

CAS 2014 PROCEEDINGS

2014 INTERNATIONAL SEMICONDUCTOR CONFERENCE

October 13-15, Sinaia, ROMANIA

**NATIONAL INSTITUTE FOR RESEARCH AND DEVELOPMENT
IN MICROTECHNOLOGIES (IMT Bucharest)**



**under the aegis of
Romanian Academy
Electrochemical Society Inc.**



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2014 INTERNATIONAL SEMICONDUCTOR CONFERENCE

**37th Edition
October 13-15, Sinaia, Romania**

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FOREWORD

Welcome to the 37th edition of the *International Semiconductor Conference (CAS 2014)* www.imt.ro/cas! This event is taking place every year since 1978. It was organized by ICCE until 1996 as an *Annual Semiconductor Conference* (in Romanian – *Conferinta Anuala de Semiconductoare* – CAS). Since 1997, the organizer of CAS has been the **National Institute for Research and Development in Microtechnologies** (IMT Bucharest, www.imt.ro), and the conference profile is **micro and nanotehnologies** (including semiconductor electronics).

Since 1991, CAS was opened to foreign participants, becoming an international conference. Starting with the 1995 edition, this conference is an **IEEE (Institute of Electrical and Electronic Engineers) event**, being sponsored by the IEEE Electron Devices Society. As mentioned above, **the Conference profile** has been gradually extended from semiconductor device physics and technology (including semiconductor materials and microelectronics) to **micro- and nanotechnologies** in general, including micro-nanoelectronics and micro-nanosystem technologies. Nanostructures and nanostructured materials are also considered.

CAS 2014 is sponsored by *IEEE Electron Devices Society*, under whose authority the CAS 2014 Proceedings is published, by IMT Bucharest, the IEEE - Romania Section and the Electron Devices Chapter. This year, the companies Carl Zeiss Instruments, S.C. SITEX 45 SRL, InterNET SRL and S.C. New Style Trans Prest SRL are co-sponsors of the Conference. The conference is also held under the aegis of the *Romanian Academy* as well as under the aegis of the *Electrochemical Society, Inc.*, including the European Local Section of the Electrochemical Society, Inc.

CAS 2014 presents a total number of 70 papers (59 regular and 11 invited). The authors are from 13 countries on 3 continents (10 countries from Europe). The quality of papers accepted for publication in the Proceedings was guaranteed by the evaluation made by referees (including members of the International Advisory Committee and of the Technical Program Committee). We would like to thank all the reviewers for their devoted work.

This year we have **11 invited speakers** from Bulgaria, France, Italy, Germany, Serbia, Spain, and Romania. We are grateful to the invited speakers for their effort to make our Conference more attractive, as well to the distinguished scientists chairing the various sessions of CAS 2014.

We would like to wish to all participants a successful participation in this Conference and a pleasant stay in Sinaia.

Dan Dascalu
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THE EFFECT OF ANNEALING IN NITROGEN ATMOSPHERE ON THE STRUCTURE, PHOTOLUMINESCENCE AND ELECTRICAL PROPERTIES OF Li AND Cu DOPED SOL-GEL ZnO FILMS

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Abstract—The effect of annealing in air and in nitrogen atmosphere on the structure, luminescence emission and electrical properties of ZnO, Li:ZnO and Cu:ZnO doped thin films prepared by sol-gel method was investigated by scanning electron microscopy, X-ray diffraction, photoluminescence and resistivity measurements. The films annealed in nitrogen demonstrate smoother surfaces, improved crystallinity and conductivity. The residual stress in doped films changes from tensile type, when annealed in air, to compressive type in the case of annealing in nitrogen atmosphere. The effect is associated with the density of lattice defects in the films annealed in nitrogen.

Keywords—Li, Cu doped ZnO; thin films; sol-gel; structural parameters; stress; photoluminescence; I-V characteristics.

1. Introduction

ZnO thin films doped with Li and Cu are presently investigated for applications such as light emitting diodes, solar cells, gas sensors, transparent electrodes due to improved properties acquired in the presence of these doping elements [1, 2].

One of the most attractive methods used for the synthesis of doped ZnO thin films given the accurate control of the composition is the sol-gel method. The precursor type, the deposition rate and the temperature of pre-treatment for each deposited layer, as well as the temperature and time of the final annealing have been extensively analyzed in relationship with the films structure and physical properties [3]. Nevertheless, the effect of annealing atmosphere as a significant factor that can affect the structural and opto-electric properties of ZnO thin films still has to be clarified.

In this study we comparatively investigated the structure and opto-electrical properties of ZnO thin films and the ZnO thin films doped with 3wt.% Li or Cu prepared by sol-gel method and annealed in air and nitrogen ambient.

2. Experimental Details

ZnO thin films undoped and doped with Li or Cu were deposited on Si/SiO₂ substrates by sol-gel method, using spin coating. The flowchart of the process is presented in Fig. 1.

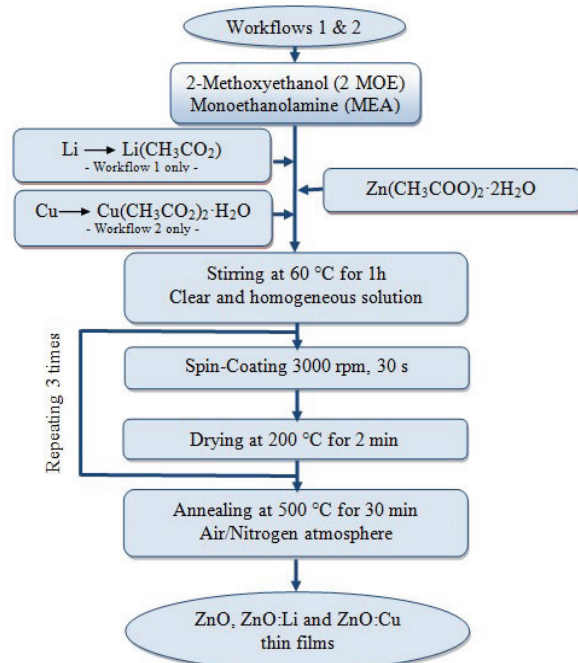


Fig. 1. Flowchart of the preparation methodology of Li and Cu doped ZnO thin film.

The process comprised two steps:

i) *Sol preparation.* The sol was prepared by dissolution of zinc acetate dehydrate, ZAD (purity 99.50%), in 2-methoxyethanol, and then a monoethanolamine (MEA) was added to the mixed solution. The total concentration of the solution was maintained at 0.35 mol/L. This precursor solution was mixed at approximately 60 °C for 1 h to yield a clear and homogeneous sol. The doping solution was prepared from lithium acetate (Li(CH₃CO₂), LiAc (99.95%)) dopant source of Li, and copper acetate

monohydrate ((Cu(CH₃CO₂)₂·H₂O), CuAc (99%)) dopant source of Cu. The concentration of lithium or copper in the solution was 3 wt. %.

ii) *Sol deposition.* The solution was spin-coated onto the substrates at 3000 rpm for 30 s. After each layer deposition, the gel film was stabilized by a pre-treatment at 200°C for 2 min. The deposition process was repeated three times and led to the formation of films with a thickness of about 60 nm. After deposition of the last coating layer, the annealing in air or in nitrogen atmosphere was carried out at 500°C for 30 min.

The surface morphology of the films was analyzed by using a Field Emission Scanning Electron Microscope (FE-SEM)-Nova NanoSEM 630. The structural characteristics were determined with a SmartLab X-ray thin film diffraction system - Rigaku at grazing incidence (GIXRD). The photoluminescence (PL) emission spectra were recorded with an Edinburgh FL920 fluorometer, at 310 nm excitation wavelength. The electrical characteristics of the films were measured by using a 4200-SCS/C/Keithley system coupled with an EP6/Suss MicroTec microprober.

3. Results and Discussion

The surface morphology of ZnO, ZnO:Li and ZnO:Cu doped thin films is presented in Fig. 2.

The FE-SEM images reveal rippled surfaces structure of the films. The structural features have a higher density on undoped ZnO films surface, Fig. 2a). Their density is reduced on the surface of the ZnO:Li doped films presented in Fig. 2b), and on the ZnO:Cu films surface, which shows larger and thicker ripples, Fig. 2c). All the films annealed in nitrogen ambient show smoother surfaces than the films annealed in air. The effect may be clearly observed in the case of the ZnO films, Fig. 2 d), and mainly in the case of ZnO:Li films, Fig. 2e), while large ripples can be observed on the surface of ZnO:Cu films presented in the Fig. 2f).

Similar rough surfaces were reported for ZnO and ZnO:Cu doped thin films prepared by sol-gel, spin-coating technique, annealed in ambient atmosphere. A primary mechanism proposed for the ripples formation takes into consideration the presence of the OR (hydroxyl or alkoxy) groups in the sol solution [4]. The structural features, fiber like stripes and wrinkles form when the films had substantial losses of those OR groups. A relatively smooth surface morphology can be

obtained when the starting materials provide enough OR groups.

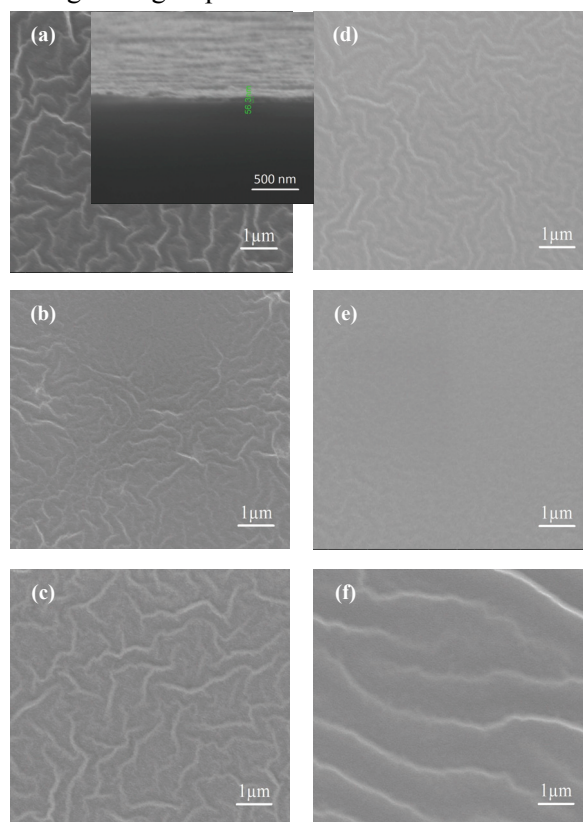


Fig. 2. FE-SEM micrographs of the ZnO (a), ZnO:Li 3wt.% (b), ZnO:Cu 3wt.% (c) thin films *annealed in air* and ZnO (d), ZnO:Li 3wt.% (e) and ZnO:Cu 3wt.% (f) thin films *annealed in nitrogen*.

Another mechanism proposed for the formation of these wrinkles is based on coating and drying steps during the deposition process [5]. It was suggested that the surface wrinkles appear due to a dried layer that is formed initially at the film-air interface and then extends inside the film, with drying time. Under the surface the layer remains wet for a period of time. The capillary forces of liquid phase generate an upward force that acts on the dried layer and causes wrinkling. In our case, we consider that the presence of the OR groups has the major effect on wrinkles formation. This phenomena is more pronounced for the films annealed in air compared with films annealed in nitrogen ambient.

All the films present a polycrystalline structure with a slight preferential orientation along (101) direction, see Fig. 3. It can be observed that the main diffraction peaks are situated at $2\theta=31.81^\circ$, 34.44° , 36.29° which corresponds to (100), (002), (101) planes of ZnO wurtzite structure.

The peaks correspond to the standard (JCPDS card No. 00-036-1451).

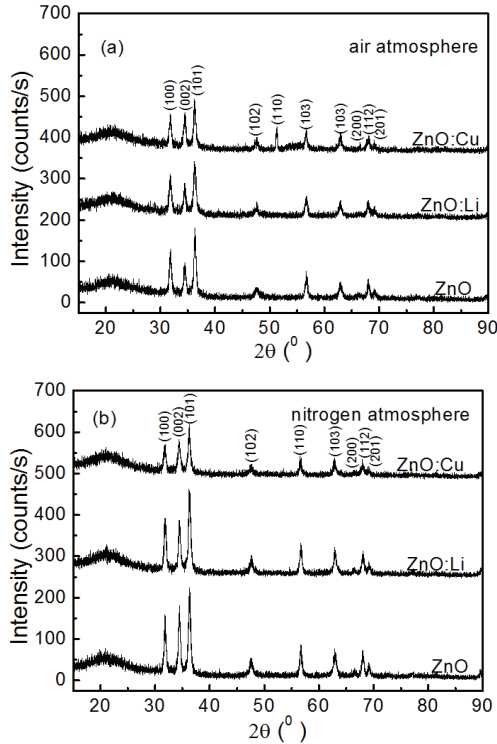


Fig. 3. XRD patterns of ZnO, ZnO:Li 3wt.%, ZnO:Cu 3wt.% thin films annealed in air (a) and in nitrogen (b).

The lattice constants a , c calculated from the diffraction measurements are listed in Table 1.

Table 1. The lattice constants a and c of undoped and of the Li, Cu:ZnO doped thin films.

Thin film	Annealing atm.	a [Å]	c [Å]	c/a
ZnO	air	3.247	5.202	1.6023
ZnO:Li		3.244	5.202	1.6036
ZnO:Cu		3.245	5.203	1.6036
ZnO	nitrogen	3.252	5.202	1.6000
ZnO:Li		3.245	5.210	1.6056
ZnO:Cu		3.248	5.213	1.6051
JCPDS	air	3.250	5.207	1.6023

The XRD patterns shown in Fig. 3 a) reveal that the doped films have a reduced crystallinity comparatively with the undoped ones. This result is consistent with the previous reports which evidenced the deterioration of the crystallinity in Cu doped ZnO films when concentration of Cu increases [6]. The difference between the ionic radii of Zn^{2+} , 0.074, and of Li^+ , Cu^+ , Cu^{2+} ions, 0.060, 0.096 and 0.072 nm respectively, may cause a lattice distortion and the deterioration of the films crystallinity. The XRD patterns of the films annealed in nitrogen atmosphere are

presented in Fig. 3 b). The intensity of (100), (002), (101) peaks is increased in the patterns of the undoped and ZnO:Li doped films, still it remains unchanged in the pattern of ZnO:Cu film.

The doped films annealed in air show a slight decrease in the lattice parameter a comparatively with the undoped films and standard value, as may be observed in Table 1. The same tendency is observed for the films annealed in nitrogen. Moreover, in this latter case, the doped films have larger values of c parameter comparatively with the undoped films and the standard value. The c/a values listed in Table 1 show significant variations comparatively with the ideal hexagonal structure especially for the films annealed in nitrogen. These variations suggest that the lattice strain, ε , and residual stress, σ , induced by doping impurities change as function of the annealing ambient.

The average crystallite size in the films, D , and the values of lattice strain ε and of the residual stress, σ , resulted from the XRD measurements are presented in Table 2.

Table 2. The average crystallite size D , the lattice strain ε and the residual stress, σ for the films annealed in air and nitrogen.

Thin films	Atm.	D (nm)	ε [%]	σ [GPa]
ZnO	Air	17	0.23	0.18
ZnO:Li		13	0.11	0.18
ZnO:Cu		15	0.12	0.13
ZnO	N ₂	16	0.12	0.18
ZnO:Li		18	0.24	-0.18
ZnO:Cu		12	0.21	-0.31

The average crystallite size D and the lattice strain, ε values were calculated using the Rietveld method. The residual stress in the films is determined from the relation [7]:

$$\sigma = -233 \cdot \frac{c_{film} - c_{bulk}}{c_{bulk}} [\text{GPa}]$$

where c_{film} is the lattice constant of ZnO thin films and $c_{bulk}=5.206$ Å is the lattice parameter of ZnO in the bulk, that is the unstrained lattice parameter.

The data listed in Table 2 show that the stress is tensile in all the films annealed in air and becomes compressive in the doped films annealed in nitrogen. This observation suggests that, more than being related to the difference in the ionic radius between Zn and Li or Cu, the

residual stress is determined by the mechanism by which impurities are incorporated into the ZnO lattice when the annealing is carried out in air or in nitrogen.

Fig. 4 shows the photoluminescence spectra of the films annealed in air and nitrogen ambients.

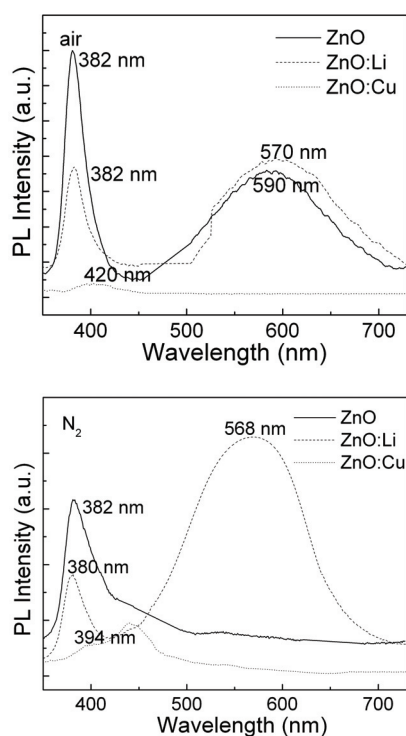


Fig. 4 PL spectra of the films annealed in air (a) and nitrogen (b).

The PL emission spectra of the films annealed in air, Fig. 4 a), show emission bands situated at about 380 nm (3.26 eV) related to near band edge emission and a broad emission band peaked at about 570 nm which has been attributed to structural point defects such as oxygen vacancies or interstitial atoms [8]. The emission related to point defects almost vanished in the case of ZnO and ZnO:Cu films annealed in nitrogen as can be observed in Fig. 4 b). At the same time an intense and well resolved emission band situated at 569 nm (2.17 eV) is present in the PL spectrum of ZnO:Li films. Our results evidence that the annealing in nitrogen strongly reduces these types of point defects in ZnO and ZnO:Cu films, but not in ZnO:Li films. The measured resistivity values are $8.27 \times 10^5 \Omega \cdot \text{cm}$ to $2.35 \times 10^4 \Omega \cdot \text{cm}$ for ZnO annealed in air and nitrogen atmosphere, respectively. Also, the resistivity decreases from $1.05 \times 10^6 \Omega \cdot \text{cm}$ in ZnO:Li films annealed in air to a value of $1.31 \times 10^5 \Omega \cdot \text{cm}$ in ZnO:Li films annealed in nitrogen atmosphere.

4. Conclusions

Thin films of ZnO and Li or Cu 3 wt.% doped ZnO have been prepared by sol-gel spin-coating method and were annealed in air and in nitrogen atmosphere.

The annealing in nitrogen atmosphere leads to ZnO and ZnO:Li thin films with improved surface morphology and crystallinity. The PL emission related to lattice defects is strongly reduced in ZnO and ZnO:Cu films, but not in ZnO:Li films, suggesting that different mechanisms assist Li and Cu incorporation in ZnO. The resistivity of ZnO and ZnO:Li films annealed in nitrogen decreases by an order of magnitude comparatively with the films annealed in air.

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