

EFFECT OF TEMPERATURE AND FILM THICKNESS ON RESIDUAL STRESS AND MICROSTRUCTURE OF ZnO THIN FILMS

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ABSTRACT

ZnO precursor solutions were synthesized by sol-gel technique. Microstructure and residual stress of the ZnO thin films were examined as a function of temperature and film thickness. ZnO thin films were grown on glass substrate using sol-gel dip-coating method and annealed at various temperatures (500-700 °C) for 30 min under air. Residual stress for ZnO /glass substrate was determined analytically. The structure of thin films were characterized by X-Ray diffraction. The surface morphologies and microstructure of ZnO samples were investigated by Scanning Electron Microscope.

Keywords: Residual stress, Sol-gel, Thin films

1. INTRODUCTION

ZnO-based nano materials are widely used in many commercial and technological applications such as optoelectronics, cosmetics, surface acoustic wave devices, spintronic, varistors, and biomaterials [1-6]. Therefore, many researchers focuses on the new properties of ZnO nano materials depending on synthesizing conditions using many methods. Among these methods the Materials synthesis by sol gel method exhibits formation of cracks because of annealing temperature. It is necessary to figure out the effect and formation of cracks so that a batter coating could be made. Therefore to understand the residual stresses is essential.

The aims of this study are to investigate characterization and processing parameters of ZnO thin films on glass substrate and to calculate analytically residual stresses for the homogeneous ZnO thin films.

2. EXPERIMENTAL PART

ZnO solutions were synthesized by sol-gel technique using the chemicals, Zinc acetate dehydrate (Fluka), methanol (CH₃OH) and monoethanolamine (MEA, C₂H₇NO) were used to obtain clear homogeneous solutions. Glass substrate were dipped into the Zn₀O solution and then pulled through the vertical furnace. The further details of preparation procedure of ZnO thin films were clearly detailed in the literature [1-5].

Phase identification of ZnO samples was revealed by Rigaku diffractometer with Cu K_a radiation ($\lambda=1.5418 \text{ \AA}$) for 2θ scan between 10 and 90 degrees. Sample morphologies were investigated by means of monitored images with the model of JEOL, JSM-5910LV scanning electron microscope (SEM) tool.

3. RESULTS AND DISCUSSION

3.1 Structural Analysis

X-ray diffraction of ZnO thin films deposited by sol-gel dip coating technique at 600°C was observed in Fig. 1. The pattern is identified to be a single phase ZnO with wurtzite hexagonal structure.

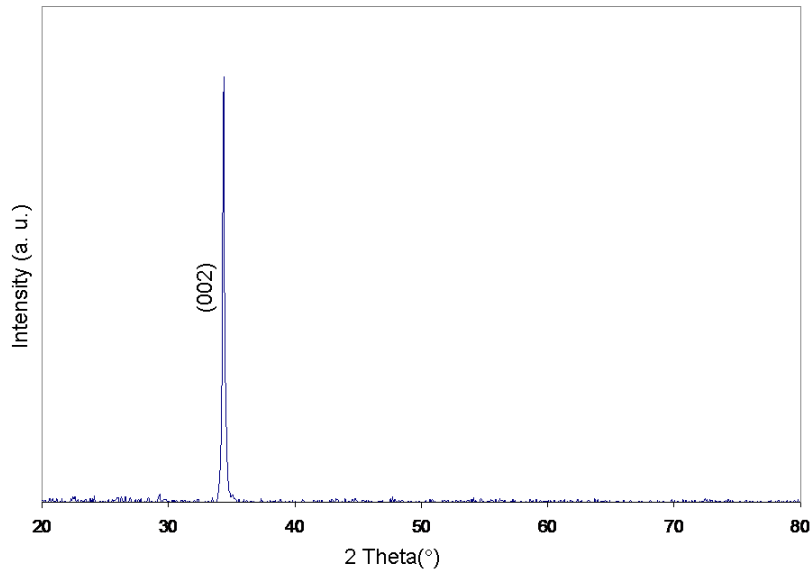


Fig.1 The x-ray diffraction patterns of the ZnO thin film for thickness 207nm.

Crack-free, smooth, pinhole-free, dense and uniform thin films are grown on glass substrate by sol-gel dip coating process. Figs. 2a, and 2b show the SEM surface morphology of ZnO thin film. The thin films depict smooth, dense and crack and pinhole free surface. The thickness of the the thin film was approximately 207 nm as shown Fig 2c.

3.2 Thermal Stress of ZnO/Glass substrate

Thickness of thin film in the top and bottom of substrate illustrated in Fig. 3. As shown in the Fig. 3, the width of ZnO/Glass substrate system is very long than the thickness [7-9], So the stress components in the middle of system was calculated as a thick plate. The residual stress analysis for glass substrate with layers (ZnO) in the middle can be analyzed considering stress components are completely free of surface traction [10]. The further details of calculation of the residual stress analysis were clearly detailed in the previous studies [7-9].

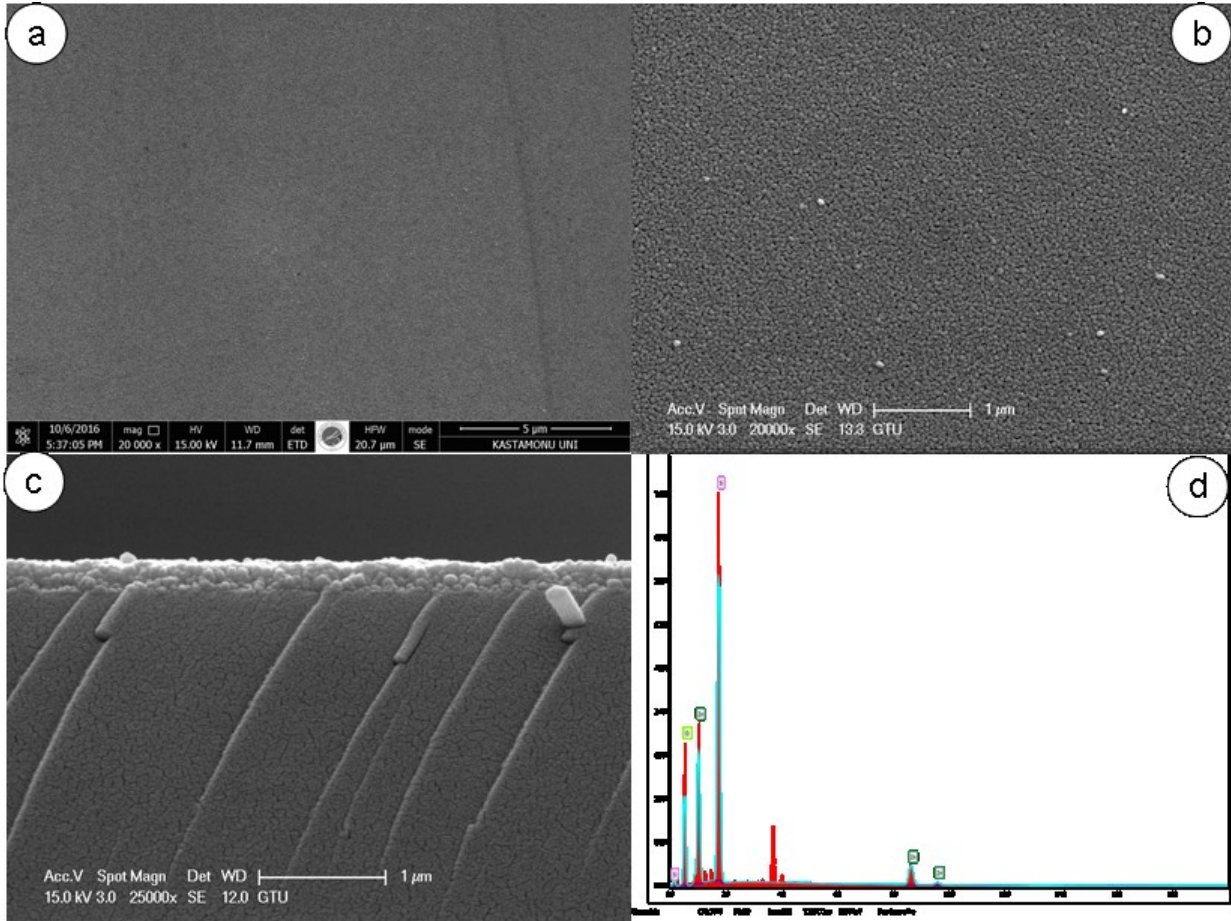


Fig. 2: SEM micrographs of a) and b) ZnO, c) Cross-section SEM micrograph d) EDS of ZnO film on glass substrate.

Table 1 Variation of stress components

Film thickness (nm)	$\sigma_{xx}=\sigma_{zz}$ (GPa)			$\sigma_{xx}=\sigma_{zz}$ (MPa)		
	ZnO Thin Film			Glass Substrate		
	500°C	600°C	700°C	500°C	600°C	700°C
124	0.22	0.26	0.31	-0.05	-0.06	-0.07
207	0.22	0.26	0.31	-0.08	-0.1	-0.12
348	0.22	0.26	0.31	-0.015	-0.18	-0.22

The stress component are calculated for ZnO/glass substrate system as a function of temperature between (500° C, 600° C, and 700°C), film thickness (124nm, 207nm and 348nm) and substrate thickness 0.1 mm. Determined values of stress components, $\sigma_{xx} = \sigma_{zz}$ are given in Table 1. As seen from Table 1 stress component values of glass substrate are compressive, but stress component values of thin films are tension.

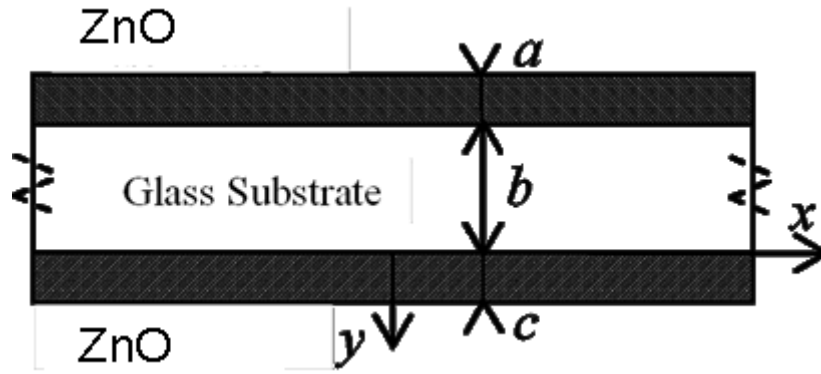


Fig. 3 Section of Glass substrate and ZnO

4. CONCLUSIONS

ZnO thin films were prepared on glass substrate by using a sol-gel technique. Residual stress analysis and microstructure of ZnO thin films on glass substrate are examined as a function of film thicknesses and temperature. It is found that stress component values of glass substrate are compressive, but stress component values of thin films are tension, so we conclude that ZnO thin films could be prepared by using sol-gel dip coating technique.

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5. REFERENCES

- [1] A. Gungor and L. Arda, 11 th International Research/Expert Conference, “Trends in the Development of Machinery and Associated Technology”, TMT (2007), 1543-1546
- [2] A. Gungor, D. Akcan, L. Arda, 18 th International Research/Expert Conference, “Trends in the Development of Machinery and Associated Technology”, TMT (2014), 133-136
- [3] L. Arda, D. Akcan, a. Güngör, Journal of Trends in the Development of Machinery and Associated Technology Vol. 19, No. 1, 2015, ISSN 2303-4009 (online), p.p. 61-64
- [4] A. Gungör, D. Akcan, L. Arda, Journal of Trends in the Development of Machinery and Associated Technology Vol. 19, No. 1, 2015, ISSN 2303-4009 (online), p.p. 65-68
- [5] Z. K. Heiba and L. Arda, Structural properties of Zn_{1-x}Mg_xO nanomaterials prepared by sol-gel method“, Cryst. Res. Technol. 44, (2009) No. 8, 845-850.
- [6] Z.K. Heiba, L. Arda, XRD, XPS, optical, and Raman investigations of structural changes of nanoCo-doped ZnO, J. Mol. Struct. 1022 (2012), pp. 167–171.
- [7] L. Arda, S. Ataoglu, O. Bulut, IEEE Trans. Appl. Supercon, 19 (2009) 3291–3294
- [8] S. Ataoglu, L. Arda, N. Kadioglu, O. Cakiroglu, O. Bulut, A. N. Gulluoglu, and I. Belenli, , IEEE Trans Appl. Supercon, 19 (2009) 2375–2378
- [9] L. Arda, S. Ataoglu, Z. Abdulaliyev, O.A. Sacli, J. of Alloys and Compounds 470 (2009) 404–407
- [10] B.A. Boley and J.H. Weiner, Theory of Thermal Stresses. New York: Wiley, 1960