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RESEARCH ARTICLE

H_2 Gas Sensing of α-Fe_2O_3/MgO

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ABSTRACT

In this study, MgO (F1) thin films were grown using pneumatic spray pyrolysis and MgOα-Fe_2O_3 (F2) thin films were grown using magnetron sputter. ($Mg(NO_3)_2.6H_2O$)+($(CH_2)_6N_4$) was used as a precursor. The crystal structure and surface morphology of the prepared films were analyzed. XRD spectra show a tendency to grow towards (220) in both films. The intensity of the main peak decreases in the second film due to Fe_2O_3. Additionally, growth was also observed in the (312) plane in the second film. As can be seen from the SEM images, when α-Fe_2O_3 is coated, it was observed that the surface morphology did not change significantly and nanocubes are formed. In the sensor tests performed, while no response was obtained in the F1 film, the sensor response of the F2 film was obtained. It revealed that α-Fe_2O_3 film could improve the sensor performance of MgO films.

Contents

1. Introduction

The importance of nanotechnology in the field of materials is increasing day by day. This is because Nanostructured materials have unique properties such as high mechanical stability, high strength and excellent thermal conductivity. However, preparing nanostructures suitable for various applications is a great challenge. The size and shape properties of a material, which are direct influencing factors, can be tuned through preparation techniques and engineering experimental parameters in the synthesis of nanostructured materials. Another way to tune properties is through the use of composite materials. In general, composite materials consist of two or more different materials that enhance the properties of each material. The construction of multifunctional nanomaterials that combine two or more functions in a single geometry has been achieved by surface coating technique, creating a new class of materials called surface-decorated nanostructures. Surface-decorated nanostructures have been reported for various applications such as sensors, solar cells, light-emitting diodes in photodetectors [1].

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Various metal oxide nanostructures such as Fe_3O_4, ZnO, TiO_2, MgO and $CoFe_2O_4$ have been synthesized for wide applications. Magnesium oxide thin films attract great scientific and technological interest due to their important application properties. Biocompatibility, low cost and non-toxicity were determined as the reasons for choosing MgO materials. MgO is a well-known photocatalyst with unique chemical, mechanical, optical and electrical properties. Magnesium oxide (MgO) has been used as a chemical sensor due to its catalytic property as well as the large mobility of electrons. High surface reactivity, wide band gap, chemical and thermal stability are other properties it has. However, due to its wide band gap, the dominant light absorption of MgO is in the UV range. In the band gap of MgO, transition metal ions can form intermediate energy states. This causes the band gap to narrow. MgO is an inorganic material with a direct band gap. Excellent transparency and biodegradability have increased research interest in MgO thin films for environmentally friendly device applications, making it suitable for many industrial, medical and scientific practical applications. Moreover, the ease of preparation of the compound is facilitated by the high oxygen affinity and low melting temperature of magnesium [1–4].

In a similar study, MgO nanocubes were prepared by chemical vapor deposition methods, and then the decoration or formation of the shell layer on the surface of MgO nanocubes was optimized by the change of Zn precursor during the drop coating process. To study the H2 sensing response, the flexibility of the prepared sensor was examined by changing the bending angle of the substrate. From these analyses, it was revealed that the ZnO-decorated MgO nanocubes prepared were quite suitable for flexible hydrogen sensor. In the past years, various surface-decorated nanostructures have been reported, such as surface-decorated nanoparticle structures, surface-decorated nanowires, surface-decorated nanocubes, metal-decorated graphene, and surface-decorated carbon nanotubes. Hydrogen leak detection is most important as it is odorless, light, tasteless, colorless, flammable and highly explosive in the concentration range of 4 to 75% by volume. In case of leakage during the production, storage and transportation of hydrogen gas, it can lead to disastrous consequences such as accidental explosion. Hydrogen safety is one of the most important safety indicators in fuel cell vehicles (unlike other types of alternative energy vehicles). Catalyst, thermal conductivity, electrochemical, resistance based, work function based, mechanical and optical methods etc. are used to detect hydrogen. Different detection technologies such as have been used. And among these, gas sensors show promising outputs. The novelty of the current work is the use of MgO together with ZnO and its application as a flexible gas sensor [1].

MgO/ZnO nanocomposite has been reported to be potentially used for various applications such as photocatalysis, biomedicine, thermal management applications, solar cells, and biosensors. For electrochemical sensing applications, modification of the sensor surface using metal oxide nanocomposite plays an important role in charge transfer and increases the sensitivity of the modified sensor for analyte detection. There are no research studies yet focusing on the use of MgO/ZnO nanocomposite in the production of modified potentiometric coated wire sensors. In polymeric membrane-covered wire sensors, the metal wire substrate used is highly conductive and the sensing surface is coated with the membrane mixture of the selected analyte. These sensors are very easy to construct, give an excellent dynamic potential response, are generally stable, and show higher selectivity than other conventional types of the same analyte [5]. In other similar work, due to its unique properties, layered structured Clay was used in sensor preparation to improve the properties. Clay-modified electrodes are widely used in voltammetry studies on metals and bioactive compounds in water [4].

Nanocrystalline metal oxides have high reactive edges and surface-to-volume ratios. However, unusual lattice planes have made these materials interesting. In addition, they are among the materials of interest due to the presence of corner defect points. The reasons why researchers are particularly investigating magnesium oxide (MgO) are as follows: nano-sized alkaline earth metal oxides have high surface reactivity and adsorption capacity compared to their counterparts. It has been observed that when different types of Fe_2O_3/MgO nanomaterial were prepared by various methods, nanorods with high surface area were obtained [6]. The catalytic efficiency of MgO/Fe_2O_3 composites revealed their effectiveness. Therefore, MgO/Fe_2O_3 composites can be used for various industrial applications [7]. The first benefit of the α-Fe_2O_3 and MgO compatibility is to improve the properties of the single oxide in the α-Fe_2O_3/MgO composite, including increased surface area and decreased photoinduced electron-hole pair recombination rate. The presence of MgO improves the photocatalytic activity of the α-Fe_2O_3/MgO nanocomposite by increasing light absorption [8].

Spray pyrolysis (SP) using cheap and safe solvents such as water and alcohol is promising for new applications. SP is preferred in studies for thin film growth since it is a cost-effective and efficient method to obtain CZTSSe based solar cells [9]. Additionally, SP is a suitable technique for producing a variety of materials, including thin films with large surface areas at relatively low temperatures. Studies have shown that thin ferrite films with similar or better properties to films produced by other methods are obtained by spray pyrolysis deposition of mixed metal citric acid complexes in ethylene glycol solutions [10].

The magnetron sputtering technique is preferred because of its low cost, high-speed deposition, dense film build and excellent film-substrate adhesion, and the ability to deposit thin films on a variety of substrates for industrial applications. The surface morphology of Mg-doped Fe_2O_3 thin films grown by magnetron sputtering to create inexpensive H2 sensor designs plays an important role in improving the sensitivity of gas sensors [11].

2. Material and Method

2.1. Pneumatic Spray

For MgO (F1) film growth purposes the glasses selected as substrates were placed in the heater. When the substrate temperature is 450°C, the distance between the nozzle and heater is 55 cm and the angle is 45°. 0.1 M and 100 ml solution was prepared by using $Mg(NO_3)_2$ and $(CH_2)_6N_4$ salts in the ratio of 1:0.25, respectively. Sigma-Aldrich company was preferred. The amount of solution applied to the substrates is 50 ml and the procedure time is 40 minutes. The first film grown on the substrate, F1, was grown with pneumatic spray. The relevant parameters are given in Table 1.

2.2. Magnetron Sputter

For α-Fe_2O_3 (F2) film growth purposes in the current research, α-Fe_2O_3 thin films were deposited on glass substrates at 450°C substrate temperature using the DC magnetron sputtering technique. A gun with a mirrored field configuration was equipped with 2-inch Fe (99.99 % purity) sputtering targets. DC sputtering voltage applied to the Fe target was varied as 150 W. The distance between the target and substrate was 57 mm from both targets in all cases. The substrate plate was located at the top of the sputtering chamber. Substrate rotation was applied during depositions. The substrate rotation speed was 3 rpm.

For all depositions, the sputtering chamber was evacuated to 4×10^{-6} Torr by a mechanical pump and turbo molecular pump and then filled with high purity (99.99 %) Ar gas. Ar and O_2 flows were maintained inside the chamber during depositions with a flow rate of 43 sccm and 2 sccm, respectively. Chamber pressure was sustained to be 8.3×10^{-3} Torr. 40 min were spent on each deposition procedure. The F2 film grown on the F1 film was grown with magnetron sputter. The relevant parameters are given in Table 2.

Table 1 Pneumatic Spray parameters

Substrate	Thin films	Salts for precursors solutions (in deionized water)	Molar ratio of precursors solutions	Substrate Temperature (^{0}C)	Carrier gas	Film Growth Process time (min)
Glass	MgO (F1)	$(Mg(NO_3)_2.6H_2O)+(CH_2)_6N_4)$	1:0.25	450	air	40

Table 2 Magnetron Sputter parameters

Substrate	Thin films	Target	Gas ratio	Substrate Temperature (^{0}C)	Carrier gas	Film Growth Process time (min)
MgO (F1)	α-Fe_2O_3(F2)	Fe	43 sscm Ar 2 sscm O_2	450	Ar	40

2.3. Sensor Measurement

Gas sensor tests were carried out with a homemade system with a volume of 0.5 lt. All measurements for a concentration of 1000 ppm were carried out at 300 °C by applying dry air and H_2 gas. The applied voltage value is 0.5 V.

3. Results

3.1. Crystal structure

XRD results showed that F1 and F2 thin films were formed in the polycrystal. The peaks observed in Figure 1 are confirmed by the JCPDS card number "00-027-0759". Only the (111) plane belongs to the JCPDS card number "00-076-1363". The main peak of F1 is the (220) plane. The other peaks of F1 are (111), (413) and (443) planes in Figure 1(a). The peaks of F2 are (220) and (312) planes as shown in Figure 1(b).

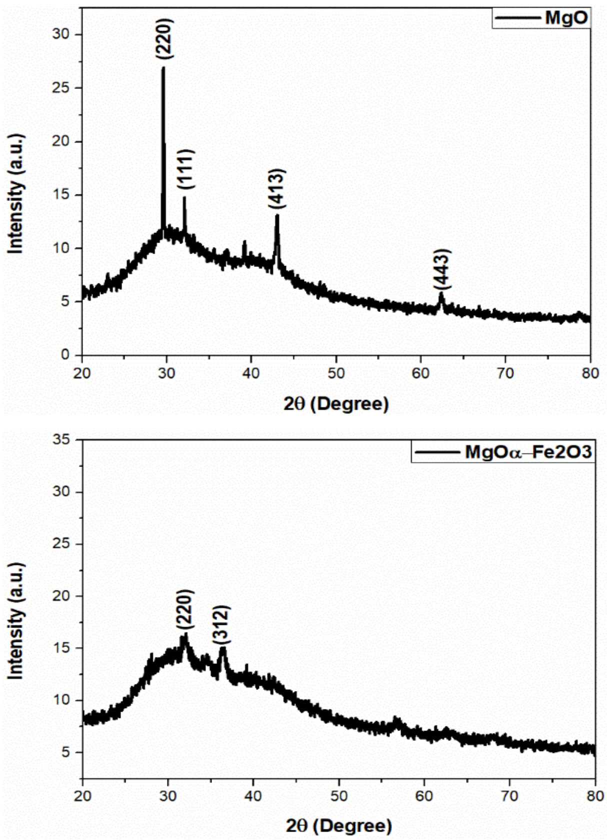

Figure 1 XRD results of a) MgO (F1) and b) MgOα-Fe_2O_3 (F2)

3.2. Surface morphology

SEM images of the F1 and F2 are shown in Figure 2 at x20,000-10,000 magnifications. As can be seen from the figure, the surfaces of the samples showed a very homogeneous growth. The growing nanostructures appear to be nanocubes with a similar structure.

a

b

c

d

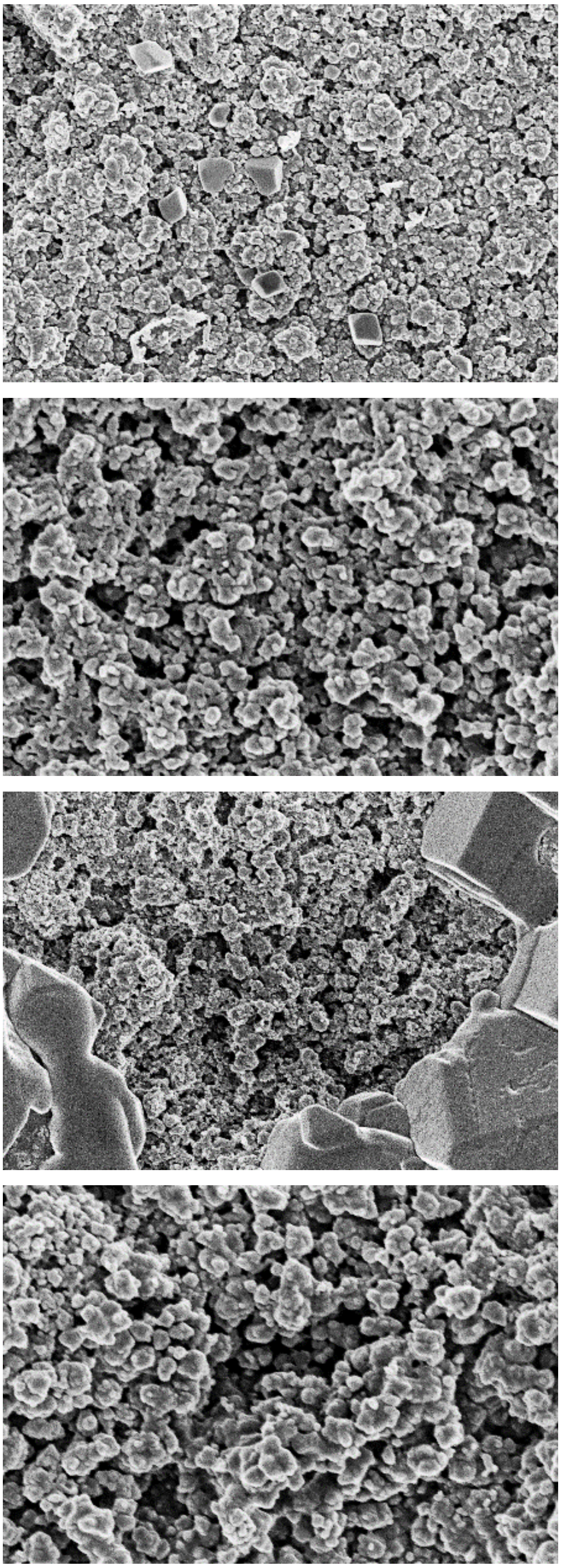

Figure 2 SEM graphs of a-b) MgO (F1) and c-d) MgOα-Fe_2O_3 (F2)

3.3. Sensor test

For gas sensor measurements, comb-shaped interdigitate electrodes (IDT) were fabricated by Al metal sputtering. The IDT contact distance between electrodes is 400 µm and the total area of the device is 1cm x 1cm. The sensor electrode metal was contacted on F1 thin films grown with Al pneumatic spray and F2 thin films grown with magnetron sputter. Hydrogen gas sensor testing was performed for 1000 ppm at 300 °C. Sensor responses are presented in Figure 3. No sensor response was received from the F1 film, but the sensor response of the F2 film is clearly seen in the graph. Dry air was applied to the film as sweeping gas and then H_2 gas was applied as detection gas. Measurements for both F1 and F2 samples were performed for 3 cycles. While dry air was applied for the first half cycle, H_2 sensing gas was applied for the second half cycle. Each cycle measurement time was 600 seconds. It is seen that the current value increases during H_2 gas measurements, and dry air brings the sensor to the initial current value.

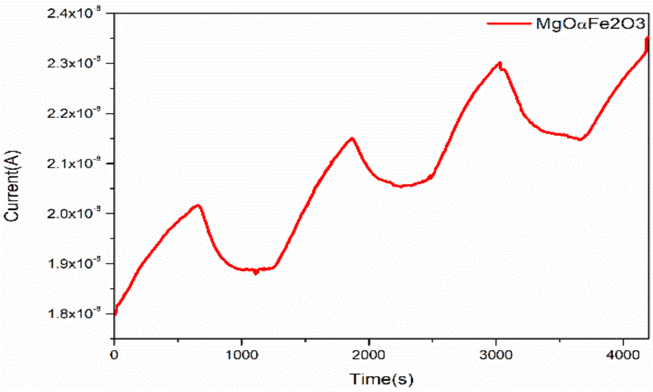

Figure 3 Sensor performance of MgOα-Fe_2O_3 (F2)

4. Conclusion

In the study, MgO (F1) film was first grown using the pneumatic spray (PS) method. Afterwards, α-Fe_2O_3 (F2) film was grown on the MgO film in order to increase the sensor performance. The crystal and morphological properties of the grown thin films (F1, F2) were examined. The effect of α-Fe_2O_3 coated with magnetron sputter was investigated. In the XRD examinations, the main peak of F1 is the (220) plane and peaks of F2 are the (220) and (312) planes with approximately the same planes. SEM results show that when α-Fe_2O_3 is coated, it was observed that the surface morphology did not change significantly and nanocubes are formed. In the first sensor test, no response was obtained in the F1 sample grown with the pneumatic spray technique. Considering the high band gap of MgO material, electrical conductivity is expected to be low. To eliminate this problem, sensor response was obtained with α-Fe_2O_3 coated using the magnetron sputter technique. The reason for this is that the α-Fe_2O_3 material provides conductivity to the film by reducing the band gap.

Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

References

[1] Pradeep N, Gopal T, Venkatraman U, Alrebdi TA, Pandiaraj S, Alodhayb A, et al. Effect of substrate bending towards chemiresistive based hydrogen gas sensor using ZnO-decorated MgO nanocubes. *Materials Today Chemistry* (2022) **26**:101200.

[2] Nion M, Kabir MH, Mona MA, Haque MJ, Shahariar N, Hafiz M, et al. Effect of Al doping on morphology and optical properties of spray pyrolized MgO thin films. *results in materials* (2021) **12**:100235.

[3] Sağlam HK, İskenderoğlu D, Güldüren ME, Güney H, Şahin B, Ertuğrul M. Analyses of Cu impurity effects on the USP grown MgO thin films: Structural, optical and photocatalytic assessments. *Optical Materials* (2023) **145**:114499.

[4] Mylarappa M, Raghavendra N, Surendra BS, Kumar KS, Kantharjau S. Electrochemical, photocatalytic and sensor studies of clay/MgO nanoparticles. *Applied Surface Science Advances* (2022) **10**:100268.

[5] Al-Tamimi SA. Prospective of modified polymeric MgO/ZnO nanocomposite sensor for potentiometric determination of chronic myelogenous leukemia medication. *Sensors and Actuators A: Physical* (2021) **331**:112949.

[6] Mahmoud HR, El-Molla SA, Saif M. Improvement of physicochemical properties of Fe2O3/MgO nanomaterials by hydrothermal treatment for dye removal from industrial wastewater. *Powder technology* (2013) **249**:225–233.

[7] Utaiyachandran M, Durairaj K, Kumar AJ, Sivakumar S, Palanivel V, Senthilkumar P. Photo-assisted assembly of MgO/Fe2O3 composites for photocatalytic Rhodamine B degradation and their mimicking activity. *Desalination and Water Treatment* (2023) **314**:263–275.

[8] Devi S, Kumar A, Singh S, Kumar V, Kumar S, Kumar R, et al. Enhanced photocatalytic activity of α-Fe2O3/MgO nanocomposites for environmental sustainability. *Ceramics International* (2024) **50**(13):24608–24617.

[9] Espindola-Rodriguez M, Sanchez Y, López-Marino S, Sylla D, Placidi M, Neuschitzer M, et al. Selenization of Cu2ZnSnS4 thin films obtained by pneumatic spray pyrolysis. *Journal of Analytical and Applied Pyrolysis* (2016) **120**:45–51.

[10] Milanova M, Koleva I, Todorovska R, Zaharieva J, Kostadinov M, Todorovsky D. Polymetallic citric complexes as precursors for spray-pyrolysis deposition of thin ferrite films. *Applied surface science* (2011) **257**(17):7821–7826.

[11] Sarıtaş S, Muğlu GM, Turgut E, Kundakçı M, Yıldırım M, Şenay V. Optical, structural, morphological, and gas sensing properties of Mg-doped α-Fe2O3 thin films deposited by RF and DC magnetron Co-sputtering technique. *Physica B: Condensed Matter* (2024) **677**:415704.