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FABRICATION OF A THIN FILM GAMMA RADIATION SENSOR USING NANO METAL OXIDES

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ABSTRACT

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Copyright © 2024 by the authors. This article is an open-access article distributed under the terms and conditions of Creative Commons Attribution-Share Alike 4.0 International Public License (CC BY-SA 4.0) The development of nuclear technology has led to increased interest in radiation monitoring systems based on sensors and dosimeters that detect and measure ionizing radiation in a given environment. A gamma radiation (GR) sensor detects the presence of GR, while a GR dosimeter measures the amount of GR to which a person or object has been exposed. A thin film of Nano CuO/ZnO with a 90%:10% ratio on a Cu substrate was chosen to be fabricated using sol-gel method for GR sensing and dosimetry. This choice was the result of simulation of some metal oxides using a MATLAB program in one of my previous studies. The film was characterized as nano CuO/ZnO using X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray (EDXS) spectroscopy. Two silver paste electrodes were deposited on the film for Current-Voltage (I-V) characterization, which showed that the sensor behaved like a diode. The sensitivity of the sensor to GR was studied by irradiating it with different low doses of GR. The I-V curves of the sensor were measured after each irradiation, and the current-dose (I-D) curve was studied and plotted at a voltage of 0.5 V from which an equation for calculating the accumulated dose was deducted. The results show that the nano CuO/ZnO thin film sensor has promising properties for GR dosimetry applications in which the accumulated low dose of GR can be calculated.

KEYWORDS: Nano technology, Sol-gel method, nano metal oxide, Gamma sensor, Gamma dosimeter.

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الملخص

أدى تطور التكنولوجيا النووية إلى زيادة الاهتمام بأنظمة مراقبة الإشعاع القائمة على أجهزة الاستشعار ومقاييس الجرعات التي تكتشف وتقيس الإشعاعات المؤينة في بيئة معينة. يكتشف مستشعر إشعاع جاما وجود إشعاع جاما، بينما يقيس مقياس جرعات

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إشعاع جاما مقدار إشعاع جاما الذي تعرض له الشخص أو الجسم. تم اختيار طبقة رقيقة من نانو CuO/ZnO بنسبة 20%:01% على ركيزة النحاس لتصنيعها باستخدام طريقة sol-gel لاستشعار إشعاع جاما وقياس الجرعات. وكان هذا الاختيار نتيجة محاكاة بعض أكاسيد المعادن باستخدام برنامج MATLAB في دراسة سابقة لي. تم وصف الطبقة بأنه نانو CuO/ZnO باستخدام حيود الأشعة السينية (XRD)، والمجهر الإلكتروني الماسح (SEM)، والتحليل الطيفي للأشعة السينية المشتتة من الطاقة (EDXS). تم ترسيب قطبين من عجينة الفضة على الطبقة النانونية لتوصيف الجهد الحالي ، مما أظهر أن منتقد من إستخدام حيود الأشعة السينية (XRD)، والمجهر الإلكتروني الماسح (SEM)، والتحليل الطيفي للأشعة السينية المستشعر يتصرف مثل الصمام الثنائي. تمت در اسة حساسية المستشعر تجاه إشعاع جاما من خلال تشعيعه بجر عات منخفضة مختلفة من إشعاع جاما. تم قياس منحنيات المستشعر التيار - الجهد بعد كل تشعيع، وتمت در اسة منحنى التيار - الجرعة الحالي ورسمها بجهد 0.5 فولت تم استنتاج منه معادلة حساب الجرعة المتراكمة. أظهرت النتائج أن مستشعر النانوي الأعشية الرقيقة ورسمها بجهد 0.5 فولت تم استنتاج منه معادلة حساب الجرعة المتراكمة. أظهرت النتائج أن مستشعر النانوي الأعشية الرقيقة إشعاع جاما.

الكلمات المفتاحية : تكنولوجيا النانو، طريقة السول جيل ، طبقات أكاسيد المعادن النانوية، مستكشف جاما، محدد جرعة جاما.

1. INTRODUCTION

"There's plenty of room at the bottom", a famous lecture by Richard Feynman in 1959, is credited with being the first to present the idea of nanotechnology (NT). Scaling issues would impact a number of physical properties, as Dr. Feynman noted in his address [1]. The development of nano-characterization instrumentation in the 1980s, such as scanning microscope (STM), atomic force microscope (AFM), made the study of nanoscale materials (NSMs) easier, which led to the growing popularity of nano-based concepts. NT has been spreading into several fields since it became simpler to conduct research on NSMs. For instance, radiation dosimetry applications benefit greatly from it [2]. Materials with at least one nanometer-scale dimension (between 0.1 and 100 nm) are known as nanomaterials (NMs). NMs exhibit unique electrical, optical, and chemical properties that differ from those of their bulk counterparts [3]. They have a higher surface-to-volume ratio, lower melting points and better electrical conductivity than bulk materials. However, doping with various elements can increase the conductivity of NMs [4].

The fabrication of nanoparticles (NPs) in the range of 0-100 nanometers has gained widespread popularity in recent decades, particularly in the field of radiation dosimetry, where NTs are used to develop radiation sensors and dosimeters. Dosimeters are devices that measure the amount of ionizing radiation that a person or object has been exposed to. They are used in a variety of settings, including nuclear medicine, radiology, and industrial safety. Dosimeters are an important tool for radiation safety. They can be used to track the amount of radiation that people are exposed to, and they can also be used to ensure that radiation levels are within safe limits. There are many different types of sensors, but they all work on the same basic principle. They contain a material that is sensitive to radiation, and when the material is exposed to radiation, it changes in some way. This change can then be measured to determine the amount of radiation that was received. The selection of a sensor type for a specific application is dependent on the energy range of the radiation to be detected.

GR sensors are often used in conjunction with gamma dosimeters. The sensor can be used to quickly survey an area for GR, while the dosimeter can be used to measure the total dose of GR that a person has been exposed to over time.

Metal oxides (MOs) are promising materials for radiation sensing and dosimetry applications due to their unique properties that can be altered by gamma rays. Various deposition

techniques, such as sol-gel, thermal evaporation, and screen printing, are used to fabricate MO films with desired properties. The extent of these changes can be correlated with the radiation dose received, providing a quantitative measure of exposure.

The current study aims at the low-cost nano GR sensor fabrication using sol-gel deposition technique to fabricate nano CuO/ZnO with 90%:10% thin film on a copper substrate according to the a previous study [5]. A simple sol-gel method was used to deposit nano CuO/ZnO films [6].

The film structure was analyzed by XRD, and the analysis of the surface morphology and film composition was made through (SEM) and (EDXS). The effects of gamma radiation on nano CuO/ZnO thin films were investigated by measuring and evaluating the changes in their current-voltage (I-V) characteristics after exposure to various radiation doses. The sensing properties of the nano CuO/ZnO thin film were then characterized as shown in the following sections.

2. Materials and Methods

2.1 Sol-gel method

Sol-gel method is a wet process used to deposit materials from a stable solution (sol) into a linked network (gel) after hydrolysis or condensation, and finally, the metal oxide particles can be obtained upon the removal of the solvent. The sol-gel process is used for better control of all the reactions involved in solid synthesis. It is simple to create homogeneous multi-component systems, and molecular precursor solutions can be combined to create homogeneously mixed oxides [6]. Metal oxides or metal precursors in solution undergo thermal degradation, hydrolysis, and condensation. The result is the formation of a stable solution, known as the sol. Hydrolysis or condensation reactions increase the viscosity of the precursor solution, forming a gel. The particle size can be controlled by adjusting the precursor concentration, temperature, and pH. A maturing stage is required to promote the growth of solid mass. By detaching the unstable reagents NP are created [6]. **Fig. 1.** illustrates the sol-gel method.



Fig. 1. Sol-gel method diagram.

2.2 Raw materials

Copper nitrate trihydrate (Sigma aldrech, 97%), Zinc Nitrate (Techno Pharmchem, India, 94.9%) salts, Ethanol (Sigma aldrich, Germany) were used in this research.

The used substrate was copper (purity 98%), a plate of thickness 0.2 cm, then the plate was divided into small pieces (1.5cm x 1.5cm).

2.3 Synthesis of CuO-ZnO nanocomposite

In this research, a GR sensor was fabricated using the sol-gel dip coating method to deposit a thin film of CuO:ZnO 90%:10% on a copper substrate. The mixture of the metal oxides CuO/ZnO 90%:10% was chosen based on our previous study [5].

The coating process involved the following procedures as shown in Fig. 2.



Fig. 2. Nano CuO/ZnO on Cu substrate sensor preparation process diagram.

Copper nitrate trihydrate (12.08 g, fine grad Cu(NO₃)₂. $3H_2O$, MW= 241.60 g/mol) were dissolved in 30% ethanol (30 ml, C₂H₆O, MW= 46.07 + 70 ml deionized water), Zinc nitrate (9.47g, fine grad Zn(NO₃)₂, MW= 189.4gm/mol) were dissolved in 30% ethanol (30 ml, C₂H₆O, MW= 46.07 + 70 ml deionized water), then 90 ml from copper nitrate solution were mixed well with 10ml from zinc nitrate solution on a magnetic stirrer. After stirring for 1 hour, sodium hydroxide solution was dropwise added to the mixture until the sol-gel completely precipitated, indicating the formation of the metal oxide network structure. The gel was overloaded on a copper plate and then heated in oven at 800 °C for 1 hr.

The coating process of fabrication of CuO/ZnO on Cu substrate sensor involved the following procedures in details in Fig. 2.

The substrates were polished using emery paper and washed with acetone. Copper nitrate was dissolved in ethanol using a magnetic stirrer while heated at 80°C for 1 hr to form the first precursor solution. Zinc nitrate was dissolved in ethanol using a magnetic stirrer while heated at 80°C for 1 hr to form the second precursor solution.

The use of a magnetic stirrer while heating at 80°C is essential for the successful synthesis of nanoparticles by the sol-gel method. Stirring ensures homogeneous mixing, prevents sedimentation, and enhances reaction kinetics, while heating promotes gelation, evaporation of solvents, and crystallization of nanoparticles. These factors contribute to the formation of uniform, well-dispersed, and high-quality nanoparticles.[6]

Then 90 ml from the first precursor solution was mixed with 10 ml from the second precursor solution (to form CuO:ZnO 90%:10%). The Cu substrates were dipped inside the solution. Then Sodium hydroxide solution was drop wise added to the mixture and then stirred well with a magnetic stirrer while heated at 80 °C for 1 hr.

The solution was left for 1 day to precipitate on the Cu substrates. The Cu substrates were coated with the precipitate and the produced gel of the solution. This indicated the formation of the

metal oxide network structure. Then the coated substrates were withdrawn and air dried for 2-3 hours (until dried totally). The dried-coated substrates were placed in a ceramic container and maintained at room temperature inside the muffle furnace (Model VULCAN A-550 NEY lab furnace max temperature 1100°C).

To enhance the adhesion of nano-CuO/ZnO on the Cu substrates, sintering was conducted in the muffle furnace in eight stages with the sintering ratw of 50 °C/min. The furnace was programmed to reach 1/8 of the desired temperature (100 °C for a target temperature of 800 °C). Once this temperature was reached, the furnace was set to 2/8 of the desired temperature (200 °C), and so on, until the target temperature was reached. The furnace was then gradually cooled down wat cooling rate 25 °C/min after one hr of maintaining the sensor at the target temperature. After one day, the furnace's temperature returned to the surrounding air's temperature and the coated substrates were removed from the furnace. The thickness of the thin film is about 0.2 mm. The substrates were coated with silver paste with about 0.1 mm thickness using a small brush and was left to dry for 20 min to serve as electrodes for electrical study.

2.4 Characterization methods

The samples were investigated for their crystalline structure, surface morphology and optical properties. The XRD patterns were measured on an XRD (Philips Model PW 1800, 40KV, 40 mA, 100000 count/sec, Eindhoven, Netherlands) A SEM was used to observe the surface morphology of CuO/ZnO NPs. In this research, SEM (FEI Quanta 250 FEG, Magnification: Up to 3,000,000x, Resolution: 0.8 nm at 15 kV, 1.2 nm at 30 kV, Romania) TM was used to characterize the microstructure morphologies and crystal structures of the as-synthesized CuO/ZnO NCs. EDXS technique was used to investigate the ratio of Cu and Zn elements in the specimen. The element distribution of the as-synthesized NCs was analyzed using (Zeiss Smart EDX, Resolution: 125 eV at 5.9 keV, Beam size 100 µm, Germany) TM EDXS mapping analysis.

2.5 Gamma irradiation of the films

The exposure of NMs to GR has a great effect on their optical, electrical and physical properties [7]. In this research, the CuO/ZnO films were exposed to low GR using ¹³⁷Cs in a research Gamma irradiator unit of Cs-137 at National Center for Radiation Research & Technology (NCRRT), Egyptian Atomic Energy Authority, Nasr City, Cairo, Egypt. The radiation source was Gamma Cell-40 (Cesium 137), which allowed for a homogeneous irradiation distribution. The dose rate during the experimental phases was 0.33 Gy/minute. The irradiation was made to investigate the effect of GR on its electrical properties. Four identical samples were irradiated; the first sample was irradiated at dose equal to 1Gy which was irradiated for 3min in the unit, the second sample was irradiated at dose equal to 3Gy which was irradiated for (3x3=9 min), the third sample was irradiated at dose equal to 6Gy (3x6=18 min). The last sample was irradiated at successive doses of 1 Gy (3 min) then 2 Gy (6 min), and finally, 3 Gy (9 min), with I-V characteristics measured after each irradiation.

2.6 Electrical measurements

The I-V graphs for each film exposed to a certain GR dose were examined. Those graphs were used to determine current intensity and sensitivity.

3. RESULTS AND DISCUSSION

3.1 Characterization of CuO/ZnO thin film

3.1.1 XRD analysis study

The XRD analysis analysis was performed to identify the phases and confirm the crystallinity of the synthesized CuO, ZnO, and CuO-ZnO NPs. The resulting diffractograms are presented in **Fig. 3**.

The XRD pattern of the CuO-ZnO composite in **Fig. 3** shows characteristic ZnO peaks at $2\theta^{\circ} = 32.65^{\circ}$, 35.42° , and 36.38° . Other ZnO diffraction peaks are slightly shifted from 56.1° and 62.89° to 58.17° and 61.45° , with the peak at 35.42° having the highest intensity. The ZnO peaks in **Fig.3** are the same as those reported in the literature [JCPDS (36.1451)], confirming the crystalline structure of ZnO, which suggests the successful preparation of the ZnO in the sensor composition. Moreover, **Fig. 3** shows the characteristic peaks of CuO at $2\theta^{\circ} = 38.5^{\circ}$, 48.5° , 61.5° , 66.5° , and 68° . The CuO/ZnO NCs diffraction peaks in **Fig. 3** indicate good crystallinity, reflecting the successful integration of the two MO NPs. All diffraction peaks match those reported for CuO in the literature (JCPDS, file No. 01-080-1916). XRD analysis also proved the presence of the Cu metal with sharp and high intensities peaks at 43.5° , 50.5° and 74.5° [8].



Fig. 3. XRD spectra of the prepared thin film sensor Nano ZnO/CuO on Cu metal substrate.

3.1.2 SEM analysis study

Microscopic investigation is an effective technique for determining the nature of a NM, since the morphological characteristics (size, shape, and roughness) of NPs are significant parameters to influence their applications [9].

A SEM is an electron microscope that can produce high-resolution images of surfaces by using a narrow beam of electrons that scans the sample's surface.

SEM micrographs of the CuO/ZnO NC at different magnifications are shown in **Fig 4.** These micrographs reveal the surface morphology of the prepared thin film sensor, as well as the size of the ZnO/CuO NPs, which ranges from 80 nm to 110 nm. The micrographs also clearly show that the Cu and Zn elements are uniformly distributed, confirming that CuO and ZnO are well-distributed in the NCs. This will be advantageous for the transfer of charged carriers between ZnO and CuO. Additionally, obtained micrographs of the CuO/ZnO NC showed the coarsely and porosity morphology surface of the CuO doped with ZnO, which covered the copper substrate.



Mag = 20.00 K X

Mag = 25.00 K X

Fig. 4. SEM micrograph images illustrate the morphological features of the prepared CuO/ZnO sensor showing NPs at different magnifications.

3.1.3 EDXS analysis study

The EDXS analysis of CuO/ZnO NPs was performed to investigate their morphological composition and confirm their purity.. The resulting spectra are shown in **Fig. 5(a-d)** Copper, Oxygen, and Zinc were detected in the CuO/ZnO EDX spectra. The Cl peak that was noticed in the EDX spectra may be caused by the water used in the preparation process. The presence of an oxygen peak in the EDX spectrum provides further evidence that the copper and zinc were present in their oxidized form [8]. The purity of the prepared NMs is evident from the absence of impurity peaks in the EDX spectra. To confirm the elemental composition and homogeneity of the sensor, EDX spectra were acquired at multiple spots on the prepared nano CuO/ZnO sensor on a Cu substrate. The EDX spectra presented in **Figures 5a**, **5b**, **5c**, **and 5d** reveal the presence of Cu, Zn, and O elements, with weight percentage; Cu = 73.13 %; O = 24.43 %; Zn = 2.44 %. It also indicated that copper content was high compared to the other elements due to its percentage in the composition (CuO:ZnO is 90%:10%) and because of the copper substrate. **Fig. 5(a-d)** (inset) shows

the images of the prepared sensor at different spots and the prepared sensor's elemental analysis demonstrated good agreement with the XRD data.



Fig. 5d.

Figs. 5a-5d. EDXS showing composition analysis of the prepared nano CuO/ZnO sensor on Cu substrate; (Inset): shows the images of the prepared sensor at different spots.

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 Table 1 provides a summary of the weight percentage for each elemental component of the NPs and their atomic percentage.

Element	Weight %	Atomic %	Error %
O K	24.43	56.24	10.01
Zn K	2.44	2.74	8.33
Cu K	73.13	41.02	1.56

Table 1. The weight percentage and atomic percentage of the different elements.

3.1.4 Current–Voltage Characteristics for CuO/ZnO Films

In this research, the constant voltage method was used as depicited in Fig. 6.



Fig. 6. I-V characteristics investigation (the constant voltage method's fundamental setup).

In this measurement setup, the sensor, the electrometer, and a constant voltage source, V, were connected in series. Due to the electrometer's negligible voltage drop, nearly all of the voltage is applied across the thin-film sensor with two electrodes. An electrometer was used to gauge the resulting current [10]. Three identical films were used; the first sample was irradiated at dose equal to 3Gy, the second sample was irradiated at dose equal to 6Gy and the third one was irradiated at several successive doses, namely, 1 Gy followed by 2 Gy followed by 3 Gy. **Fig. 6.** depicts the constant voltage method's fundamental setup.

The I-V characteristics were recorded for each film without Gamma irradiation, and after being exposed to the above GR doses. **Figs. 7(a-c)** show the plots of the I-V characteristics for CuO/ZnO films as-deposited and gamma irradiated certain gamma doses.



Fig. 7c. (at 1 Gy followed by 2 Gy followed by 3 Gy).

Fig. 7a, 7b and 7c I–V Plots for CuO/ZnO as-deposited and gamma irradiated CuO/ZnO films.

Fig. 7a, 7b and 7c. showed the following results:

- As the voltage increases, the current increases as well (Direct relationship between voltage and current). As the radiation dose increases, the amount of energy deposited increases too and hence the number of free electrons causes the current to increase depending on the characteristics of both: radiation and dosimeter. So, nano CuO/ZnO thin film could be considered as effective materials for electrical conductivity and dosimetry.

The measured current increases as the irradiation dose increases at constant voltage.

- Note that the measured current of the film irradiated at a dose equal 3Gy (fig. 7a) is equivalent to the measured current of the film irradiated at a dose equal to 1 Gy followed by 2 Gy then measured (1Gy + 2Gy = 3Gy) shown in **Fig. 7c.** and the measured current of the film irradiated at a dose equal 6 Gy (fig. 7b) is equivalent to the measured current of the film irradiated at a dose equal to 1 Gy followed by 2 Gy followed by 3 Gy then measured (1Gy+ 2Gy + 3Gy = 6Gy) **Fig. 7c,** which means that the film accumulate the charges resulting from the interaction of GR with the nanomaterial that constituents the film, hence the measured dose represents the accumulated dose received through the successive irradiation processes. So the prepared nano film can be exploited as Gamma dosimeter. Gamma dosimeter can store the number of gamma rays that it has detected over time. This allows the dosimeter to measure the total dose of gamma radiation that a person has been exposed to, even if the person has been in and out of the areas with high radiation levels.

3.1.5 Dose-Current Relationship

It's clear from the above results that, when applying certain voltage to the irradiated dosimeter, the resultant current depends on the dose received by the dosimeter. Then calibration of the dosimeter to develop dose-current relationship which can be used to estimate the real dose. From **Fig. 7c**, it is clear that the curves are well distinguished at 0.5 volt. At this voltage, the following table was developed:

Radiation Dose (Gy)	Resultant Current (A)
0	8.540E-06
1	9.999E-06
2	1.150E-05
3	1.370E-05

Table 3. The values of Radiation Dose and Resultant Current at 0.5 V

From **Table 2.** the relationship between the values of the radiation dose received by the sensor and the resultant current when applying voltage equals 0.5 V can be represented by **Fig.8**.



Fig. 8. relationship between the values of the radiation dose received by the sensor (dosimeter) and the resultant current when applying voltage equals 0.5 V.

From **Fig. 8**, the mathematical relation between the values of the radiation dose received by the sensor (dosimeter) and the resultant current when applying voltage equal 0.5 V using curve fitting using Excel can be given by:

$$D = -1E + 16 I^3 + 3E + 11 I^2 - 2E + 06 I + 3.2451$$

where;

I represents the Resultant Current (A) when applying voltage equal 0.5 V **D** represents the Radiation Dose (Gy) received by the dosimeter

This formula can be used to estimate the received dose by the dosimeter. This is an empirical equation whose free parameters is specified by a set of experimental data to obtain this formula for predicting dose as a function of resulting current at a certain voltage.

Conclusions

A very promising area of research for GR sensing is nano MO films. In this research, a nano thin film of CuO/ZnO with CuO:ZnO 90%:10% ratio on a Cu substrate was prepared for the purpose of GR sensing by a simple sol-gel method. XRD, SEM and EDXS were used to characterize the prepared film. XRD analysis confirmed the phase identification and crystallinity of the obtained CuO, ZnO, and CuO/ZnO NPs. The XRD peaks were in total agreement with the standard values of the Joint Committee on Powder Diffraction Standards (JCPDS) data, which proved the crystalline structure of the NPs and suggested the successful preparation of the oxide NPs in the sensor composition.. Also, the elemental analysis using SEM and EDXS of the prepared sensor shows good agreement with the results of XRD.

Two electrodes were made using silver paste for the I-V characterization. The prepared samples demonstrated excellent I-V characteristics dependency at low Gamma ray doses up to 6Gy,

so thin film nano CuO/ZnO can be considered as effective materials for electrical conductivity and dosimetry. The measured current increases as the irradiation dose increases. The film accumulated the charges resulting from the interaction of GR with the NM that constitutes the film, hence the measured dose represented the accumulated dose received through the successive irradiation processes. So the prepared nano film can be exploited as Gamma ray dosimeter. A dose -current relationship was made at 0.5 V, which was used to make the equation to calculate the received dose.

Considering all the previous results and the other advantages of film oxide, such as its simplicity and low cost, show that the prepared film has promising properties for use in GR dosimetry applications.

Abbreviations

GR	Gamma Radiation	
NT	Nano Technology	
NSMs	Nano Scale Materials	
NMs	Nano Materials	
NPs	Nano Particles	
XRD	X-Ray Diffraction	
SEM	Scanning Electron Microscope	
EDXS	Energy Dispersive X-ray Spectroscopy	
I-V	Current- Voltage	
I-D	Current-Dose	

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