

# STUDY OF GAS SENSITIVITY OF (MGO+NI) MEMBRANES PREPARED BY PULSED LASER DEPOSITION

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**ABSTRACT:-** The pure (MgO) membranes mixed with nickel (Ni) were prepared at the proportions of (0,1,3,5,7) using the method of pulsed laser deposition (PLD), as the deposition process was carried out on glass bases at room temperature, with a thickness of nm (200) where the effect of nickel mixing on the structural and optical properties was studied, as the results of X-ray diffraction showed that all the membranes that were prepared as having a cubic structure (Cubic) as well as within the structural characteristics The surface topography was studied with (AFM) and the results showed a decrease in the values of granular size, but through visual examinations, it was observed that the values of absorbency and absorption coefficient increase with increasing mixing ratio, while the optical energy gap decreases with increasing mixing with nickel (7.79-3.43)eV. As for the values of sensitivity to gas, where they were increasing with temperature and the percentage of nickel addition, the highest percentage of sensitivity (42.12%) was obtained at the percentage of nickel addition (7%Ni).

**Keywords:** Thin film, sensitivity, deposition, XRD, AFM, optical properties.

## Introduction

Thin films are very thin layers of various materials such as metals, semiconductors, insulators, and polymers. Their thickness ranges from a few nanometers to only several micrometers[1,2].

Thin films have the following characteristics[3,4]:

1. Ultra-small thickness: As I mentioned, they are very thin, much thinner than a human hair.
2. Large surface area: Because of its small thickness, the surface area-to-volume ratio is very high.
3. Controllability of properties: The chemical and physical properties of thin films can be controlled by the selection of their constituent materials and preparation methods.

4. Multiple applications: Thin films are used in many fields such as electronics, optics, sensors, and functional coatings.

have a broad range of uses in multiple disciplines, including microelectronics, optical fibers, and solar energy. These membranes have peculiar attributes, including a high degree of electrical conductivity, a high degree of optical transparency, and a long lifespan, all of which are singular and Applications Magnesium oxide Microelectronics, Photonics, Solar energy and Sensors[5,6].

## 2. Working method and materials

The substances in Table 1 represent the properties of the substances employed.

Table 1: The specifics of the materials employed

| Material | Pure  | density | Company      |
|----------|-------|---------|--------------|
| MgO      | %99.9 | 4.5     | Industries   |
| Ni       | %99.7 | 7.2     | BHP Billiton |

## 3. Preparation of models

The samples were weighted using a delicate equilibrium of (0.001) of German origin, resulting in a total of 2gm of magnesium oxide and nickel. The proportions of nickel in the overall weight were 0,1,3,5, and 7, with magnesium oxide making up the remaining percentage. The samples were made and their proportions taken, and then they were put in a mold with a diameter of 12 mm and subjected to high pressure using a hydraulic press from Sky Spring (5Ton). To guarantee that consistent,

well-capitalized samples are acquired, the pressure and time are carefully adjusted. The hydraulic piston mechanism is shown in Figure (1).

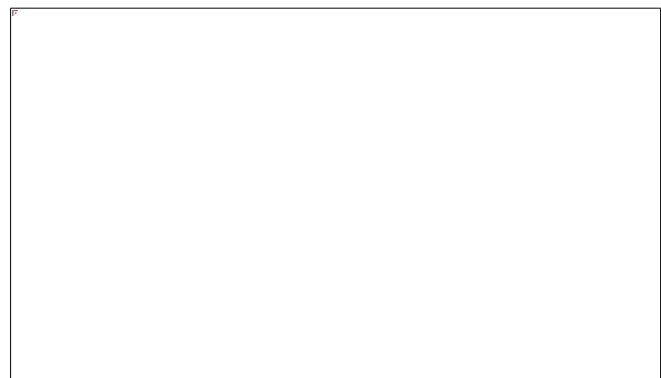


Figure 1: Hydraulic press

## 4. Deposition process

Following the pressing process, the samples are processed to get them ready for membrane formation. A pulsed laser device of the Nidum - Yak type, as seen in Figure (2), was used. Its 500mJ capacity allowed it to heat the sample's surface tightly and create a thin layer of material that was originally from China on the surface of the glass base, which measures (50 \* 20 \* 1.2) To guarantee accurate membrane creation and excellent quality, laser parameters like power, frequency, and duration are carefully regulated during the laser deposition process. Table (2) describe the laser device's specs that are employed in the deposition procedure. study including electronics, solar energy, and optical technologies that need for accurate, superior installations. Nickel-impregnated magnesium oxide samples are prepared to form the membrane and achieve excellent results in technical research and manufacturing applications that require precise, high-quality installations, such as optical technologies, solar panels, and electronic devices, using the sampling and laser staging process.

Table 2: The specifications of the pulsed laser device used in sedimentation are described.

| Properties  | Value        |
|-------------|--------------|
| Rang Energy | (100-1000)mJ |

|            |              |
|------------|--------------|
| wavelength | (532-1064)nm |
| Frequency  | (1-6)HZ      |

5-Structural and visual examinations

5-1 Thickness measurement

Measuring thin film thickness facilitates comprehension of the behavior and physical characteristics of membranes. The performance of related applications can be enhanced and membrane design improved with the usage of this knowledge. The thickness of the prepared membranes was measured by the diffraction method of the laser beam using a helium-ene laser source (He-Ne), lens, and sample. There will be optical interference (dark and luminous lines) after the laser light passes from the lens to the eye, and the thickness is calculated as in equation.(1)

W = Δx λ / x 2 ..... (1)

Whereas:- W is the thickness of the membrane, the path of the laser's deviation is Δx, the interference hem is located at x, and the laser's wavelength is 632.8nm.

5-2 Optical measurements

Optical analyses of the membranes of magnesium oxide that were coated with different percentages of (0,1,3,5,7) of nickel were conducted using ultraviolet light, the spectrometer type SP-8001 and its wavelength between (200-1100) nm were used to measure visual assessments, the sample was highlighted and the specific amount of light that was absorbed was recorded. This measurement can provide information about the degree to which light is absorbed and the alterations to the electronic structure of the material, subsequently, we can calculate the remainder of the visual exam, including transmittance, energy gap and absorption.

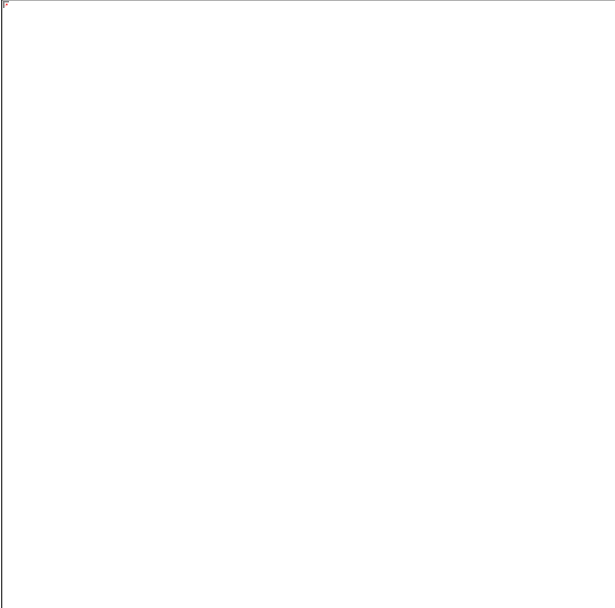


Figure 2: UV spectrophotometer.

6-Synthetic measurements

6-1 X-ray diffraction test X-ray

XRD screening is a powerful analytical technique used to study the crystal structure and chemical composition of solids. This technique is based on the phenomenon of X-ray interference when interacting with crystalline material. When a beam of X-rays falls on a sample of material, these rays propagate in different ways depending on the atomic arrangement and crystal structure of the sample. This contrasting interference of X-rays creates a unique pattern of diffraction, which reflects the properties of the crystalline material. This diffraction pattern

is captured using an X-ray detector, and then analyzed to infer information about the crystal structure and chemical composition of the sample. This information can be used in various fields such as materials science, chemistry, geology and others.

6-2 Atomic Force Microscopy (AFM)

Analyzing and inspecting surfaces at the atomic and particle levels is done through the process of inspection. AFM measures the forces between the sample surface and itself by interacting with it using a sensitive needle. Using a needle to apply a tiny compressive force, a thorough map of the surface is made, and its many characteristics are examined by measuring the change in height and associated forces. The Bruker Dimension Icon Atomic device Force Microscope was used to examine the produced membranes under an atomic force microscope.

7-Results and discussion

7-1 Structural properties

The structural changes in the crystal form of pure magnesium ( MgO) films combined with nickel ( Ni) were studied by X-ray diffraction to determine the changes in crystal structure of the materials, the size of the crystal, the process of uniformizing the constituent parts of the prepared membranes, the study of the topography of the membranes by atomic force microscopy (AFM), the particles' roughness and their size

7-1-1 X-ray diffraction

Has demonstrated the results of an X-ray analysis of pure magnesium membranes (MgO), as the appearance of magnesium oxide is observed in the case of a pure sample and through Figure (3), it is apparent that the X-ray spectrum of (MgO) membranes is of a cubic structure, the pattern of diffraction and the locations of the peaks of the various samples prepared by the pulsed laser method of sedimentation, all of which are observed. As demonstrated in Table (4) and this is associated with the international card (ASTM) (ICDD) and numerical (1197-001-00), but after being mixed with nickel, nickel appeared with three peaks (111), (200) and (220) when augmenting the percentage of nickel in the mixture, the intensity of the peaks increased, and the intensity of the magnesium oxide peaks decreased, according to the international card (00-154-1139), the crystal structure of nickel is cubic in composition, but after being added to magnesium oxide, it has the same crystal structure as the other components (Cubic)[7]. This indicates that the material is beginning to have a more uniform composition and are growing in size more gradually.



This is consistent with the results of the crystal size study by Nagu and his team[8].

Figure (3) X-ray diffraction of pure and nickel-added (MgO) membranes.

7-1-2 Atomic Force Microscope

Through this examination, the topography and topography of the surfaces of the prepared membranes were studied using laser sedimentation technology, as the samples were scanned with dimensions (μm2 2×2), and it is clear through the shapes (4) a, b, c, d, e for three-dimensional and one-dimensional images decreasing in the values of both surface roughness (Surface Roughness) and square root of the square of medium roughness (RMS) with a decrease in the granular size of all prepared membranes when increasing the grafting rates and this indicates the smoothness of the surface and the small granules, as is observed from the shapes (4- a, b, c, d, e) The appearance of granular clusters indicates that nickel atoms (blemish atoms) were formed in the form of separate islands and is consistent with the results of X-ray diffraction obtained earlier., Table (2) shows the values of surface roughness, square of average

roughness (RMS) and grain size calculated using atomic force microscopy (AFM)[9,10

Table 2: AFM Test Results for MgO:Ni Membranes

| Sample   | Average roughness (nm) | r. m. s (nm) | Average diameter (nm) |
|----------|------------------------|--------------|-----------------------|
| MgO pure | 6.894                  | 6.937        | 85.987                |
| 1%Ni     | 6.261                  | 6.536        | 84.341                |
| 3%Ni     | 5.659                  | 5.561        | 81.657                |
| 5%Ni     | 5.193                  | 4.784        | 77.458                |
| 7%Ni     | 4.136                  | 4.256        | 70.395                |

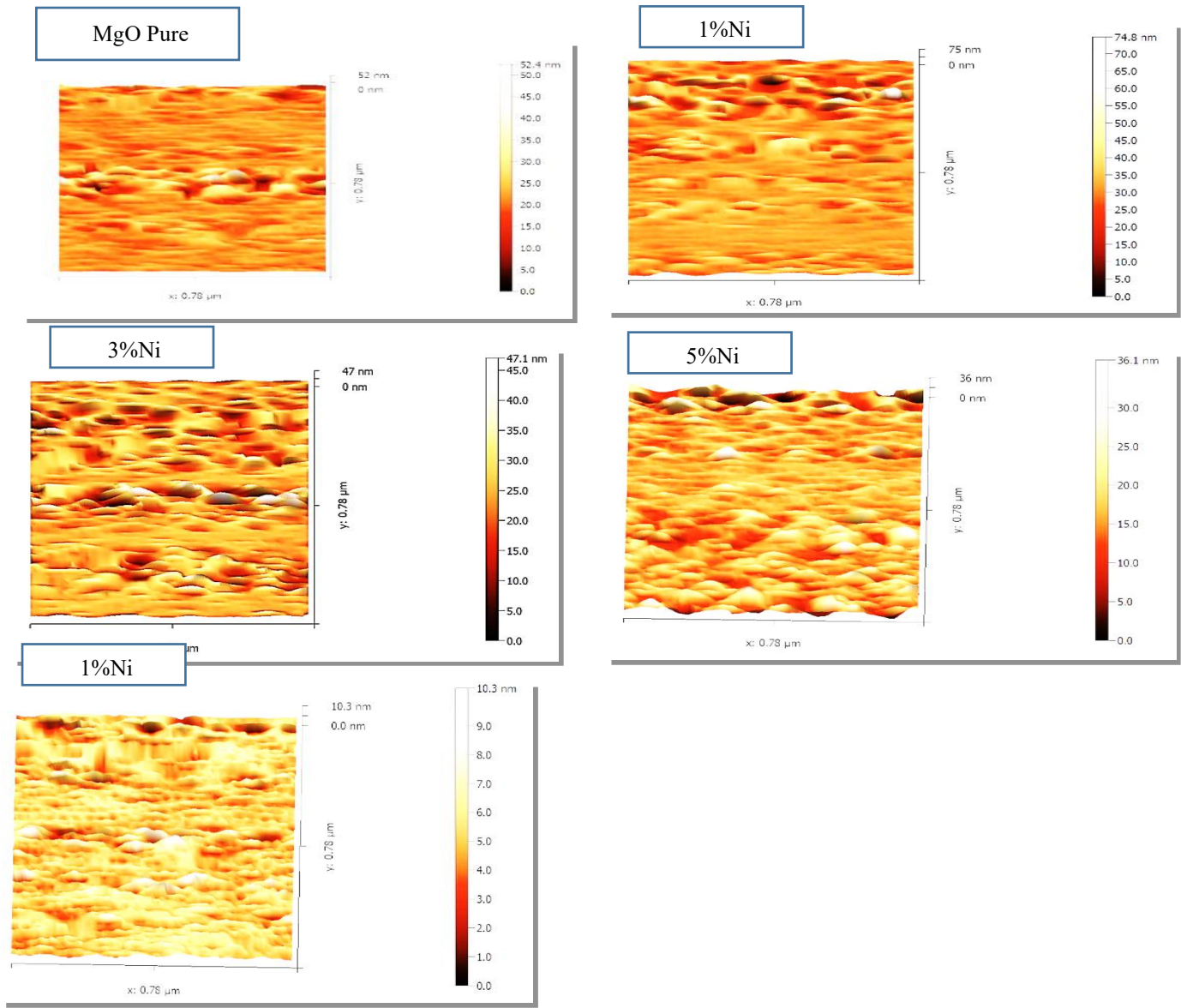


fig (4) Images and results (AFM) for all pure magnesium dioxide membranes and nickel-added.

7-2 Optical Measurements

7-2-1 Absorption

The results of the absorption values of pure magnesium oxide (MgO) films combined with nickel (Ni) that were produced by laser-induced Deposition decreased as the wavelength of the light increased, as demonstrated in Figure (5), the value of absorption is greatest at short wavelengths (300nm) and then the value of absorption decreases as the wavelength increases until the value of absorption is at its lowest value at the wavelength of nm (1100), which indicates that the prepared membranes have a high degree of absorption at the visible area, this property is beneficial to them in regards to electronic applications, such as solar cells, and decreases as the wavelength increases, this is because the energy of the photons that are incident is less than the value of the energy gap of the semiconductor, this prevents the electron from migrating from the valence beam to the conduction beam. The cause of the increase in the absorption values with an increase in nickel concentrations is the generation of defect sites, which leads to an increase in the energy state within the band gap. The elevated concentration of the defect area with a high concentration of Ni causes an increase in absorption[11,12].

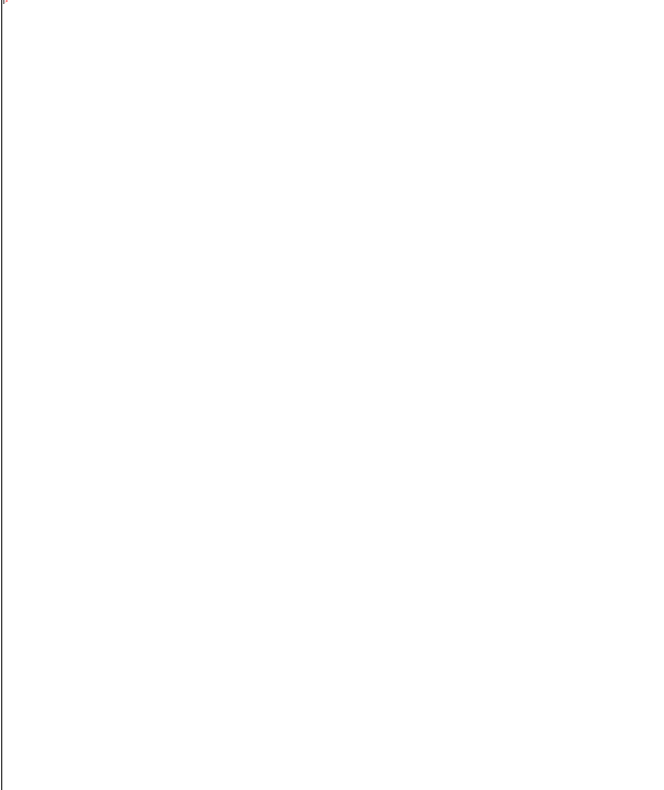


Figure 5: Absorption spectrum of magnesium oxide membranes

7-2-2 Absorption Coefficient ( $\alpha$ )

Figure (6) illustrates the association between the absorption coefficient and the energy of photons in membranes that were prepared by the pure pulsed laser method in a concentration of (0,1,3,5,7). It was discovered that the values of the absorption coefficient of membranes are identical to ( $\alpha$  10,000 <paraphrase> It was found that the values of the absorption coefficient of membranes are equal to ( $\alpha$  10,000 Through Figure (6), it's evident that the process of adding nickel to the mixture increases the values of the absorption coefficient, this means that the membrane's material began to form crystals and become uniform when combined with nickel, and the process decreased the amount of crystalline defects and decreased the local levels of crystallinity in the forbidden energy range, both of which were caused by defects or shortfalls in the structure[13].

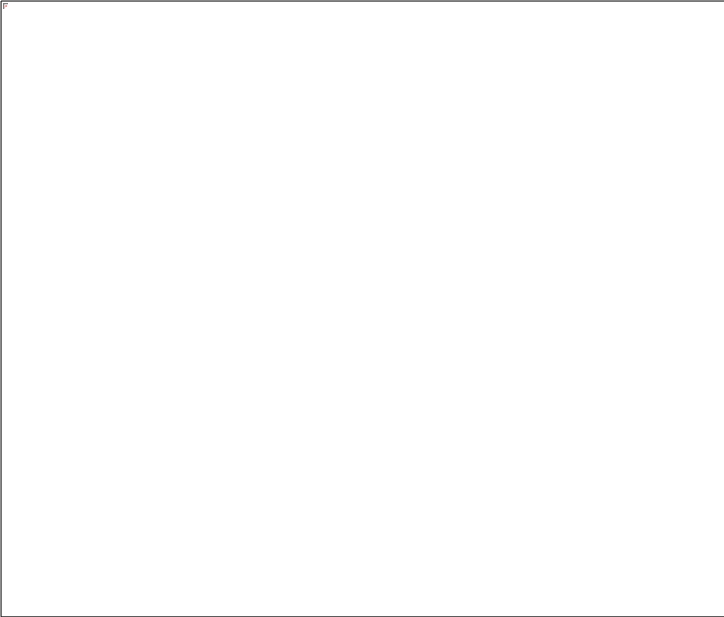


Figure 6: The relationship between the absorption coefficient and photon energy of magnesium oxide membranes.

7-2-3 Transmittance

Figure (7) illustrates the relationship between permeability and wavelength, if the results indicate that the value of permeability decreases as the percentage of nickel in the mixture increases, then the spectrum of permeability is highest for the visible spectrum region, and (7) shows the spectrum of permeability associated with the pure chemical compound of magnesium oxide in the presence of nickel at different concentrations. The figure demonstrates that the transmittance decreases as the mixing rate increases, but it begins to increase at a slow rate and for all of the prepared membranes when the wavelength increases, this is because the spectrum of the prepared membranes is similar to the photodetector's transmittance spectrum, which is why the prepared membranes are permeable to the visible and infrared regions[14].

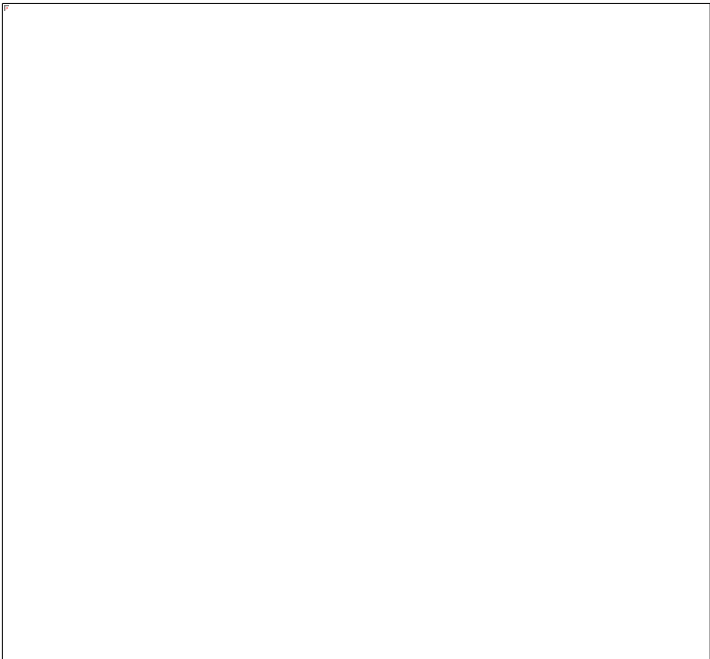


Figure 7: Permeability spectrum of prepared magnesium oxide membranes

### 7-2-4 Energy Gap

The optical energy gap of membranes made from (MgO) and pure chemicals as well as nickel-tinged has been determined. A direct relationship has been observed between  $(\alpha h\nu)^2$  and the energy of the photons that are incident on it ( $h\nu$ ). This figure demonstrates that the change is linear over a range of energy values (the range of the visible region). The value of the energy gap of the pure magnesium oxide

membrane was 7.79 eV, but when combining magnesium oxide with nickel, the energy gap began to decrease, the increase in composition led to a decrease in the value of the energy gap from 6.72 eV for pure membranes to 3.43 eV for the membrane added to it and the decrease was caused by the presence of surface defects at the tail of the absorption spectrum[15].

Figure 8: Energy gap values of prepared magnesium oxide membranes

### 8- Sensitivity

The results of the gas sensor showed that the increase in operating temperature leads to an improvement in the sensitivity of the membranes, it is clear in numbers that the sensitivity of all membranes increases with increasing operating temperature. The reason for this is due to the increased rate of surface interaction of the membrane with the target gas. The maximum peak values are seen at certain temperatures called the optimum temperature and then decrease, the increase and decrease in

sensitivity indicates the phenomenon of gas absorption, the sensitivity increases at operating temperature up to (300) °C, it is clear from Figure (9) the results of gaseous sensitization of different percentages of nickel addition to magnesium oxide, where the maximum sensitivity to the gas was found to be ((NO<sub>2</sub>) to be (42.12%) at (200) °C at the percentage of nickel addition (7%Ni), and the reason for this is due to the improvement And good crystallization[16].

Figure 9: Gas sensitivity to prepared membranes

### 8-Conclusions

Through structural investigations, the addition of nickel led to a decrease in crystal size, as for visual examinations, it led to an increase in absorption, the absorption coefficient, the decrease in permeability, and the decrease in the energy gap, additionally, the method was compared to previous studies that employed the method of laser depositing membrane formation, this method is preferred, and membranes that are formed by it have a lack of impurities during the preparation and sedimentation process.

As for the values of sensitivity to gas, where they were increasing with temperature and the percentage of nickel addition, the highest percentage of sensitivity (42.12%) was obtained at the percentage of nickel addition (7%Ni).

### References

- 1.Kaiser, Norbert. "Review of the fundamentals of thin-film growth." *Applied optics* 41.16 , 3053-3060,(2002).
- 2.Ramesh, Ramaroorthy, and Nicola A. Spaldin. "Multiferroics: progress and prospects in thin films." *Nature materials* 6.1 ,21-29,(2007).
- 3.Setter, N., et al. "Ferroelectric thin films: Review of materials, properties, and applications." *Journal of applied physics* 100.5 (2006).
- 4.Boudiar, Meriem, et al. "Sol-gel derived Zn doped MgO thin films and their waveguides." *Journal of Sol-Gel Science and Technology* , 1-11,(2023).
- 5.M. A. Khan, J. W. Park, and E. Y. Lee, "MgO thin films deposited by pulsed laser deposition: structural, optical, and electrical properties," *Thin Solid Films*, vol. 496, no. 1-2, pp. 152-156, (2006).
- 6.S. X. Wang, Z. M. Zhang, S. H. Ji, L. P. Liu, C. L. Xu, and Z. G. Liu, "Structural and optical properties of MgO thin films prepared by reactive magnetron sputtering," *Materials Letters*, vol. 57, no. 4, pp. 714-717, (2003).
6. J. F. Pierson, "MgO thin films for electronic applications," *Journal of Vacuum Science and Technology B*, vol. 9, no. 4, pp. 2115-2121,( 1991).
7. Nagu, A., et al. "Additive-free vapour-phase hydrogenation of benzonitrile over MgO-supported Ni catalysts." *Research on Chemical Intermediates* 46 , 2669-26819,(2020).
8. Imontasser, A., Parveen, A. Probing the effect of Ni, Co and Fe doping concentrations on the antibacterial behaviors of MgO nanoparticles. *Sci Rep* 12, 7922 (2022).

- 9.Tolstova, Yulia, et al. "Heteroepitaxial growth of Pt and Au thin films on MgO single crystals by bias-assisted sputtering." *Scientific Reports* 6.1 , 23232,(2016).
- 10-Budde, Melanie, et al. "Structural, optical, and electrical properties of unintentionally doped NiO layers grown on MgO by plasma-assisted molecular beam epitaxy." *Journal of Applied Physics* 123.19 (2018).
- 11.Liu, Zhengqing, et al. "Room temperature stable CO x-free H<sub>2</sub> production from methanol with magnesium oxide nanophotocatalysts." *Science Advances* 2.9 (2016): e1501425.
- 12.Anandan, K., D. Siva, and K. Rajesh. "Structural and optical properties of (ZnO/MgO) nanocomposites." *Int. J. Eng. Res. Technol* 7.8 (2018): 493-499.
- 13.Obeid, B. G., A. S. Hameed, and H. H. Alaaraji. "Structural and optical properties of TiO<sub>2</sub>: MgO thin films preparing at 373K." *Digest Journal of Nanomaterials and Biostructures* 12.8 (2017).
- 14.Khalaf, M. K., Al-Kader, D. S. A. & Salh, J. M. Effect of thickness and type of substrate on optical properties of chromium oxide thin film prepared by sputtering magnetron. *IOP Conf. Ser. Mater. Sci. Eng.* 1105, 012065 (2021).
- 15.Almontasser, A., A. Parveen, and A. Azam. "Synthesis, Characterization and antibacterial activity of Magnesium Oxide (MgO) nanoparticles." *IOP Conference Series: Materials Science and Engineering*. Vol. 577. No. 1. IOP Publishing,( 2019).

16-Pradeep, Natarajan, et al. "Magnesium oxide nanocubes deposited on an overhead projector sheet: synthesis and resistivity-based hydrogen sensing capability." *Microchimica Acta* 184 (2017): 3349-3355.(2017).  
المحضرة بالترسيب بالليزر (MgO+Ni) دراسة التحسينية الغازية لأغشية

النبيضي

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الخلاصة:-

( وينسب Ni) النقية والمضاف اليها النيكل ( MgO)حضرت أغشية ( حيث كانت عملية ( PLD) باستخدام طريقة الترسيب بالليزر النبيضي (0,1,3,5,7)% (200) وكذلك تمت دراسة nmالترسيب على قواعد زجاجية بدرجة حرارة الغرفة ، وبسلك تأثير الاضافة بالنيكل على الخصائص التركيبية والبصرية ، إذ بينت نتائج حيود الأشعة كذلك Cubicالسينية أن جميع الأغشية التي تم تحضيرها بأنها ذات تركيب مكعب ( ) وبينت AFMضمن الخصائص التركيبية تم دراسة طوبوغرافية السطح بمجهر القوة الذرية ( قيم الحجم الحبيبي ، أما من خلال الفحوصات البصرية لوحظ أن قيم النتائج انخفاض في الامتصاصية ومعامل الامتصاصية تزداد بزيادة نسبة الخلط بينما فجوة الطاقة البصرية تقل ( أما بالنسبة لقيم الحساسية للغاز حيث كانت تزداد 3.43-7.79 V بزيادة الخلط بالنيكل ( مع درجة الحرارة ونسبة اضافة النيكل فقد تم الحصول على أعلى نسبة حساسية (42.12 % ) (Ni7% عند نسبة اضافة النيكل)

الكلمات المفتاحية

، الخصائص البصرية AFM، XRDالغشاء الرقيق، التحسينية ، الترسيب ،