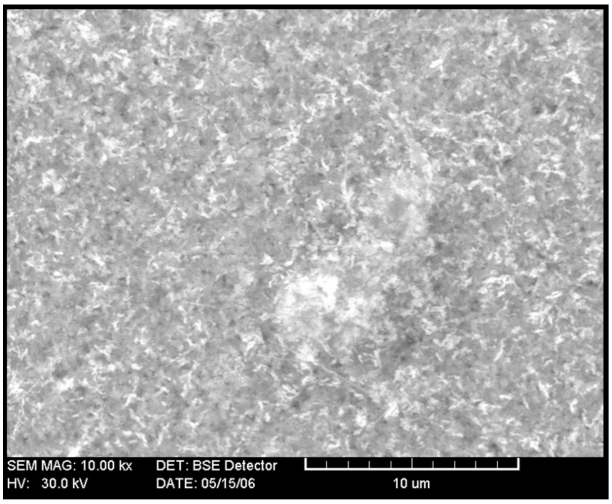


Figure 5. Scanning electron microscopy of zinc oxide layer, obtained on an alumina substrate by oxidation at 450˚C for 2 hours.

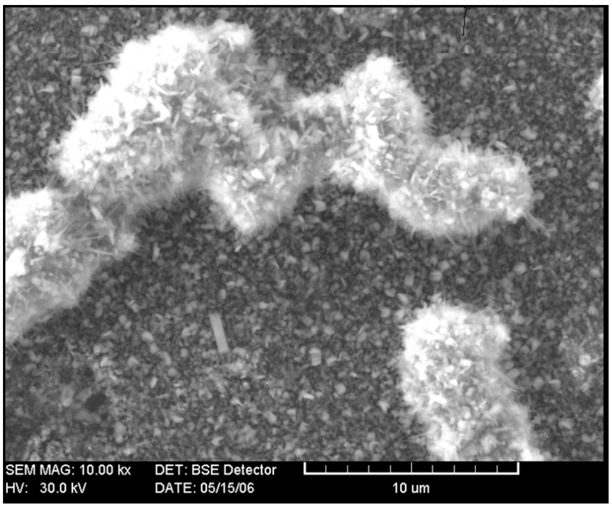


Figure 6. Scanning electron microscopy of zinc oxide layer, obtained on a glass substrate by oxidation at 450˚C for 2 hours.

The analysis of X-rays diffraction results and scanning electron microscopy allowed us to determine the grains sizes which are about 60nm for the layers prepared by thermal oxidation on alumina substrates and 30 nm for the layers obtained by sputtering on glass substrates. This nano-size grain gives the material a large specific surface favouring a strong adsorption of gas.

Measures of optical transmission (**Figures 7-10**) made on the zinc oxide layers allowed us to identify that the values of the gap are varying from 3.2 eV to 3.3 eV for layers prepared by sputtering and 3.02 eV for layers obtained by thermal oxidation. The layers have a good optical transparency in the visible.

4. Conclusions

The zinc oxide layers prepared by various techniques have a preferential orientation of the grains according to the plans (002).

The layers obtained by thermal oxidation on the alumina substrates have a more homogeneous aspect compared with those prepared by the same technique on glass substrates.

The techniques used in elaboration of the samples allowed us to obtain layers with grains sizes about 60 nm for the materials prepared by thermal oxidation on alumina substrate and 30 nm for those prepared by sputtering

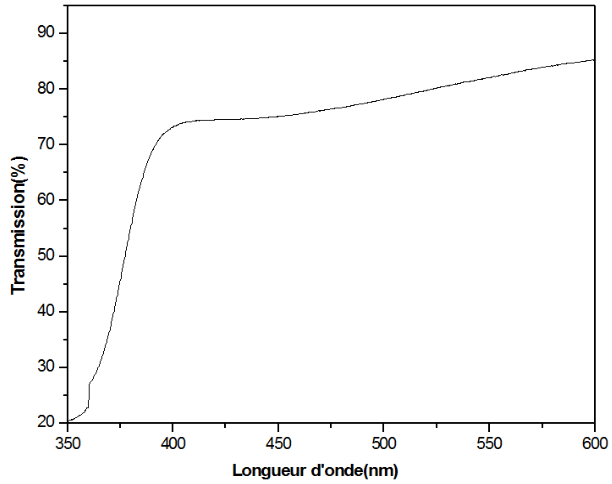


Figure 7. Optical transmission of a zinc oxide layer obtained by sputtering on a glass substrate, (thickness = 240 nm).

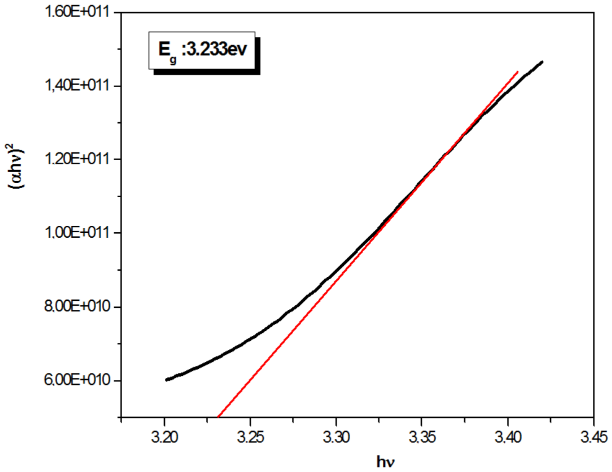


Figure 8. Curve $(\alpha \upsilon h)^2 = f(h\upsilon)$ of a zinc oxide layer obtained by sputtering on a glass substrate, (thickness = 240 nm).

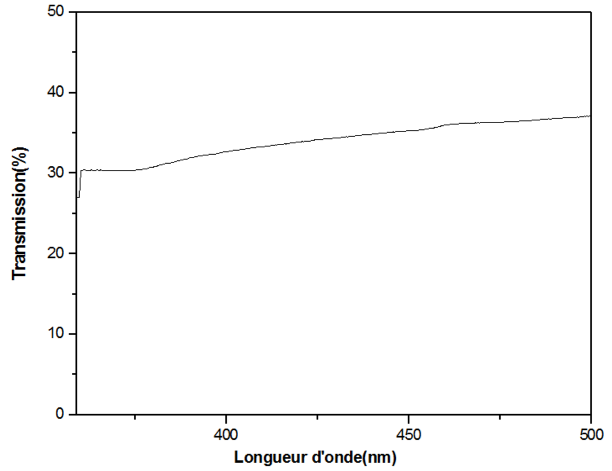


Figure 9. Optical transmission of a zinc oxide layer obtained on a glass substrate by oxidation at 420˚C for 2 hours.

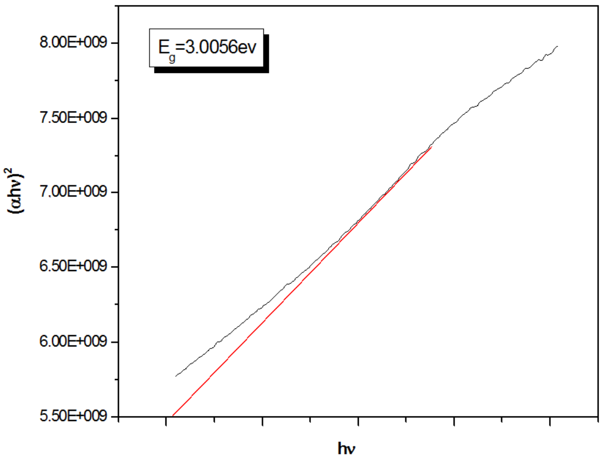


Figure 10. Curve $(\alpha \upsilon h)^2 = f(h\upsilon)$ of zinc oxide layer obtained by thermal oxidation on a glass substrate at 420˚C for 2 hours.

on glass substrates. This nano-size grain gives the material a large specific surface favouring a strong adsorption of gas.

The values of the gap are varying from 3.2 eV to 3.3 eV for layers prepared by sputtering and 3.02 eV for layers obtained by thermal oxidation. The layers have a good optical transparency in the visible.

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