VOL. 18, NO. 15, AUGUST 2023 ISSN 1819-6608
ARPN Journal of Engineering and Applied Sciences

www.arpnjournals.com

ANALYSIS OF MATERIAL COMPOSITION WITH ENERGY DISPERSIVE OF X-RAY SPECTROSCOPY METHOD

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ABSTRACT

ZnO is a semiconductor material with an energy band gap of 3.37 eV at room temperature, this causes ZnO to work in the UV light range. In this study, ZnO material doped with Nitrogen with a doping percentage was 10%. Deposition of ZnO: N material was carried out on a glass substrate using a spray coating technique. A composition test was performed using SEM-EDX. SEM-EDX is a device that has a wide magnification range to observe a sample from an overall view, with images of nanostructures at very high magnification. SEM-EDX serves to analyze topography, morphology, composition, and scale information from SEM image images on a material. Composition testing using SEM-EDX tools can result in pure impurity, pure oxide, and pure. For the results of pure impurity test results were obtained in the form of elements of elements C, N, O, and Zn with a mass of 14.46%, 0.53%, 54.70%, and 30.30%. For the oxide test on ZnO doping N 10%, the results were obtained in the form of elements of N, O, and Zn elements, each with a mass of 2.45%, 19.17%, and 78.38%. For pure test on ZnO doping N 10%, the results are in the form of elements of N, O, and Zn elements, each with a mass of 0.90%, 57.80%, and 41.31%.

Keywords: ZnO doping, spin coating, SEM-EDX.

Manuscript Received 7 March 2023; Revised 28 September 2023; Published 11 October 2023

INTRODUCTION

ZnO is a semiconductor material with an energy band gap of 3.37 eV at room temperature, this causes ZnO to work in the UV light range (Reddy *et al.*, 2013). ZnO has properties such as high chemical stability, good electrical properties, high light transmittance, can coat substrates very well and strongly, optical and piezoelectric, bio-safe, biocompatible, and can be used for biomedical applications without a coating process. Gupta et al., 2006; ivanova *et al*., 2011).

Increasing the concentration of nonmetal doping on ZnO will change its energy level so that it can improve its physical and optical properties (Chauhan *et al.,* 2010; Das *et al*., 2015; Lu *et al.*, 2016; Ellmer *et al.*, 2008). The addition of doping Nitrogen (N) generally increases the crystal grain size and reduces the energy gap width by up to 2.9378 eV compared to ZnO without doping. The N-doped ZnO layer has a transmittance value with a steeper pattern than the un-doped ZnO material in the wavelength range of 300 nm to 400 nm (Sutanto *et al*., 2017). The tool commonly used to characterize the composition of ZnO:N materials is SEM-EDX.

SEM-EDX is a tool that has a wide magnification range for observing a sample from an overall view, with an image of the nanostructure at very high magnification. SEM-EDX is equipped with analytical capabilities to locate elements in the sub-micro region. SEM-EDX serves to analyze the topography, morphology, composition, and scale information from SEM images on a material.

MATERIAL AND METHOD

This research uses a glass substrate as a preparation. The glass substrate was cleaned with acetone and methanol to remove dust and oil impurities, then wiped using a tissue moistened with distilled water.

ZnO:N 10% solution was prepared by dissolving 3.046 Gr of hydrated zinc acetate into 26 Ml isopropanol at room temperature with a concentration of 0.3 M Zinc Acetate. The solution was then stirred for 15 minutes at 60 0C, then 0.831 was added. Ml solution of Monoethanolamine (MEA) in a stirrer until the solution is homogeneous. 15 minutes later, still in a stirrer state, 0.083 g of urea was added to the solution and waited for 15 minutes.

Then the material deposition process is carried out. The process of deposition of a thin layer of ZnO:N on a glass substrate using the Spray Coating technique. The dried glass substrate was placed on a hot plate at 450 0C for 60 minutes. Then at the same temperature the ZnO:N solution is sprayed on the glass substrate evenly. The spray process is carried out with a pause of 10 seconds on each spray. After the spray process is complete, the sample is allowed to stand for 60 minutes still at the same temperature, then after 60 minutes, the temperature is lowered slowly to 0 0C.

The characterization of the ZnO:N thin film used a JEOL JSM 6510 SEM-EDX tool to see the composition contained in the 10% ZnO:N thin film sample. The

characterization of the sample using the SEM-EDX tool was carried out using the coating method.

RESULTS AND DISCUSSIONS

This study was conducted to determine the elemental composition contained in ZnO material that has been doped by Nitrogen with a doping percentage of 10%. Figure 1 is an image of the deposition of material on a glass substrate using a spray coating technique with an annealing temperature of 450 ℃.

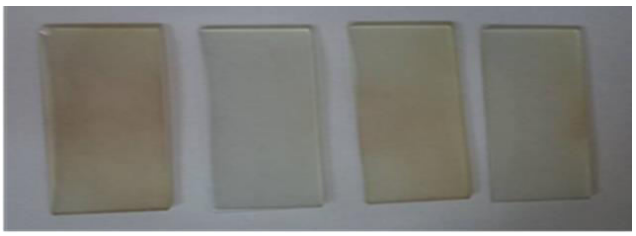

Figure-1. Characterization of 10% ZnO:N Thin Film Using SEM-EDX.

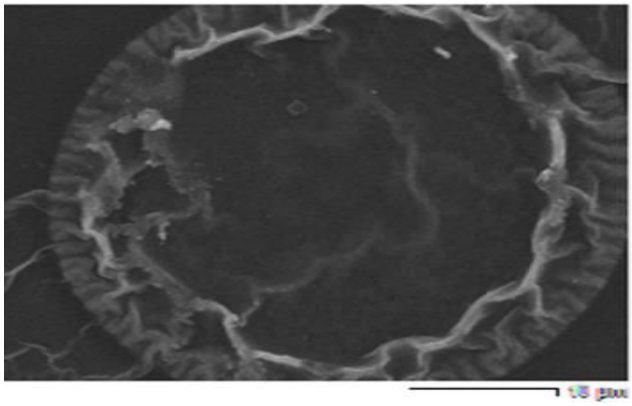

Figure-2. SEM image of N-doped ZnO with 3000× magnification

Figure-2 shows the morphology of the 10% ZnO:N material as a result of SEM images. Based on the picture above, it can also be seen the graph of the composition of the ZnO:N material in the form of the EDX test results is shown in Figure-3.

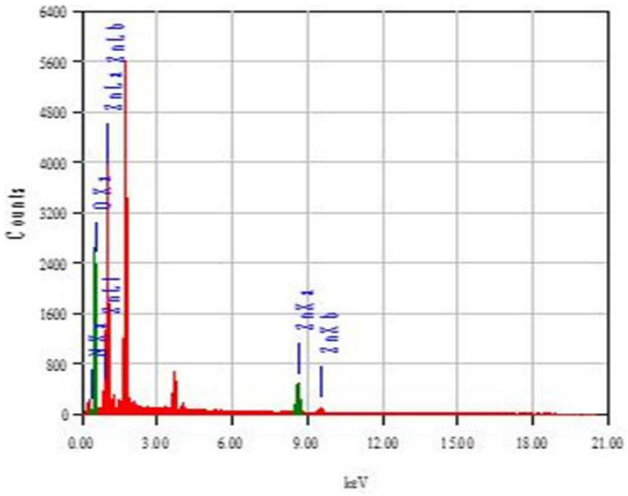

Figure-3. Pure impurity.

Figure-3 is a graphic image of the EDX test results in the form of pure impurity. Based on the Figure-3, it can be seen that the 10% ZnO:N sample contains several elements. The composition of each element can be seen in the Table-1.

Table-1. The composition of ZnO:N 10% is pure impurity.

Element	KeV	Massa (%)	Sigma	Atom (%)	K
C K	0.227	14.45	0.06	23.50	3.5008
N K	0.392	0.53	0.11	0.74	0.6632
O K	0.525	54.70	0.14	66.71	66.7324
Zn K	8.630	30.30	0.22	9.05	29.1037
Total		100.00		100.00	

Table-1 is the scan result of the EDX test of ZnO material that has been doped with 10% N element with an energy range of 0-20 KeV. The table above shows the results of the EDX test in the form of pure impurity. Based on the table above, it can be seen that there are elements C, N, O, and Zn each with a mass of 14.46%, 0.53%, 54.70%, and 30.30%. it can be seen that the largest mass is in the element O. The element carbon arises because, during sample preparation, the sample is attached to the specimen using carbon tape. From the EDX test, it can also be seen that the number of atoms contained in each element is 23.50%, 0.74%, 66.71%, and 9.05%.

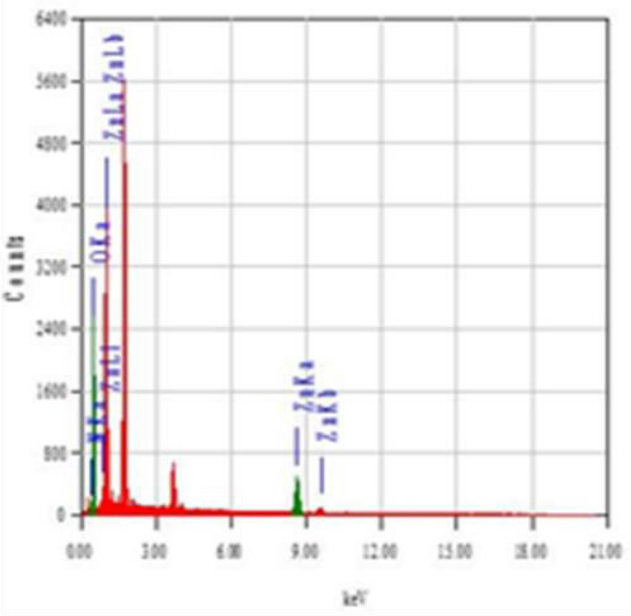

Figure-4. Pure oxide.

Figure-4 shows an image of the EDX test results in the form of pure oxide. Based on the graph above, it can be seen that the 10% ZnO:N sample contains several elements. The composition of each element can be seen in the table below.

Table-2. Composition of ZnO:N 10% in the form of pure oxide.

Elemen	KeV	Massa (%)	Sigma	Mol (%)	Compoud	Massa (%)	Cation	K
N K	0.392	2.45	0.35	12.73	N	2.45	0.00	4.05
O		19.17						
Zn K	8.630	78.38	1.66	87.27	ZnO	97.55	24.00	95.95
Total		100.00		100.00		100.00	24.00	

Table-2 shows the scan result of the EDX test for ZnO material that has been doped with 10% N element with an energy range of 0-20 KeV. The table above shows the results of the EDX test in the form of pure oxide. Based on Table-2, it can be seen that there are elements of N, O and Zn with a mass of 2.45%, 19.17%, and 78.38%, respectively. It can be seen that the largest mass is in the element Zn. Element O arises because of the oxidation process. The table also shows that the ZnO component was formed with a mass of 97.55% and still contained N elements but with a small mass of 2.45%. Figure 5 shows a graphic image of the EDX test results in the form of pure. Based on the graph above, it can be seen that the 10% ZnO:N sample contains several elements. The composition of each element can be seen in the Table-3.

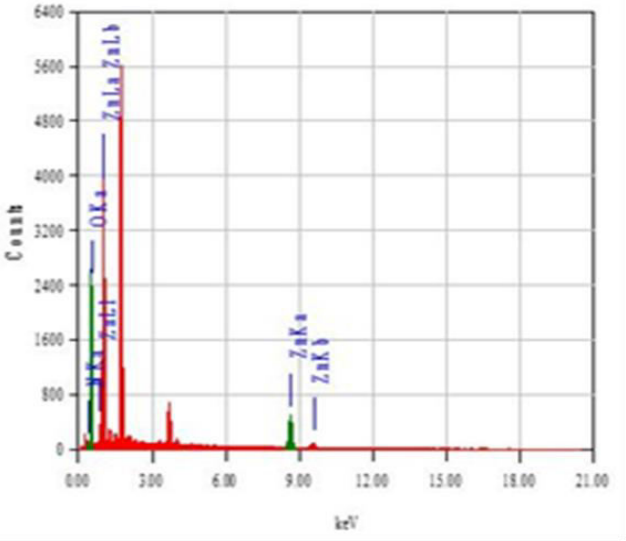

Figure-5. Pure.

Table-3 shows the result of the EDX test scan for ZnO material that has been doped with 10% N element with an energy range of 0-20 KeV. The table above shows the results of the EDX test in the form of pure. Based on the table above, it can be seen that in the K shell, there are elements of N, O and Zn with a mass of 0.90%, 57.80%, and 41.31%, respectively. It can be seen that the largest mass is in element O. From the EDX test, it can also be seen that the number of atoms contained in each element is 1.48%, 83.85%, and 14.67%. The number of atoms of element O and element Zn should be the same, but the results show that the number of atoms of element O is greater than that of Zn because of the many oxidation processes so element O requires more energy than element N, which is 0.525 KeV.

Table-3. The composition of ZnO:N 10% is pure.

Elemen	KeV	Massa (%)	Sigma	Atom (%)	K
N K	0.392	0.90	0.10	1.48	1.27
O	0.522	57.80	0.15	83.85	68.76
Zn K	8.630	41.31	0.31	14.67	29.98
Total		100.00		100.00	

CONCLUSIONS

The EDX tool can analyze ZnO samples doped with 10% N. The results obtained are pure impurity, pure oxide, and pure test results. For the pure impurity test results were obtained in the form of elements C, N, O, and Zn with a mass of 14.46%, 0.53%, 54.70%, and 30.30%, respectively. It can be seen that the largest mass is in element O. From the EDX test, it can also be seen that the number of atoms contained in each element is 23.50%, 0.74%, 66.71%, and 9.05%. For the oxide test on 10% N-doped ZnO, the results were elements N, O, and Zn with masses of 2.45%, 19.17%, and 78.38%, respectively. The component formed is ZnO with a mass of 97.55% and there is still an N element but with a small mass of 2.45%. For the pure test on 10% N-doped ZnO, the results obtained were elements of N, O, and Zn with masses of 0.90%, 57.80%, and 41.31%, respectively. From the EDX test, it can also be seen that the number of atoms contained in each element is 1.48%, 83.85%, and 14.67%.

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