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Influence of precursor type, deposition time and doping concentration on the morphological, electrical and optical properties of ZnO and ZnO:Al thin films grown by ultrasonic spray pyrolysis

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ABSTRACT

The influence of precursor type, deposition time and doping concentration on the morphological, optical and electrical characteristics of ZnO and ZnO:Al thin films and nanostructures deposited by ultrasonic spray pyrolysis at 400 °C on glass substrates was investigated. A deposition period of 30 min resulted in compact, uniform samples with thickness of 30 and 45 nm, and growth rates in the order of 1 and 1.5 nm/min for the Zn acetate and Zn nitrate precursors, respectively. The as-grown films showed good adhesion and excellent optical transmission. Moreover, films grown using Zn acetate precursor exhibited considerably low resistivity, being 14 Ω·cm for a deposition time of 30 min. Surface roughness was observed to increase with deposition time, while Al doping up to 5% reduced the resistivity down to 4 Ω·cm without affecting the optical transmission of the films. Finally, films grown using Zn nitrate were observed to exhibit better photoconductivity than those grown with Zn acetate.

1. Introduction

The next generation of photonic devices requires transparent electrodes to be lightweight, flexible, cost effective and compatible with large-scale manufacturing. Tin doped Indium Oxide (ITO), the most widely transparent electrode used today in display, photovoltaic and lighting industry, presents severe drawbacks including high cost, mechanical limitations and possibly health risks. Despite that, ITO is mostly used, primarily because there are very few other materials exhibiting optimal combination of optical transparency and conductivity. As a result, other transparent conductive materials are required, exhibiting the same or even superior performance, lower cost and better physical properties than ITO can offer. Regarding possible candidates for the replacement of ITO, intense research efforts are going on regarding transparent conductive oxides, including pure and doped ZnO, In_2O_3 and SnO_2 [1,2]. The targets of these research efforts include large-scale fabrication at relatively low cost, compatibility with flexible substrates and improved performance.

Pure and/or doped zinc oxide films have attracted a lot of attention because of their physical, chemical, electrical and optical properties, such as availability in nature, nontoxicity, high optical transparency in the visible region, wide band and low resistivity, to name but a few properties that have enabled applications as transparent electrodes for solar cells, liquid crystal displays and organic light emitting diodes [3,4]. Many techniques have been used to deposit ZnO and ZnO:Al films on different substrates, including Chemical Vapor Deposition [5], Pulsed Laser Deposition [6], Molecular Beam Epitaxy [7], sputtering [8], spray pyrolysis [9], sol–gel [10] and electrodeposition [11]. Compared to other deposition techniques, spray pyrolysis offers several advantages like non-vacuum use of inexpensive equipment, ease of large scale adoption and possibility of automation for industrial use. Moreover, using spray pyrolysis, one can control the basic structural and morphological characteristics of the as-grown material through the growth conditions, leading to films exhibiting the required functionality for particular applications. Substrate temperature, concentration of precursor solution, type and pressure of the carrier gas, geometric characteristics of the spraying system and spraying rate are the parameters that can significantly affect the properties of the as-grown films. Regarding the deposition of ZnO and ZnO:Al films with properties suitable for transparent electrodes, several reports exist in the literature [12–18], reporting resistivity values varying in the range of 10^{-1} up to 10^3 Ω·cm. Moreover, it has become clear that resistivity is strongly correlated with the structural and morphological characteristics of the films.

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In this work we investigate the influence of precursor solution, deposition time and Al-doping concentration on the morphological, electrical, and optical characteristics of pure or aluminum doped ZnO films deposited using ultrasonic spray pyrolysis on Corning glass substrates. However, emphasis is not given on the minimization of the resistivity but on the determination of how the growth conditions are affecting optical transmission, electrical conductivity and surface roughness.

2. Experimental details

ZnO structures were grown by the Ultrasonic Spray Pyrolysis technique on Corning glass square inch substrates (Corning Eagle 2000 Borosilicate Glass, Specialty Glass Products) for various deposition periods using 0.5 M precursor solutions of: (a) zinc acetate ($Zn(CH_3COO)_2 \cdot 2H_2O$) and (b) zinc nitrate ($Zn(NO_3) \cdot 6H_2O$) in ultrapure water (18.2 MΩ·cm), after stirring for 15 min at 25 °C. Before deposition, the substrates were cleaned for 10 min using a piranha solution ($H_2SO_4/H_2O_2 = 3/1$, with H_2SO_4 and H_2O_2 provided from Sigma-Aldrich with concentrations 95.0–98.0% and 30% respectively), rinsed with ultrapure water and dried under N_2 gas flow. The deposition was performed at 400 °C with an Ultrasonic particle generator (Model 241 PG, Sonaer Inc.), using an about 59 kPa N_2 flow and a frequency of 2.4 MHz allowing 100% particle generation. The spraying conditions were optimized before deposition, and the respective values were chosen to be: 8 mm nozzle diameter, 3.5 cm nozzle length and 5 cm distance between nozzle and substrate. The average diameter of the misted droplet was approximately calculated from an expression given by Lang [19]:

$$D_d = 0.34\left(\frac{8\pi\gamma}{\rho f^2}\right)^{1/3}$$

where D_d is the droplet diameter, γ is the solution surface tension, ρ is the solution density and f is the applied ultrasonic frequency. The diameter of the misted droplets in our experiment was calculated using the above expression and was found to be around 2.3 μm.

The Al doped ZnO samples were deposited using the same experimental conditions mentioned above, employing a 0.5 M solution of zinc acetate and aluminum nitrate ($Al(NO_3)_3 \cdot 9H_2O$) in ultrapure water, as precursor. The nominal % molar concentration of Al in the precursor solution was 1, 3 and 5%, respectively. After growth, all samples were thoroughly washed with ultrapure water, and dried in air.

The crystal structure of the ZnO and ZnO:Al samples was determined by X-ray Diffraction (XRD), using a Rigaku (RINT 2000) diffractometer with Cu *Ka* X-rays, while their surface morphology was studied by means of a Field Emission Scanning Electron Microscope (FE-SEM, JEOL JSM-7000F) and an Atomic Force Microscope (AFM) in tapping mode (Digital Instruments — Nanoscope IIIa). The root mean square (RMS) surface roughness of the ZnO seed samples was determined using the Scanning Probe Image Processor (v. 3.3.5.0) image processing software for nano- and micro-scale microscopy from Image Metrology. The optical transmission of the samples was recorded using a Shimatzu UV-2401 spectrometer over the wavelength range of 250–1000 nm, while their thickness was measured using a stylus profilometer (alpha-step 100, Tencor). Finally, the electrical resistivity of the pure and Al-doped ZnO samples was determined with a four-probe conductivity measurement setup, using silver paste in order to form the required electrical contacts, while the photoconductivity measurements were performed under the illumination of 10 mW/cm^2 UV-A radiation, coming from an HPK 125 W Philips Hg lamp with main emission wavelength at 365 nm.

3. Results and discussion

Initially, the spraying conditions (such as nozzle diameter, nozzle length, distance between nozzle and substrate and substrate temperature) were optimized in such way that compact samples covering uniformly the substrate could be deposited. It was observed that deposition periods longer than 30 min are required in order to produce samples with good adhesion (passing the Scotch tape test) and long term stability (having similar properties after approximately six months).

Ultrasonic spray pyrolysis was then employed for the growth of ZnO thin films on Corning glass substrates for a deposition time of 30 min using aqueous precursor solutions of Zn acetate and Zn nitrate. The optical transmission of the as-grown ZnO samples was observed to be excellent for both precursors used, as indicated by the UV–visible transmission curves of Fig. 1 for the spectral region 250–1000 nm. For both precursor types, the ZnO samples were found to be highly transparent in the visible wavelength region with an average transmittance of about 80–90%, with a fall-off for wavelengths shorter than 380 nm, which are characteristic for good quality ZnO coatings. Moreover, an additional absorption band centered near 345 nm was clearly recorded, band that can be attributed to excitonic resonances [20]. The optical energy gap (E_{gap}) of the samples, determined using "Tauc" plots of α^2 as a function of $h\nu$, was found to be 3.27 and 3.26 eV for the ZnO samples grown from Zn acetate and Zn nitrate precursor solutions, respectively, similar to the E_{gap} values reported in the literature for high quality ZnO films grown using spray pyrolysis [12,21].

Besides the excellent optical transmission, ZnO samples were observed to exhibit good electrical response, at least in the case of the Zn acetate precursor. In particular, a resistivity of 14 and 5300 Ω·cm was measured for ZnO samples grown onto glass using Zn acetate and Zn nitrate precursor solutions, respectively. In general, the resistivity of pure or doped ZnO films grown by spray pyrolysis is strongly correlated with the structural and morphological characteristics of the films, with its reported value varying in the range of 10^{-1} up to 10^3 Ω·cm [12–18]. Therefore, it will be interesting to investigate how the morphology of our ZnO samples affects their resistivity and how this can be minimized.

Fig. 2 presents the XRD patterns of the ZnO samples grown for 30 min. As can be seen, the films exhibit a wurtzite polycrystalline structure, in agreement with the results of other research groups [22]. One can notice from Fig. 2 that the contribution from the (002) plane is the strongest, especially in the case of zinc nitrate precursor solutions, indicating a tendency of preferential growth of crystallites perpendicular to the surface (*c*-axis oriented). Using the (002) peak broadening (full width at half maximum value) from the XRD pattern and the Scherer's formula, the crystallite size (d) was determined to be 25 and 19 nm for ZnO samples grown from Zn acetate and Zn nitrate precursor solutions, respectively. Moreover, the thickness of the as-grown ZnO samples was measured to be 30 and 45 nm for the Zn acetate and the Zn nitrate precursor, respectively, with growth rates 1 and 1.5 nm/min, respectively. To understand the growth mechanism of the ZnO films

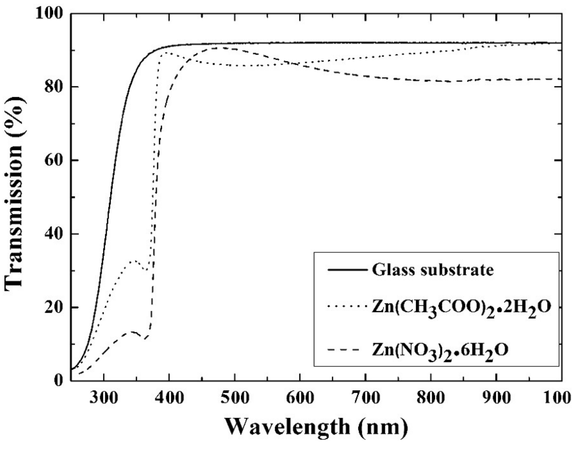

Fig. 1. Optical transmission of ZnO films grown by ultrasonic spray pyrolysis for 30 min using as precursor 0.5 M aqueous solutions of: (a) zinc acetate and (b) zinc nitrate.

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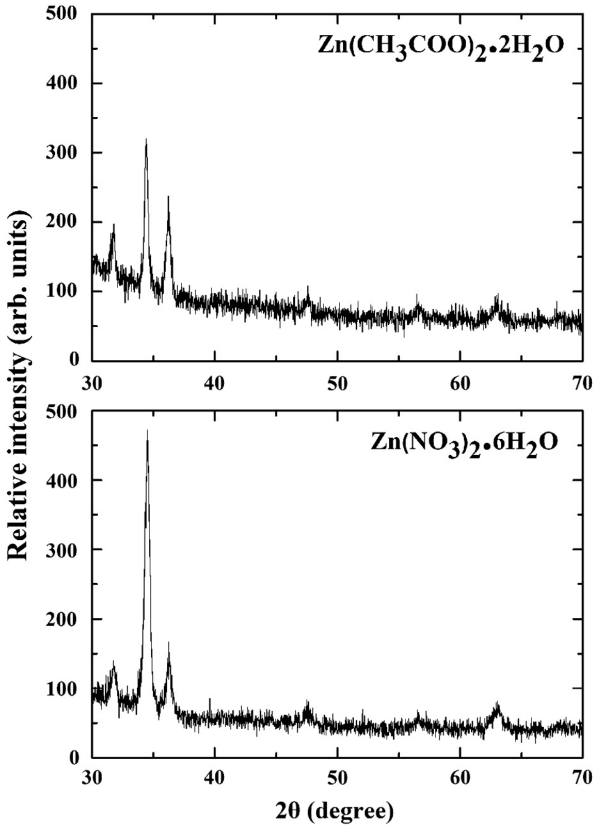

Fig. 2. XRD patterns of ZnO films grown by ultrasonic spray pyrolysis for 30 min using as precursor 0.5 M aqueous solutions of: (a) zinc acetate and (b) zinc nitrate.

obtained with the two Zn precursors, one should take into account the thickness and the XRD data of the samples. The larger growth rate for the case of Zn nitrate is also reflected in the XRD data presented in Fig. 2, since the corresponding XRD pattern exhibits stronger (002) diffraction peak, indicating that the preferential growth orientation of the ZnO samples perpendicular to the glass substrates occurs with Zn nitrate precursor [22]. The observations for the intensities of the (002) peak of the as-grown ZnO samples for the two precursors used, clearly indicate a difference in the crystal growth rate, depending on the nature of the precursor [23]. The surface morphology is shown in Fig. 3, which presents FE-SEM images of the samples grown for 30 min using Zn acetate and Zn nitrate as precursors. As can be seen, the samples are quite smooth granular films, not really homogeneous, consisting of grains with diameters of about 20 and 50 nm for Zn acetate and Zn nitrate precursors, respectively. The corresponding RMS roughness was estimated to be around 2.4 and 4.9 nm. These values indicate quite low roughness, a property needed in applications like organic solar cells where large roughness and high 3D structures can lead to local shunts since the photoactive layers are normally quite thin. Moreover, ZnO samples grown using Zn acetate precursor appeared to be more compact, while, for the case of Zn nitrate precursor, a distance of almost 50 nm, similar to the diameter of the grains, exists between grains.

Based on the above experimental observations, ZnO thin samples grown by spray pyrolysis for 30 min using Zn acetate precursor solution seem to be promising materials for transparent electrodes, since they exhibit excellent optical transparency and quite low resistivity. In contrast, the samples grown using Zn nitrate precursor present quite high resistivity, due to their different surface morphologies (larger RMS roughness and longer distance between grains), which limits their applicability as transparent electrodes but possibly opening perspectives for optical applications requiring optical confinement and high ordering.

In a trial to further improve the conductivity of the ZnO samples grown using Zn acetate as precursor, the deposition time was increased up to 120 min. When we increased the deposition time, the as-grown samples appeared to become highly nanostructured due to the coexistence of nanorods (with a diameter in the range of 20–30 nm) and hexagonal nanopetals (with a diameter of around 400–450 nm and a thickness of 20–30 nm), both grown perpendicular to the surface (see Fig. 5). Moreover, the samples exhibited higher roughness, approaching 47.4 nm for 60 min deposition. For even longer deposition periods (120 min), the nanopetals were observed to agglomerate the surface consisting mainly of flat plates with nanorods standing between them.

The optical transmission of the samples was slightly reduced, remaining however quite high, being between 60 and 70% in the visible spectral region even for long deposition periods (60, 120 min). An interesting observation concerns a small red shift of the optical energy gap with increasing deposition period, approaching 3.24 eV, indicating a similar behavior to that reported in the literature for ZnO thin films grown by spray pyrolysis [24]. The crystallinity of the samples was significantly improved with increasing deposition time, as it can be seen in Fig. 4, with the contribution from (002) plane of the ZnO wurtzite structure dominating the XRD pattern. Moreover, the crystallite size was found to increase, approaching 33 nm for 120 min deposition time. Finally, the resistivity of the samples was reduced down to 6 Ω·cm for 120 min deposition.

Although the increase of deposition time resulted in an improvement of the crystallinity of the samples, the respective improvement in the conductivity is not significant, which can be attributed to the drastic change of film's morphology. As determined from AFM characterization, samples that were grown for 120 min exhibit a 15 times larger RMS roughness than those deposited for 30 min. This large RMS roughness seems to affect the conductivity negatively, since this cannot be significantly improved. At the same time, the samples are not suitable for organic solar cells due to their large roughness. On the other

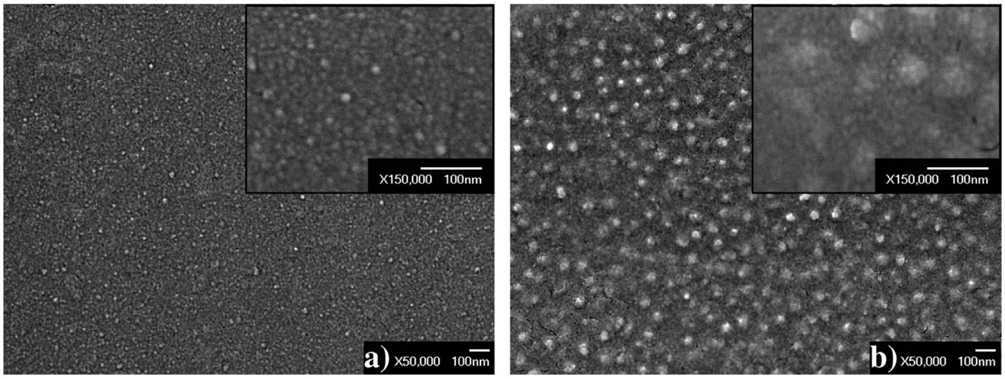

Fig. 3. FE-SEM images of ZnO films grown by ultrasonic spray pyrolysis for 30 min using as precursor 0.5 M aqueous solutions of: (a) zinc acetate and (b) zinc nitrate.

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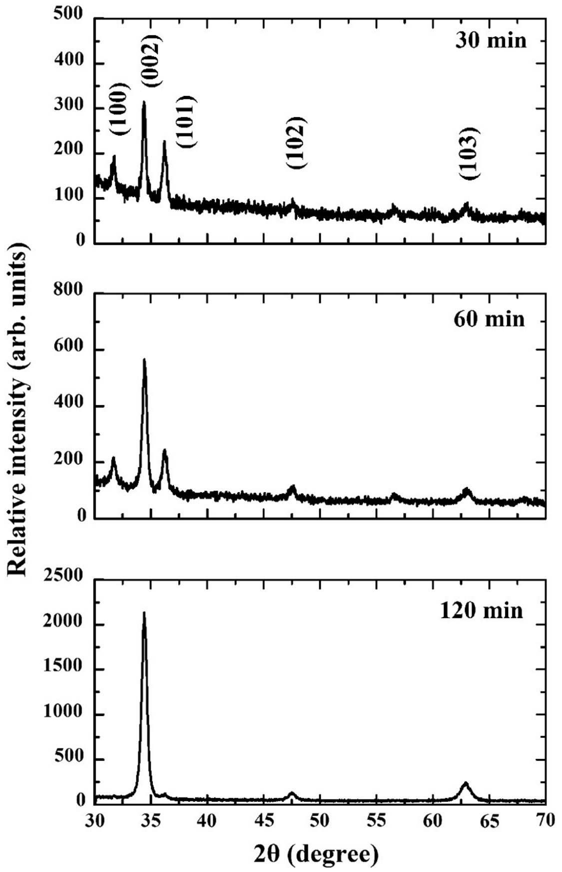

Fig. 4. XRD patterns of ZnO films grown by ultrasonic spray pyrolysis for various deposition times using as precursor 0.5 M aqueous solution of zinc acetate.

hand, this high roughness can be advantageous in amorphous and microcrystalline silicon thin film solar cells, since it can improve their efficiency due to light scattering. Finally, the increase in the deposition time caused a drastic increase in the coverage of the samples, while the height of the nanostructures described above approaches ~1 μm for a deposition period of 120 min (and a growth rate of ~9 nm/min), which may be attributed to the increase of the ZnO growth rate after the initial nucleation stage.

In another trial for reducing the resistivity of the spray-deposited ZnO samples, Al doping was performed using 0.5 M solutions of zinc acetate and aluminum nitrate in ultrapure water as a precursor at nominal concentrations of 1, 3 and 5% Al in ZnO, respectively, keeping the deposition time at 30 min. Under these conditions, homogeneous thin films were derived with excellent optical transmission, being higher than 80% in all cases, although the thickness of the films increased with Al-doping, reaching ~65 nm for 5% Al concentration. Moreover, as shown in Fig. 6, Al-doping improved the crystallinity of the samples, which were dominated by the (002) plane of ZnO wurtzite structure, implying preferential orientation of crystallites perpendicular to the surface. A comparison of the XRD patterns of Al doped ZnO thin films (Fig. 6) with those of pure ZnO thin films (Fig. 4, top), both grown for 30 min using Zn acetate precursor, reveals a considerable increase in the wurtzite (002) peak intensities for the Al-doped cases, which can be attributed to an enhancement of the crystallinity because of the substitution reaction between Zn and Al. Indeed, the difference in the atomic radii between Zn and the dopant Al atoms can probably lead to interstitial reactions, which may result in more intense wurtzite (002) peaks in the XRD patterns as well as higher growth rates. The increase of growth rate for the Al-doped samples was clearly indicated by their thickness, which was found to be double than that of the undoped ZnO samples. The intensity of the (002) XRD peak was observed to be almost unaffected by the Al concentration, while the crystallite size was found to decrease, approaching 19 nm for 5% Al concentration. All ZnO:Al films were observed to consist of grains with a diameter of around 20 nm, a quite similar value to that observed for the undoped ZnO samples. Moreover, increasing Al-doping concentration resulted in more compact films with better shaped grains (see Fig. 7).

Fig. 8 presents the variation of the samples' resistivity as a function of the Al nitrate concentration in the precursor solution. As one can see, minimum resistivity is observed for an Al concentration of 3%, while, the resistivity is considerably larger for higher concentrations. The minimization of the resistivity at a particular Al concentration (3%), has also been observed by other research groups [25,26] and can be attributed to the fact that the excess amount of Al cannot be accommodated into the ZnO lattice due to its limited solid solubility and therefore forms neutral Al oxide and segregates at the grain boundaries.

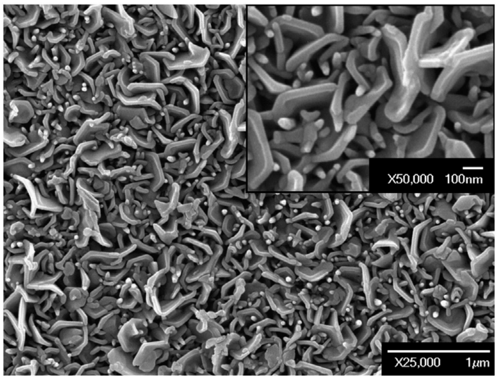

Fig. 5. SEM image of a ZnO film grown by ultrasonic spray pyrolysis for 60 min using as precursor 0.5 M aqueous solution of zinc acetate.

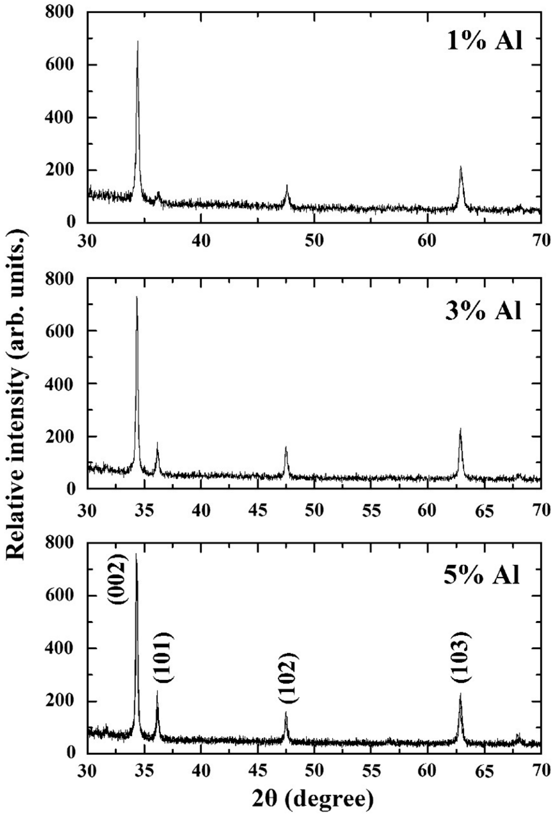

Fig. 6. XRD patterns of ZnO:Al films grown by ultrasonic spray pyrolysis for 30 min, for two different aluminum nitrate concentrations in a 0.5 M aqueous solution of zinc acetate.

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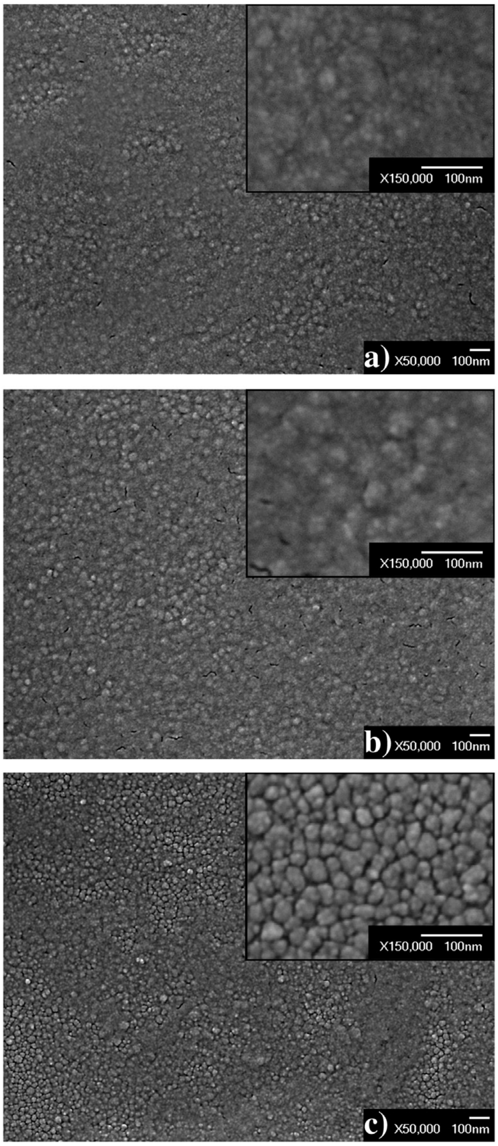

Fig. 7. FE-SEM images of ZnO:Al films grown by ultrasonic spray pyrolysis for 30 min, for 1 and 3% aluminum nitrate concentrations in a 0.5 M aqueous solution of zinc acetate.

The obtained resistivity values, even at the optimum Al concentration, are quite high compared with other values reported in the literature. However, the scope of our work was a parametric study of the influence of precursor type, deposition time and Al-doping concentration on the characteristics of pure or aluminum doped ZnO films deposited using ultrasonic spray pyrolysis on Corning glass substrates.

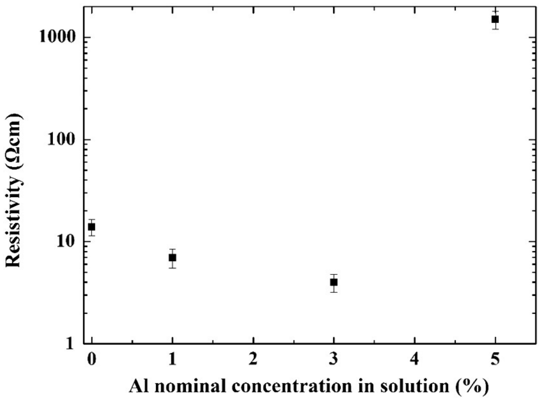

Fig. 8. Variation of the resistivity of ZnO:Al films grown by ultrasonic spray pyrolysis for 30 min as a function of aluminum nitrate concentration in a 0.5 M aqueous solution of zinc acetate.

As far as it concerns the quite large resistivity of the 5% Al concentration sample, this can be attributed to two independent factors:

(a) The fact that this is a concentration larger than the optimum one: The amount of electrically active Al atoms is reduced when the Al doping is high and excess dopant leads in the building up of carrier traps in the lattice, thus reducing their mobility.
(b) The film structuring is different: The grains of the films are separated by ~10 nm distance, almost 10 times larger distance than that measured for lower Al concentrations.

Finally, the photoconductivity of the as-grown ZnO samples was examined using UV-A radiation. Optimum photoconductive response was recorded for the ZnO samples grown using Zn nitrate precursor, resistivity reduced by a factor of 300 under UV light irradiation. In contrast, ZnO films grown using Zn acetate precursor showed only 30 times reduction in the resistivity, while the corresponding behavior for ZnO:Al films was even weaker. In general, the photoconductivity of metal oxide thin films is a complex process depending not only on photoinduced generation and transport of charges, but also on photodesorption and adsorption of oxygen molecules on the surface and at the grain boundary areas [27]. As a result, the photoconductivity can be influenced by the crystallinity, the defect and impurity concentration, the surface morphology and the oxidation processes taking place in the near-surface region of a metal oxide sample. Furthermore, the photoresponse case under study can be strongly influenced by the differences in the surface morphology and the RMS roughness of the ZnO samples. Therefore, it can be concluded that ZnO samples grown using Zn nitrate precursor give significant photoconductivity values due to their particular morphology.

4. Conclusions

ZnO and ZnO:Al thin films with different textures were deposited on Corning glass substrates, by ultrasonic spray pyrolysis using two Zn precursors, various deposition times and different Al-doping concentrations. The structural, optical and electrical properties of the as-grown samples were studied along with their morphology and surface roughness properties. It is revealed that a deposition period of 30 min is enough in order to produce uniform films exhibiting excellent optical transmission. Moreover, we have presented evidence indicating that the Zn acetate precursor leads to considerably better conductivity values. Doping with Al was found to improve the conductivity of the as-grown samples, without affecting significantly the morphology and the optical transmission of the films. Finally, the photoconductivity of the samples was examined under UV-A irradiation, revealing that pure ZnO films grown using Zn nitrate precursor exhibit considerably better photoconductivity than the Al-doped ones, while the corresponding resistivity values were reduced by a factor of 300.

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