

## Effect of nickel addition on the structural and optical properties of magnesium oxide (MgO) films prepared by pulsed laser deposition (PLD) method.

Abdul Ghaffar Rajab shater<sup>1</sup>, Abdullah Mahmoud Ali

Department of Physics - University of Tikrit - College of Education for Pure Sciences

Email: abd385696@gmail.com<sup>1</sup>, babdullah.ma1763@tu.edu.iq<sup>2</sup>

### 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 scanning electron microscopy (SEM) 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.

**Keywords:** magnesium oxide, mixing, pulsed laser deposition, optical properties, compositional properties .

## تأثير إضافة النيكل على الخواص التركيبية والبصرية لأغشية أكسيد المغنيسيوم (MgO) المحضرة بطريقة الترسيب بالليزر النبضي (PLD)

عبد الغفار رجب شتر<sup>1</sup> ، عبدالله محمود علي<sup>2</sup>

قسم الفيزياء - جامعة تكريت - كلية التربية للعلوم الصرفة.

Email: abd385696@gmail.com<sup>1</sup>, babdullah.ma1763@tu.edu.iq<sup>2</sup>

### مستخلص:

تم تحضير أغشية (MgO) النقية والمخلوطة بالنيكل (Ni) ونسب (0، 1، 3، 5، 7) باستخدام طريقة الترسيب بالليزر النبضي (PLD)، إذ تمت عملية الترسيب على قواعد زجاجية بدرجة حرارة الغرفة، وبسمك nm (200) حيث تمت دراسة تأثير الخلط بالنيكل على الخصائص التركيبية والبصرية، إذ بينت نتائج حيود الأشعة السينية أن جميع الأغشية التي تم تحضيرها بأنها ذات تركيب مكعبي (Cubic) كذلك ضمن الخصائص التركيبية تم دراسة طبوغرافية السطح بالمجهر الإلكتروني الماسح (SEM) وبينت النتائج انخفاض في قيم الحجم الحبيبي، أما من خلال الفحوصات البصرية لوحظ أن قيم الامتصاصية ومعامل الامتصاصية تزداد بزيادة نسبة الخلط بينما فجوة الطاقة البصرية تقل بزيادة الخلط بالنيكل (7.79-3.43) eV .

**الكلمات المفتاحية:** أكسيد المغنيسيوم ، الخلط ، الترسيب ، الليزر النبضي ، الخصائص البصرية ، الخصائص التركيبية .

## 1- Introduction

Thin films are one of the most important newly innovative technologies in materials science and technology[1].

Thin films have a small thickness ranging from several nanometers (nm) to several micrometers ( $\mu\text{m}$ )[1,2]. Thin films are used in a variety of applications, such as medical devices, thin electronics, renewable energy, precision manufacturing and many other fields[3,4].

### 1-1 Applications Magnesium oxide [5,6]

**Microelectronics:** Magnesium ox-

ide membranes are used as electrical insulating materials in transistors, sensors and integrated circuits. **Photonics:** Magnesium oxide membranes are used as anti-reflective coatings in optical lenses and mirrors.

**Solar energy:** Magnesium oxide membranes are used as insulating layers in solar cells.

**Sensors:** Magnesium oxide membranes are used in biological and chemical sensors.

## 2-Working method and materials

Table 1 illustrate the materials that used and their characteristics .

**Table 1: Specifications of the materials used**

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

## 3-Preparation of models

A sensitive balance of (0.001) of German origin was used to conduct weights for the samples, as a total weight of (2gm) of magnesium oxide and nickel was taken, where the proportions of nickel were (0,1,3,5,7) of the total weight and the rest of the percentage belongs to the base material magnesium oxide. After preparing the

samples and taking the proportions, the hydraulic press device manufactured by Sky Spring was used, the samples are placed in a mold with a diameter of (12mm) and exposed to high pressure by (5Ton). The pressure and time are carefully adjusted to ensure that coherent and homogeneous samples are obtained and Figure (1) shows the hydraulic piston device.



**Figure 1: Hydraulic press**

#### **4-Deposition process**

After the pressing process, the samples are processed to prepare them for membrane formation, a pulsed laser device of the type of laser (Nidenum - Yak) shown in Figure (2) was used, pulsed laser deposition technology was used with a capacity of (500mJ) to heat the surface of the sample tightly and form a thin layer of material on the surface of the glass base with dimensions (50 \* 20 \* 1.2) of Chinese origin. In the laser deposition process, laser parameters such as power, frequency and time

are precisely controlled to ensure accurate membrane formation and high quality. (2) explain the specifications of the laser device used in the deposition process. Using the sampling and laser staging process, nickel-impregnated magnesium oxide samples are prepared to form the membrane and achieve excellent results in manufacturing applications and technical research that require precise, high-quality installations, such as electronic devices, solar panels and optics technologies.

**Table 2: Specifications of pulsed laser device used in sedimentation**

| Properties           | Value           |
|----------------------|-----------------|
| Type                 | Philips PW 1840 |
| Rang Energy of laser | (100-1000)mJ    |
| Wavelength of laser  | (532-1064)nm    |
| Frequency of laser   | (1-6)HZ         |

## 5- Structural and visual examinations

### 5-1 Thickness measurement

Thin film thickness measurement helps to understand the physical properties of membranes and their behavior. This information can be used to improve membrane design and improve the performance of associated applications. The diffraction method of the laser beam was used to measure the thickness of the prepared membranes using a helium-ene laser source (He-Ne), lens and sample, after the laser light passes from the lens to the eye, there will be optical interference (which is dark and luminous lines) and the thickness is calculated as in equation (1).

$$d = \frac{\Delta y}{y} \cdot \frac{\lambda}{2} \dots \dots \dots (1)$$

Whereas:-

D: means membrane thickness.

$\Delta Y$  : is the path of laser light deviation.

Y: Interference hem width.

$\Lambda$ :Wavelength of helium-neon laser (632.8nm)

### 5-2 Optical measurements

Optical examinations of magnesium oxide membranes tinged with percentages of (0,1,3,5,7) of nickel were measured using ultraviolet (UV) rays using the spectrometer type SP-8001 and its wavelength between (200-1100) nm and Figure (2) shows an image of the device used to measure visual inspections, the sample is highlighted and the amount of light absorbed is measured at a specific wavelength. This measurement can give information about the level of light absorption and changes in the electronic structure of the material, and then through this measurement we can calculate the rest of the visual examinations such as transmittance, energy gap and absorption.



**Figure 2: UV spectrophotometer.**

## **6- Synthetic measurements**

### **6-1 X-ray diffraction test X-ray**

diffraction examination was measured for films prepared from nickel-tinged magnesium oxide, the device was used type (6100Lab X /Pl XRD), to perform the X-ray diffraction examination process can follow the following steps: -

1. Sample preparation: Preparation of pure nickel-tinged magnesium oxide membranes by pulsed laser deposition (PLD) method.
2. Prepare the XRD device: The XRD device is properly equipped and set the necessary parameters such as the deviation angle, the deviation range and the appropriate X-ray source.
3. X-ray reflexology: X-rays are directed towards the sample and reflected rays are measured. X-rays are guided by control of the angle of deviation and proper recording of reflected rays.
4. Data analysis: The data obtained from the XRD device is analyzed using special analysis programs to analyze the results, including comparing the resulting patterns with the approved international databases of known crystal patterns, determining the crystal structure, and information related to atomic arrangement and distances between crystal layers.

## 6-2 Scanning Electron Microscope (SEM)

A scanning electron microscope (SEM) is a type of electron microscope that is used to image and analyze the surface structure of solid samples at the atomic level and surface details with high resolution. It has some of the main characteristics of the scanning electron microscope: such as magnification and clarity, as the scanning electron microscope can capture high-resolution images of surfaces at a magnification of up to 1,000,000 times or more. And deep focus, as the scanning electron microscope can take clear images of different parts of the sample at the same time, unlike ordinary optical microscope.

## 7- Results and discussion

### 7-1 Structural properties

of pure magnesium (MgO) films mixed with nickel (Ni) were studied by X-ray diffraction technique to know the crystal structural changes of the materials, the crystal size, the process of homogeneity of the constituent elements of the prepared membranes, the study of the topography of the membranes by

atomic force microscope (AFM) technique, the particle roughness and grain size.

### 7-1-1 X-ray diffraction

Has shown the results of an examination using X-ray diffraction technology for pure magnesium membranes (MgO), as magnesium oxide appears in the case of pure sample and through Figure (3) that the X-ray spectrum of (MgO) membranes as having a cubic structure, by observing the diffraction pattern and knowing the locations of peaks of membranes prepared by pulsed laser sedimentation method, the appearance of levels (111), (200), (211), (220), (311) and (222). As shown in Table (4) and this corresponds to the international card (ASTM) (ICDD) and numbered (1197-001-00), but after mixing with nickel, nickel appeared with three peaks (111), (200) and (220) when adding larger percentages of nickel, the intensity of nickel peaks increases and the intensity of magnesium oxide peaks decreases, and according to the international card numbered (00-154-1139), the crystal structure of nickel is a cubic composition (Cubic), but after adding it to magnesium oxide, it has the same crystal

structure of magnesium oxide composition (Cubic), which indicates that The material is starting to homogenize more and merge with each other better, and this is consistent with the results of the crystal size Table (3) shows the results of diffraction of X-rays of membranes prepared from nickel-tinged magnesium oxide and for all mixing ratios and this is consistent with the findings of the researcher (Nagu) and his group [7] However, they were the

crystal size in their study larger and a slight difference in some peaks and their intensity and through the results of X-ray diffraction examination there was a crystal growth due to the tendency of magnesium oxide peaks to decrease their intensity by adding nickel, which explains to crystal growth However, in their study, the addition hinders the crystal growth of some peaks due to the change in surface energy[8]

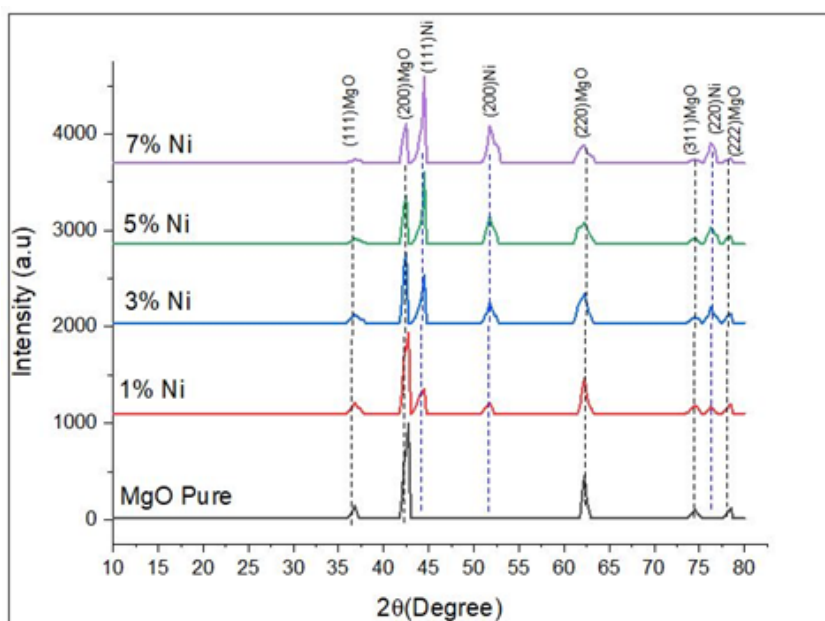


Figure (3) X-ray diffraction of pure and nickel-added (MgO) membranes.

### 7-1-2 The results of the scanning electron microscope (SEM)

The examination of the membranes of (MgO) pure and added to nickel was carried out by scanning electron microscopy (SEM) Figure (4) (a, b, c,

d) shows pictures of the electron microscope scanning the granules of pure magnesium oxide and added to nickel, as we note that the nanoparticles spherical shape and lumpy and assembled patterns and densities different and it

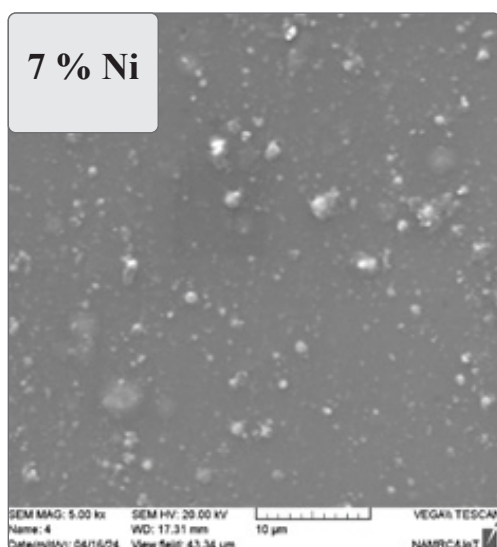
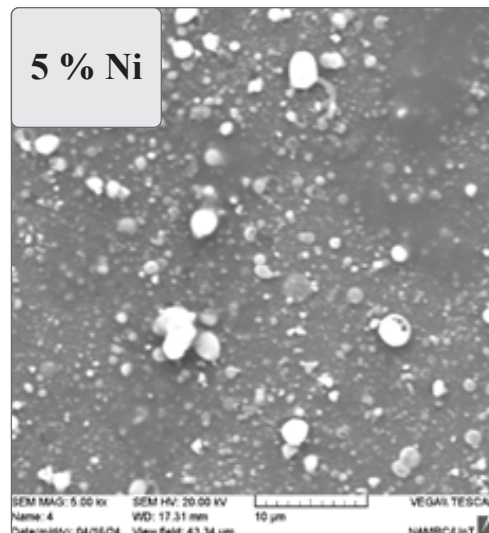
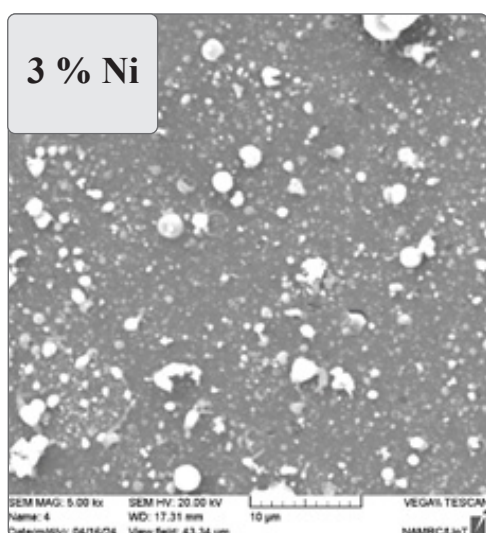
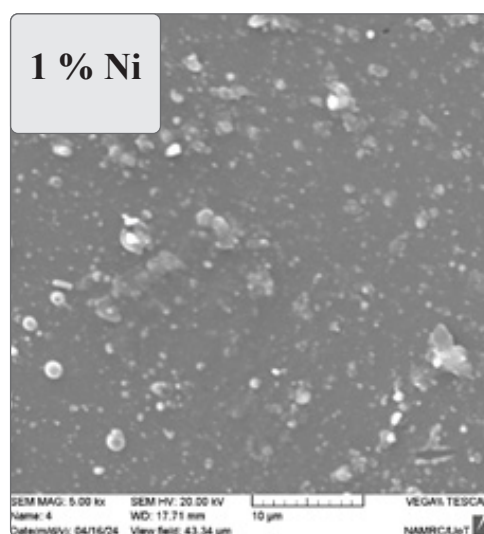
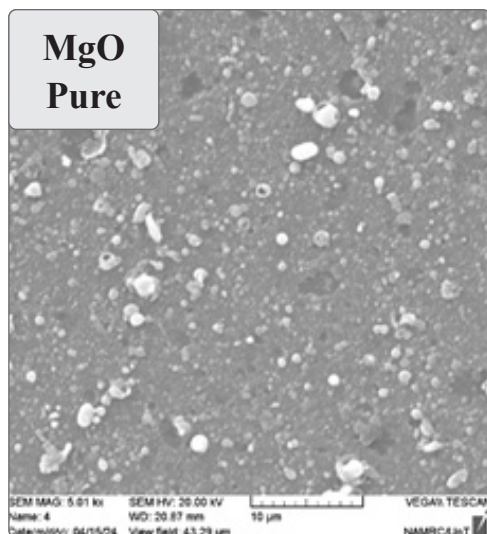
is possible to distinguish between the boundaries and this is evidence that the membrane is multicrystalline and the presence of nickel atoms in a replacement with magnesium oxide atoms and this works on the increase of collisions. Thus, the energy of molecules is lost enough to form molecular assemblies. [9]

The value of the grain size of the membranes was calculated as shown in Table (2), as we note that the increase in grafting led to a decrease in the granular size. The reason is attributed to the precipitation process as well as the process of adding nickel to magnesium oxide, meaning that there is crystal growth between the two materials. and this is consistent with the results of X-rays. We also note the membrane inlaid by (7%) in the form (4-e) is distinguished from the rest of the membranes by better diffusion and good homogeneity, and this may be due to the effect of the nickel percentage on the pure membranes compared to the rest of the ratios or to the conditions of preparation, including a difference in temperature or because the prepared solution is sometimes left for a longer time before being deposited on

the glass bases as an attempt to obtain a clear solution free of sediment and plankton[10].

**Table (2) Granular size scanning electron microscope.**

| Sample      | D <sub>AV</sub> (nm)SEM |
|-------------|-------------------------|
| MgO ( Pure) | 82.13                   |
| 1%Ni        | 79.71                   |
| 3%Ni        | 78.32                   |
| 5%Ni        | 76.98                   |
| 7%Ni        | 72.55                   |



**Figure ( 4 )  
Images and results  
(AFM) for all pure  
magnesium dioxide  
membranes with nick-  
el additive.**

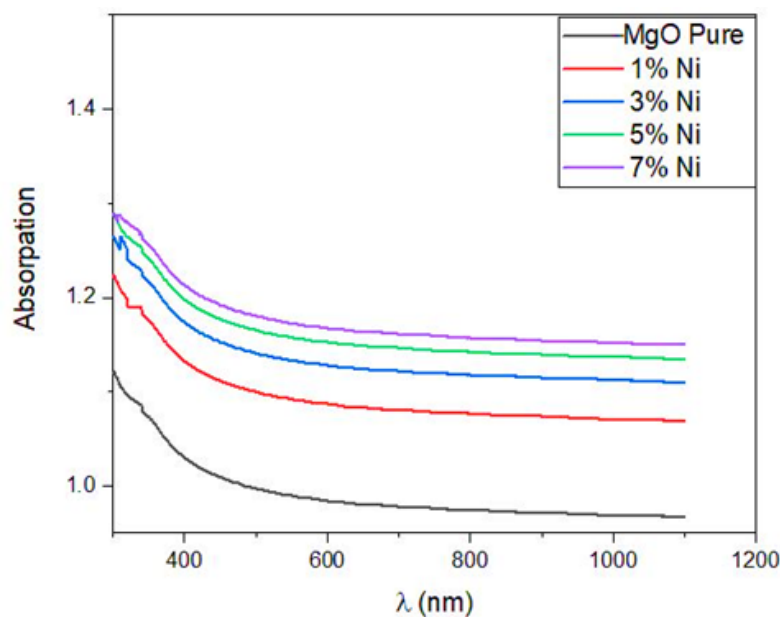
## 7-2 Optical Measurements

### 7-2-1 Absorption

The results of the absorbance values of pure magnesium oxide (MgO) films mixed with nickel (Ni) prepared by pulsed laser deposition decrease with increasing wavelength and nickel mixing process, as Figure (5) shows that the value of absorbance is greatest at short wavelengths (300nm) and then the absorbance values gradually decrease with increasing wavelength until the absorbance reaches its lowest value at the wavelength nm (1100), which indicates that the prepared membranes Nickel-tinged magnesium oxide has a great absorbency at the vis-

ible light area, which makes it suitable in some electronic applications such as solar cells, and decreases with increasing wavelength, and the reason is that the energy of the incident photon is less than the value of the energy gap of the semiconductor and this prevents the electron from moving from the valence beam to the conduction beam.[11]

The reason for the increase in absorption values with the increase in nickel mixing ratios is due to the generation of defect sites and thus the creation of additional energy states within the energy band gap. The high concentration of the defect site with the concentration of Ni thus contributes to increased absorption. [12]



**Figure 5: Absorption spectrum of magnesium oxide membranes**

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

Figure (6) shows the relationship between the absorption coefficient and photon energy of membranes prepared by pure pulsed laser deposition method tinged with mixing ratios (0,1,3,5,7). It was found that the values of the absorption coefficient of membranes are equal to ( $\alpha < 10,000$ ) and this indicates the electronic transitions that occurred are of the type of direct transitions. Through Figure (6), we can see that

the nickel mixing process leads to an increase in the values of the absorption coefficient, and this means that the membrane material began to crystallize and homogenize when mixed with nickel and that the mixing process led to a reduction in crystalline defects and a reduction in the local crystalline levels that exist within the forbidden energy gap that was generated due to defects or defects within the crystal structure[13] .

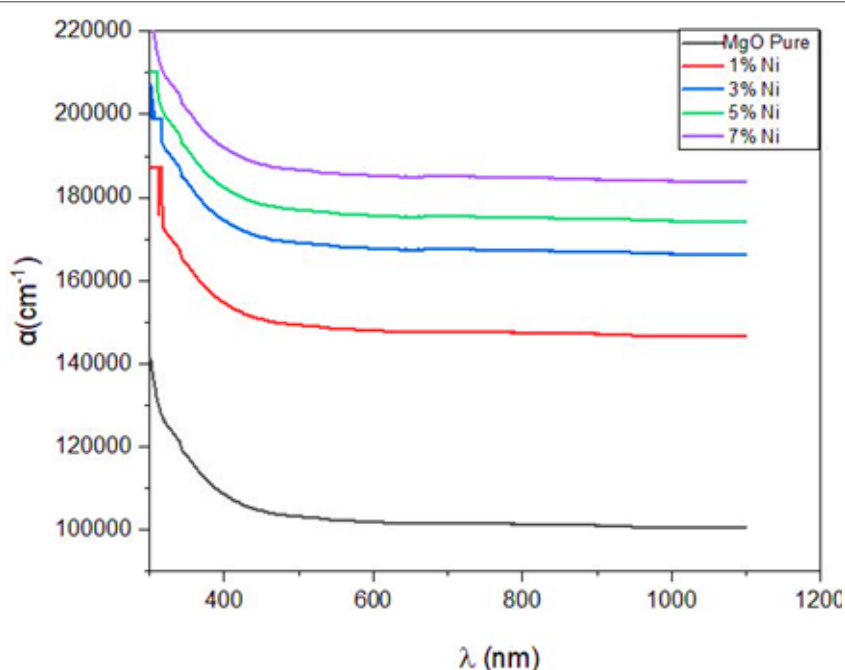


Figure 6: The relationship between the absorption coefficient and photon energy of magnesium oxide membranes.

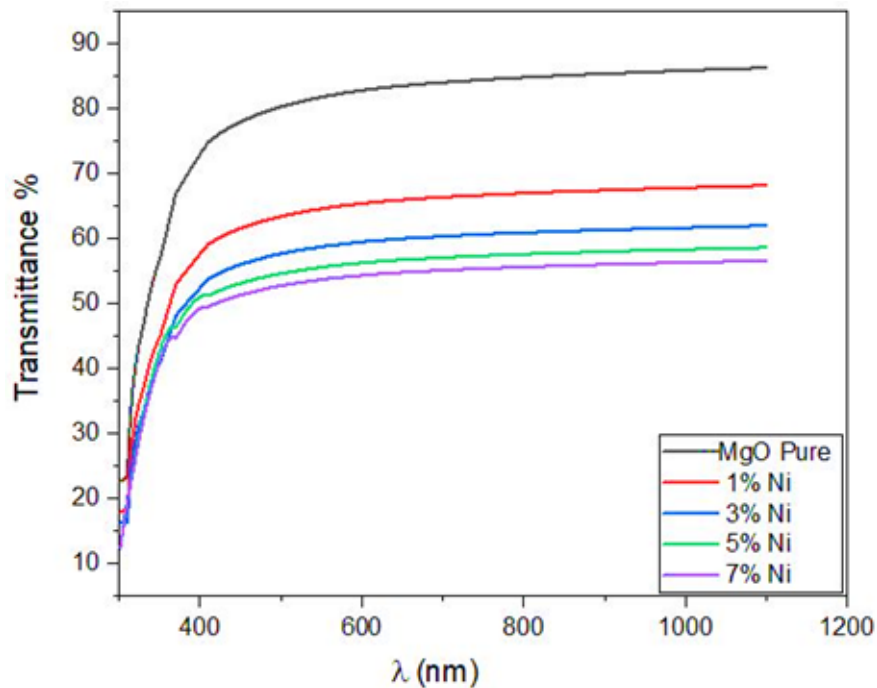
### 7-2-3 Transmittance

Figure (7) shows the relationship between permeability and wavelength, if the results show there is a decrease

in permeability values whenever the percentage of mixing with nickel increases as in Figure (7), magnesium oxide membranes have the highest

permeability by about (80-90) at room temperature within the visible spectrum region, and Figure (7) shows the permeability spectrum with the wavelength of pure magnesium oxide membranes mixed with nickel at mixing ratios of % (1,3,5,7). It can be seen from the figure that the transmittance decreases with increasing mixing rates,

but it begins to increase gradually and for all the prepared membranes when the wavelength increases, as the transmittance spectrum of these prepared membranes can be used in the manufacture of the photodetector because it is 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 prepared from (MgO) and pure and nickel-tinged has been calculated. A graphic relationship has been drawn between  $(\alpha h\nu)^2$  with the energy of the incident photon ( $h\nu$ ) as shown in Fig-

ure (8), as this figure shows that the change is linear in a certain range of photon energies (the range of the visible region). The value of the energy gap of the pure magnesium oxide membrane was equal to (7.79 eV), but when mixing magnesium oxide with

nickel, the energy gap began to decrease, the increase in mixing led to a decrease in the value of the energy gap from (6.72eV) for pure membranes to

(3.43eV) for the membrane added to it and the reason is due to the presence of surface defects to the tail of the absorption curve.[15]

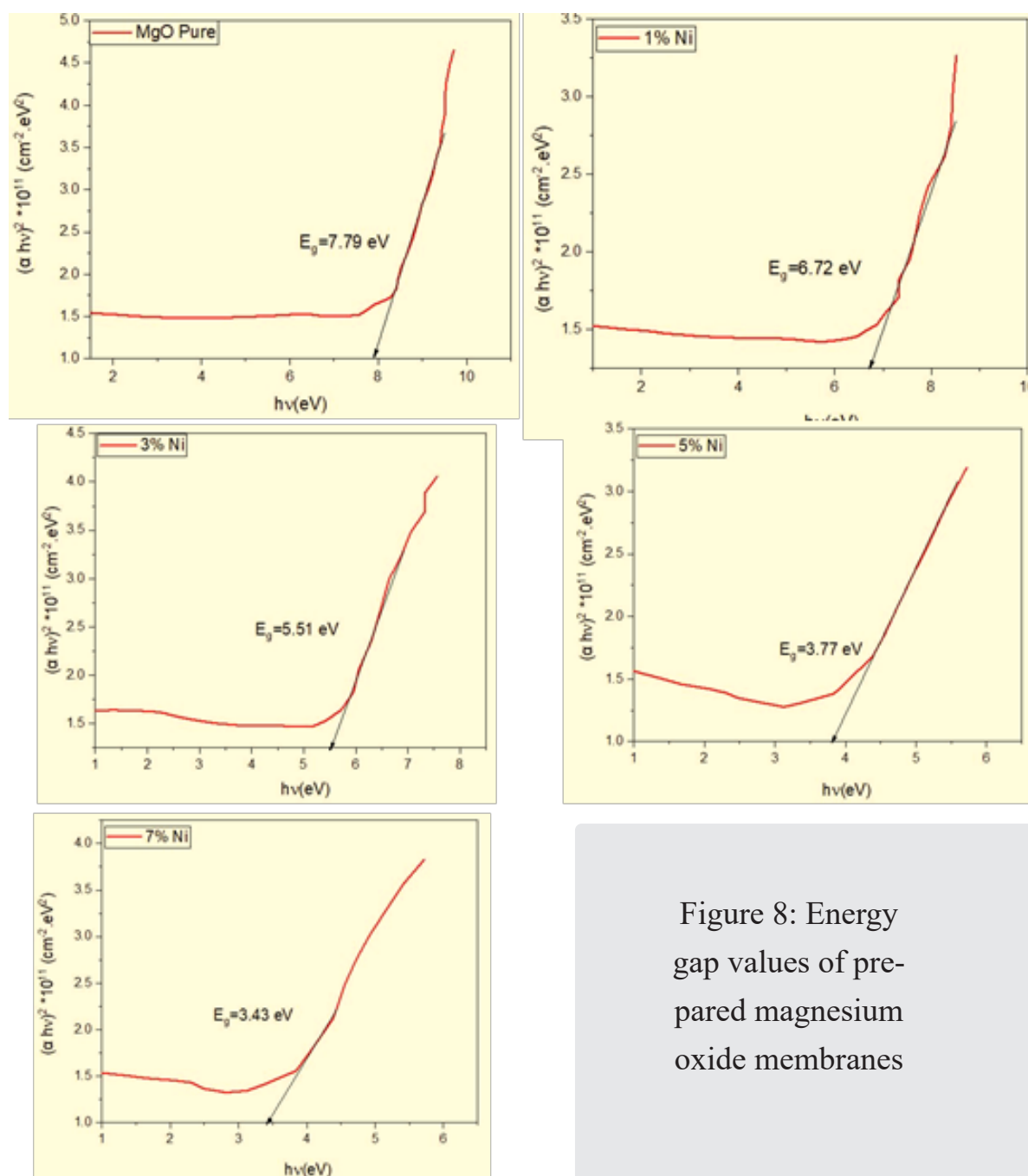


Figure 8: Energy gap values of prepared magnesium oxide membranes

## 8- Conclusions

Through structural examinations, the addition of nickel led to a decrease in crystal size, as for visual examinations, it led to an increase in absorptivity, absorption coefficient, decreased permeability, as well as a decrease in the energy gap, and by comparing the method used in the study with previous studies that the method of laser deposition of membrane formation, is an important and preferred method, and its membranes are characterized by a lack of impurities during the preparation and sedimentation process.

## References

1. Ohring, Milton. "Mechanical properties of thin films." *Materials Science of Thin Films* ,711-781,2022.
2. Sze, Simon M., Yiming Li, and Kwok K. Ng. *Physics of semiconductor devices*. John wiley & sons, 2021.
3. Petosa, Adamo R., et al. "Aggregation and deposition of engineered nanomaterials in aquatic environments: role of physicochemical interactions." *Environmental science & technology* 44.17 6532-6549,2010.
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 , 2669-2681,2020.
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. Allaedini, Ghazaleh, Siti Masrin-da Tasirin, and Payam Aminayi. "Synthesis of CNTs via chemical vapor deposition of carbon dioxide as a carbon source in the presence of NiMgO." *Journal of Alloys and Compounds* 647 ,809-814,2015.
  10. Yang, Guangmin, Qiang Xu, and Weitao Zheng. "Synthesis and characterization of amorphous hollow carbon spheres." *Journal of Materials Science* 47 , 2072-2077,2012.
  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 ,e1501425,2016.
  12. Anandan, K., D. Siva, and K. Rajesh. "Structural and optical properties of (ZnO/MgO) nanocomposites." *Int. J. Eng. Res. Technol* 7.8 , 493-499,2018.
  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.

