EFFECT OF EDX ACCELERATING VOLTAGE ON ZnO THIN FILM DOPED WITH SODIUM

Benedict Wen-Cheun Au¹, Kah-Yoong Chan^{1*}, Siow-Woon Ng², Fong-Kwong Yam²

¹Centre for Advanced Devices and Systems, Faculty of Engineering, Multimedia University, Persiaran Multimedia, 63100 Cyberjaya, Selangor, Malaysia.

²School of Physics, University Sains Malaysia, 11800 Penang, Malaysia.

**Corresponding author: kychan@mmu.edu.my*

Abstract

In recent years, owing to its unique properties, ZnO has been researched extensively for a wide range of applications including sensors, transistors and solar cells. In this work, ZnO thin film doped with sodium (ZnO:Na) was characterized using Field Emission-Scanning Electron Microscopy (FESEM) and Energy Dispersive X-Ray Spectroscopy (EDX). The FESEM image of the ZnO:Na film implies crystal growth at different orientations. The surface composition of the ZnO:Na film with approximately 350 nm thickness was investigated by EDX as a function of accelerating voltage ranging from 5 kV to 15 kV. The EDX spectra revealed that 7 kV is the most appropriate accelerating voltage for extracting elemental composition of the ZnO:Na films, which exclude the detection of the underlying substrate elements. A rectifying structure made up of P-type ZnO:Na film and N-type tin doped indium oxide (ITO) film showed typical rectifying characteristic.

Keywords: P-type ZnO:Na film, EDX accelerating voltage, FESEM, rectifying structure.

Introduction

In recent years, ZnO has been the hot topic of research due to its distinctive properties. It is a wide bandgap semiconductor of 3.37 eV with a large excitonic binding energy of 60 meV at room temperature (Janotti & Van de Walle, 2009). The distinctive properties of ZnO have drawn much attention among researchers owing to its promising applications such as sensors (Kakati et al., 2010), transistors (Fortunato et al., 2005) and solar cells (Pathak et al., 2016). Generally, ZnO is an N-type semiconductor due to the presence of native defects (Selim et al., 2007). However, P-type doping in ZnO has rarely been attempted due to the self-compensation effect in ZnO (Lai et al., 2011). In this work, sodium doped ZnO (ZnO:Na) film with thickness around 350 nm was fabricated using sol-gel spin-coating method on glass substrates. The surface morphology of the undoped and ZnO:Na films was measured. The effect of EDX accelerating voltage on the chemical composition of the ZnO:Na film was studied.

Materials and Method

Fabrication of ZnO and ZnO:Na films

Figure 1 presents the fabrication process of the ZnO:Na film. The ZnO:Na film was fabricated using zinc acetate dihydrate $[Zn(CH_3OO)_2 \cdot 2H_2O]$ as precursor for ZnO and sodium chloride (NaCl) as source of Na doping in ZnO. Isopropanol was used as solvent while monoethanolamine was used as stabilizer for preparing the sol-gel solution. The molarity of the ZnO precursor was set at 0.5 M. The mixture of chemicals containing the ZnO

precursor, Na dopant, solvent and stabilizer was stirred and aged at room temperature to yield a homogeneous transparent solution. The resulting mixture of sol-gel solution was spincoated on glass substrates. Finally, the spin-coated films were post-annealed at an elevated temperature.

Structural Characterisation

The surface morphology of the films was measured by the field emission-scanning electron microscopy (FESEM). The Energy-dispersive X-ray (EDX) was used to study the elemental composition of the doped ZnO films.



Figure 1 The fabrication process of the ZnO:Na film.

Result and Discussion

Characterisation of ZnO and ZnO:Na films

The scanning electron microscopy is a characterization technique where a focused electron beam is used to scan a sample surface. The electrons interact with the atoms in the sample and provide information on surface topography. Figure 2 shows the surface morphology of the undoped ZnO and ZnO:Na film. The undoped ZnO film exhibits uniformly distributed structures, densely packed across the growing surface. On the other hand, the ZnO:Na film has no definite shape and the growth seems to be disorientated. This observation may be due to the introduction of Na in ZnO, which causes different growth orientation.



Figure 2 The surface morphology of (a) undoped ZnO and (b) ZnO:Na film.

The EDX is an analytical method for the elemental and compositional analysis of a sample. In addition, the EDX relies on the interaction between X-ray excitation and the sample to provide elemental information of a sample. Figure 3(a) and (b) present the comparison of the undoped and ZnO:Na film using EDX measurements. It can be seen that Na was detected in the ZnO:Na film which is not revealed in EDX spectrum for the undoped ZnO film. This confirms the successful incorporation of sodium in the ZnO film matrix.



Figure 3 EDX spectra of (a) ZnO and (b) ZnO:Na films.

In addition to these, Figure 4 shows the EDX spectra for the fabricated ZnO:Na films at different accelerating voltages. From the EDX spectra in Figure 4(a) and (b), the elements detected are oxygen (O), sodium (Na) and zinc (Zn).



Figure 4 The EDX spectra of ZnO:Na film at (a) 5 kV, (b) 7 kV, (c) 10 kV and (d) 15 kV accelerating voltage.

Figure 4(c) illustrates the EDX spectrum of the ZnO:Na film at accelerating voltage of 10 kV. Compared to Figure 4(b), silicon (Si) is present in the EDX spectrum. When the accelerating voltage is further increased to 15 kV, higher silicon content and more elements are being detected along with O, Na, and Zn.

The results obtained in Figure 4(c) and 4(d) show that the high accelerating voltage penetrates beyond the ZnO:Na film and into the glass substrate. Further increase in

accelerating voltage causes further penetration into the glass substrate. Therefore, detecting more elements that are not peculiar to the ZnO:Na film.

In order to verify the P-type behaviour of the ZnO:Na film, the ZnO:Na film was integrated into a rectifying structure on commercially available N-type tin doped indium oxide (ITO) coated glass. The ZnO:Na/ITO structure exhibited typical rectifying characteristic as shown in Figure 5, which corroborates the P-type characteristics of the ZnO:Na film. Besides that, the rectifying structure has a turn-on voltage of around 1.2 V and an output current of around 35 mA at forward voltage of 5 V.



Figure 5 I-V curve of the ZnO:Na/ITO rectifying structure.

Conclusion

The sol-gel spin-coated ZnO thin films doped with Na were characterized using SEM and EDX. SEM results showed ZnO growth at different orientations when doped with Na. EDX results revealed that the most appropriate accelerating voltage is 7 kV. For accelerating voltages greater than 7 kV, the electron beam penetrates deeper into the ZnO film and reaches the glass substrate. As a result, the high accelerating voltage causes the detection of elements in the ZnO film as well as the elements in the glass substrate. The I-V curve obtained from the ZnO:Na/ITO rectifying structure confirmed the P-type characteristic of the ZnO:Na film.

Acknowledgement

This research work was funded by the Malaysian Ministry of Higher Education (MoHE)'s Fundamental Research Grant Scheme (FRGS). We would like to thank MoHE for their financial support in this work.

Conflict of Interests

There is no conflict of interests.

Reference

Fortunato, E., Barquinha, P., Pimentel, A., Goncalves, A., Marques, A., Pereira, L., & Martins, R. (2005). Recent advances in ZnO transparent thin film transistors. *Thin Solid Films*, 487(1–2), 205–211.

Janotti, A., & Van de Walle, C. G. (2009). Fundamentals of zinc oxide as a semiconductor. *Reports on Progress in Physics*, 72(12), 126501.

Kakati, N., Jee, S. H., Kim, S. H., Oh, J. Y., & Yoon, Y. S. (2010). Thickness dependency of sol-gel derived ZnO thin films on gas sensing behaviors. *Thin Solid Films*, *519*(1), 494–498.

Lai, J. J., Lin, Y. J., Chen, Y. H., Chang, H. C., Liu, C. J., Zou, Y. Y., ... Wang, M. C. (2011). Effects of Na content on the luminescence behavior, conduction type, and crystal structure of Na-doped ZnO films. *Journal of Applied Physics*, *110*(1), 2177–2184.

Pathak, T. K., Kumar, V., Swart, H. C., & Purohit, L. P. (2016). Electrical and optical properties of p-type codoped ZnO thin films prepared by spin coating technique. *Physica E: Low-Dimensional Systems and Nanostructures*, 77, 1–6.

Selim, F. A., Weber, M. H., Solodovnikov, D., & Lynn, K. G. (2007). Nature of native defects in ZnO. *Physical Review Letters*, 99(8).