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INFLUENCE OF THICKNESS ON SOME PHYSICAL CHARACTERIZATION FOR NANOSTRUCTURED MgO THIN FILMS†

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MgO Nanostructured thin films with different thicknesses (200, 400, and 600 nm) have been deposited by the chemical spray pyrolysis technique. The results confirm that the structure, morphology, optical, and electrical properties were all affected by the thickness of the film. MgO films' physical properties were examined using (XRD), (FE-SEM), (EDX), (AFM), (UV-Vis spectrophotometer), and the Hall Effect. According to the structural analysis, the films have a cubic magnesium oxide polycrystalline structure, with a preferred orientation (002). The average Crystalline Size and optical band gap are found in the range (20.79-18.99) nm and (3.439-3.162) eV respectively with an increase in thickness. The surface morphology of the films reveals that they are free of crystal defects such as holes and voids, as well as homogeneous and uniform. The EDS patterns show that the as-grown films contain magnesium and oxygen. The Hall Effect shows that electrical conductivity decreases with thickness. The experimental results show that film thickness influences the physical properties of as-grown MgO thin films and that thicker films can be used as an absorber layer in solar cell applications.
Keywords: *MgO films; Chemical spray pyrolysis; Effect of thickness; Structure; Optical and electrical properties*
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1. INTRODUCTION

Magnesium oxide, which has good electronic, optical, magnetic, electrical, thermodynamic, and mechanical properties, is one of the most intriguing and promising materials in the family of metal oxides [1,2]. MgO is a scientific and industrial substance. It is a crystalline highly ionic insulator that is stable at high temperatures. Magnesium oxide thin films have drawn a lot of interest because of their numerous applications. Because of their high transparency, good protection qualities against ion bombardment, and high durability, they're commonly used as a protective layer for AC-plasma display panels [3,4]. MgO also has a wide band gap and a low refractive index, allowing it to be used in a variety of ferroelectric superconducting and ferroelectric thin film materials and buffer layers, as well as interlayers in the structure of magnetic memory device [5,6,7,8,9]. MgO thin films have also been used as buffer layers for superconducting and ferroelectric thin films as well as interlayers with a magnetic memory device structure. MgO has a wide range of applications, including adsorption, reflecting and antireflecting coatings, electronics, fire retardants, ceramics, catalysis, chemical, toxic waste management [10,11], solar cells, laser diodes, and many other high-tech applications [12,13]. Many researchers prepared magnesium oxide films using physical and chemical methods according to the specific application of each method, such as chemical spray pyrolysis [14], electron-beam [15], Pulsed laser [16], Sol-gel [17], chemical vapor [18], and spin coating [19], etc. In the present work, we have tried to study the effect of thickness on Some Physical Characterization of Nanostructured MgO Films on glass substrates by the chemical spray pyrolysis technique.

2. EXPERIMENTAL PROCEDURES

(MgO) thin films with different thicknesses (200, 400, 600) nm were a fabrication by utilizing the chemical spray pyrolysis technique, The magnesium chloride $MgCl_2 \cdot 6H_2O$ solution was prepared by dissolving it in deionized water while stirring continuously. The substrates (glass) were thoroughly cleaned with ultrasonic and then washed with distilled water and acetone. As the carrier gas, the air was used, and the solution was sprayed onto a heated substrate using a nozzle. The nozzle's diameter was 0.3 mm. The nozzle was (30 cm) away from the heated substrate surface, and the (1.5 bar) compressor and different thicknesses were obtained by changing the number of sprays in each experiment.

X-ray diffraction was used to determine the structural properties, field emission scanning electron microscopy and atomic force microscopy were used to investigate the morphology of the thin films, a (UV-Vis) spectrophotometer was used to measure the optical properties, and Hall effect measurement was used to determine the electrical properties.

3. RESULTS AND DISCUSSION

3.1. Structural and Morphological Characteristics

The XRD exam was carried out to investigate the crystal structure type of magnesium oxide MgO films. Figure 1. shows the XRD patterns of MgO films deposited at different thicknesses (200, 400, and 600 nm) respectively. The

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detected characteristic peaks at (2Θ= 36.88º, 42.85º, 62.21º and 78.51º) of planes (111) (002) (022) and (222) attributed to the cubic magnesium oxide with space group (Fm-3m no. 225) [20,21], with (a=b=c= 4.2170 A°) and (α=β= 90°), And matching it with the standard data (JCPDS 98-000-9863) [22]. The results revealed that with the increase of MgO film thickness at (400 and 600 nm); the characteristic peaks of MgO became sharper and more intense [23].

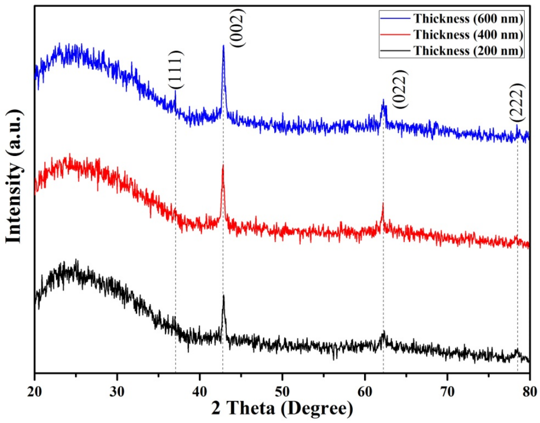

Figure 1. XRD of MgO films at Different Thicknesses.

The XRD Parameters of synthesized MgO nanoparticles was determined by Scherrer's equation at different thicknesses (200, 400, and 600 nm) to be (20.79, 19.83, and 18.99 nm) of the MgO nanoparticles respectively [24,25], the crystalline size of a film decreases with increasing thickness. This is because as the thickness of the film increases, the number of grain boundaries in the film increases, which reduces the average grain size [26, 27], this is because the surface energy of the particle increases with decreasing size, which causes the lattice parameter to contract. The magnitude of this contraction depends on the material and its surface energy [28]. Table (1) presents all synthesized MgO films' obtained parameters from the XRD.

Table 1. XRD Parameters of MgO Films at Different Thickness

Thickness (nm)	2Θ Experimental	2Θ Standard	FWHM (deg)	Crystalline Size (nm)	d_{hkl} (Å) Experimental	d_{hkl} (Å) Standard	(hkl)
200	42.81	42.85	0.3557	20.79	2.1106	2.1085	(002)
400	42.91	42.85	0.3728	19.83	2.1059	2.1085	(002)
600	42.89	42.85	0.3892	18.99	2.1069	2.1085	(002)

The morphology of MgO film surfaces was investigated using (FE-SEM), which provides high-resolution and magnification images of the surfaces. We observe that the surface structures of the prepared films are highly agglomerated aggregates of dense quasi-spherical nanoparticles. and clearly show the voids, nonuniform particle sizes, and irregular shapes in the MgO films. This is consistent with Boo J. H. et al. and Bian, J. M. et al. [29,30], As in Figure (2). The thickness of MgO sheets has a significant impact on particle size. In theory, the atoms diffuse into MgO and arrange themselves into larger particle sizes following deposition for various periods. When the thickness of MgO thin films is raised, small particles coalesce with one another, The recrystallization of particles caused by the increase in thickness encourages the reorientation of the overall microstructure [31,32].

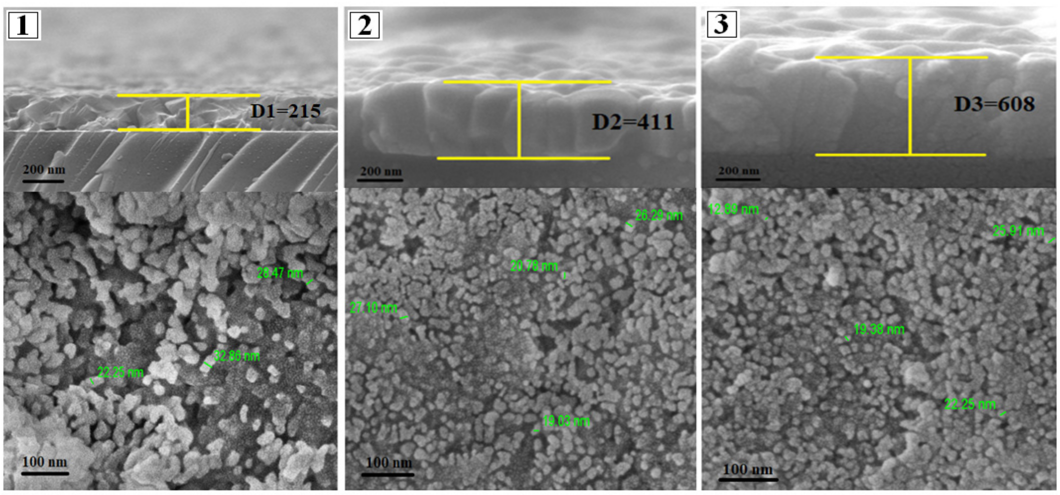

Figure 2. (FE-SEM) of MgO and the cross-section at Different Thicknesses

The Energy Dispersion X-ray Analysis (EDX) in Fig. (3) shows the presence of only the Mg and O. As shown in Table 2. The Mg: O atomic ratio is equal to 0.767 : 1 for sample 1 (200nm), 0.707 : 1 for sample 2 (400nm), and 0.648 : 1 for sample 3 (600nm).

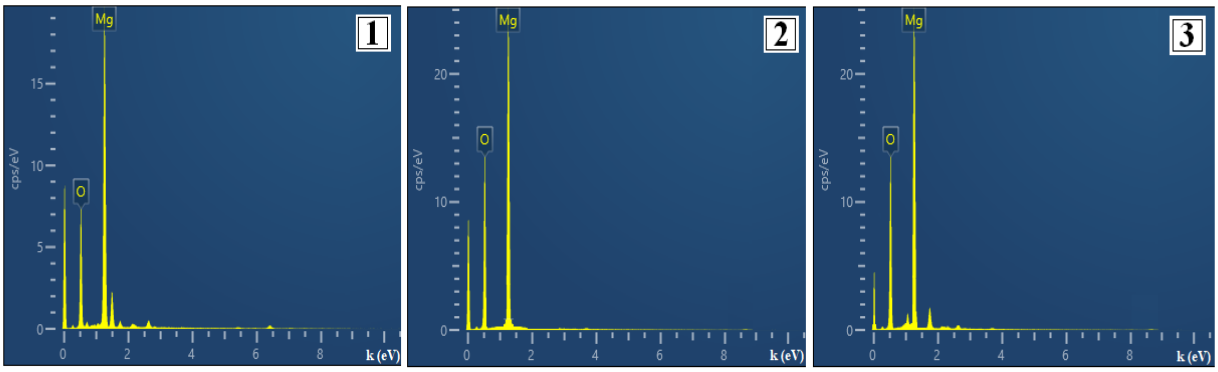

Figure 3. EDX of MgO films at Different Thicknesses

Table 2. The Ratios of the Elements of MgO from EDX

	200 nm		400 nm		600 nm	
Element	Weight %	Atomic %	Weight %	Atomic %	Weight %	Atomic %
Mg	53.84	43.43	52.16	41.45	50.42	39.33
O	46.16	56.57	48.84	58.55	49.58	60.67
Total	100	100	100	100	100	100

To recognize the surface characteristics and particle size distribution of prepared films in the current study, The AFM is presented in Fig. (4) and Table 3.

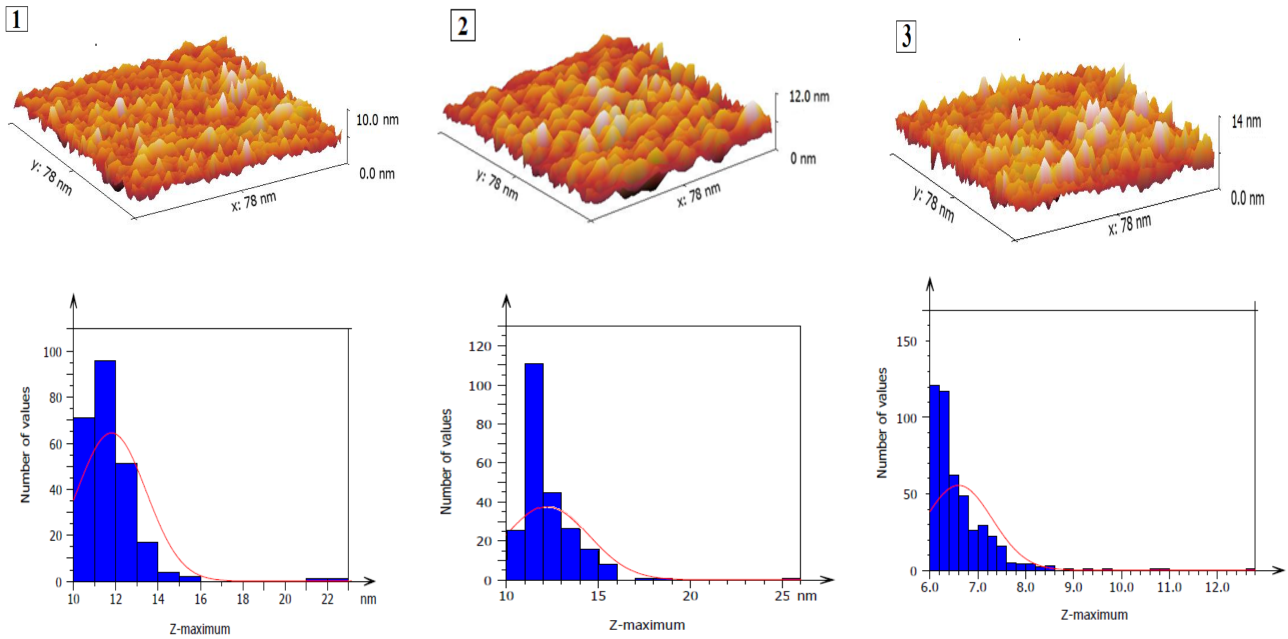

Figure 4. AFM images of MgO at different thicknesses

Table 3. Roughness and RMS of MgO Films at Different Thicknesses.

Thickness (nm)	Roughness Average Sa (nm)	RMS (nm)	Particles Size (nm)	Surface Skewness (RsK) (nm)	Surface Kurtosis (Rku) (nm)	Maximum Height Sz(nm)
200	4.541	5.139	10.970	0.138	1.884	24.77
400	4.231	5.423	10.231	0.327	3.884	27.99
600	3.194	3.833	9.191	0.580	2.493	35.41

The particle size grows with the thickness of the film and it was discovered to be uniformly distributed. Many nucleation centers form on the substrate when deposition begins, resulting in the formation of small crystallites. Because the films are only deposited for a short period of time, the small crystallites on the substrate cannot grow into large crystallites, and thus the thinner films have smaller crystallites than the thicker films [33]. The crystallinity of the films is related to the surface roughness, which can be improved by increasing the film thickness. The larger crystallite size and higher crystallinity of the films can lead to better electrical and optical properties. The surface roughness of MgO films increases with increasing film thickness, which is due to the increased number of grain boundaries and defects in the film. This can lead to increased scattering of light, resulting in a decrease in optical transmittance [34]. The crystallinity of the films improved as the film thickness increased, and the surface roughness of MgO films increased [35], as shown in Fig. 5, This result is consistent with the thickness XRD observation.

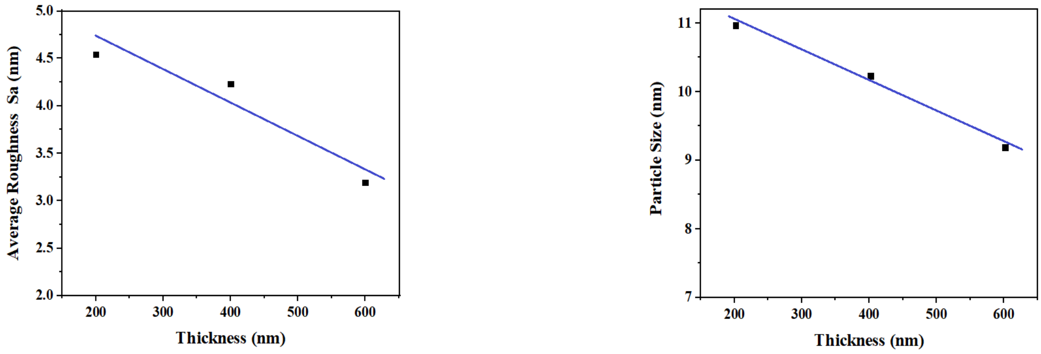

Figure 5. Average roughness and particle size with a thickness of MgO films

3.2. Optical Properties

When different thicknesses of MgO films are used during the preparation method, the optical behavior of MgO films will change dramatically as a result of the new structural and morphological features. Increasing the thickness of a material will generally decrease its optical transmittance. This is because thicker materials absorb more light, reducing the amount of light that can pass through. Additionally, thicker materials are more likely to scatter light, further reducing the amount of light that can pass through [36]. The MgO films' transmittance and reflectance spectra are shown Fig. (6).

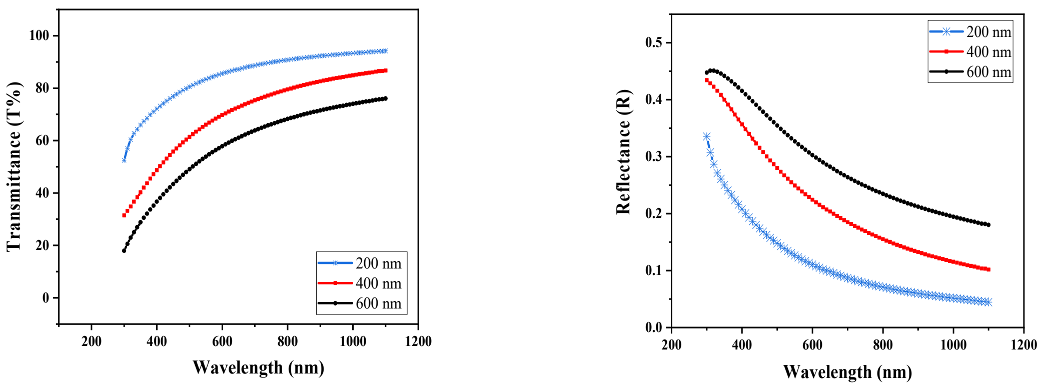

Figure 6. Transmittance and Reflectance of MgO films

In the visible range, MgO thin films have high transmittance values. It can be used in solar cell devices as an optical window. In general, the decrease in (T%) could be attributed to scattering effects, crystalline structure, and surface homogeneity, as reported by [37,38]. Transmittance is a measure of how much light passes through a material. The optical energy gap determines how much light can be transmitted through a material, as materials with larger gaps tend to have higher transmittance values [39]. Reflectance is a measure of how much light is reflected off a surface, while transmittance is a measure of how much light passes through a material. The optical energy gap affects both reflectivity and transmittance, as materials with larger gaps tend to reflect more light and transmit less [40,41].

The absorption coefficients (α) of MgO films were calculated using the Lambert equation (1) [42]:

$$\alpha = \ln\left(\frac{1}{T}\right)/d. \tag{1}$$

where: (T) the transmittance, (d) film thickness.

Figure (7) shows the calculated absorption coefficient of MgO films. The highest absorption coefficient was obtained for the films prepared with higher thickness than those prepared with less thickness.

The energy gap and particle size of the material are both affected by variations in thickness. As the thickness of a material increases, the energy gap typically decreases, while the particle size increases. This is because thicker materials have more electrons and therefore a larger number of states available for electrons to occupy. This reduces the energy gap between the highest occupied molecular orbital and the lowest unoccupied molecular orbital, resulting in a decrease in the energy gap [43,44]. The increased number of electrons also leads to an increase in particle size, as larger particles are needed to accommodate all of the electrons [45]. The following equation can be used to calculate the energy gap for the as-prepared films [46]:

$$\alpha h v = A(h v - E_g)^n. \tag{2}$$

(A) : constant, hυ : energy of the photon, n is determined by the nature of the transition. The value was (1/2) in this case (a direct band transition).

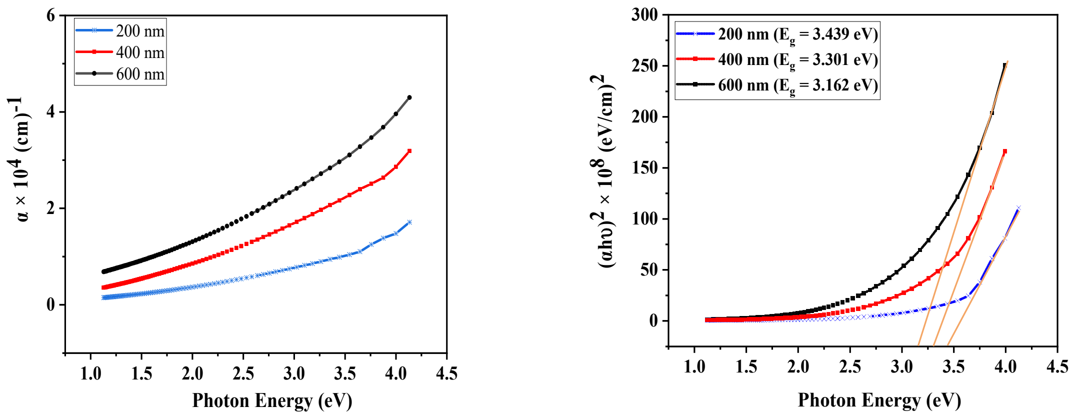

Figure 7. The Absorption coefficient and Energy gap of MgO films

The optical transmittance value is determined by the film's refractive index (no), which is influenced by the thickness of the film. As the thickness of the film increases, the refractive index increases, and the optical transmittance decreases. This leads to a decrease in the Eg value [47]. It is evident that when thickness grows, the Value of Eg rises. The Eg values are comparable to those that Płóciennik, P. et al. [48], and Tamboli, Sikandar H., et al. [49].

The higher the refractive index, the lighter is reflected off a surface, The refractive index (no) is related with reflectance (R) by the relation [50]:

$$n_o = \left[\frac{(1+R)^2}{(1-R)^2} - (k_o^2 - 1)\right]^{\frac{1}{2}} + \frac{(1+R)}{(1-R)} \tag{3}$$

The refractive index behaves very similarly to reflectance. Figure (8) shows the correlation between refractive index (n_o) and photon energy for MgO films. the refractive index increases with thickness. The equation (4) can be used to calculate the extinction coefficient (ko) [51]:

$$k_o = \frac{\alpha\lambda}{4\pi} \tag{4}$$

(k_o) extinction coefficient.

$$\varepsilon_r = n_o^2 - k_o^2 \tag{5}$$

$$\varepsilon_i = 2 n_o k_o \tag{6}$$

Figure (8) shows the refractive index and extinction coefficient of MgO films.

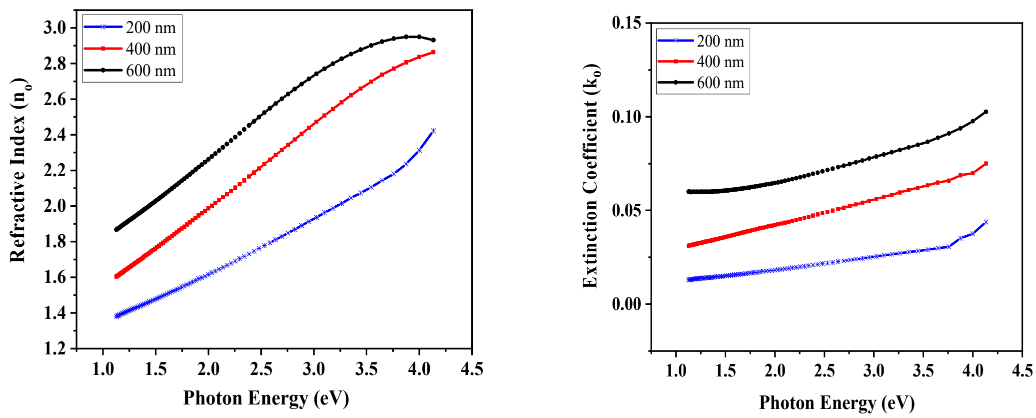

Figure 8. Refractive index and Extinction Coefficient of MgO films

The dielectric constant can be expressed as a complex number, with both real and imaginary parts. The real part of the dielectric constant represents the ability of the material to store energy, while the imaginary part represents losses due to absorption and other effects. The behavior of the real dielectric constant is associated with the refractive index and the imaginary dielectric constant with the extinction coefficient with the equation (5) and (6) [52,53], as shown in Figure (9). Table 4 shows the optical properties of MgO films at different thicknesses (200, 400, and 600 nm).

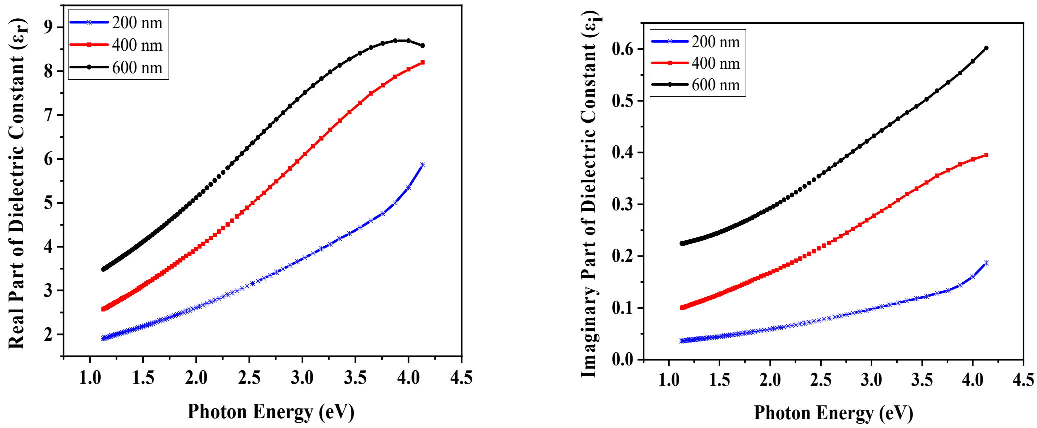

Figure 9. The real and imaginary part of the dielectric constant of MgO films

Table 4. Optical Properties of MgO Films at Different Thicknesses

Thickness (nm)	Eg (eV)	($\alpha\times10^4$ cm^{-1}) Maximum	(n_o) Maximum	(k_o) Maximum	(E_r) Maximum	(E_i) Maximum
200	3.439	1.7076	2.4241	0.0442	5.8652	0.1888
400	3.301	3.1893	2.8586	0.0742	8.1553	0.3921
600	3.162	4.3024	2.9451	0.1092	8.7144	0.6001

3.3. Electrical Properties

Hall coefficient is a measure of the strength of a material's response to an applied magnetic field [54]. The Hall coefficient can be affected by the thickness of thin films, as thinner films tend to have higher Hall coefficients due to their increased surface area and reduced number of charge carriers [55], As the thickness of the film increases, the electrical resistance increases. This is because as the thickness of the film increases, there are more atoms and molecules in the material that can impede the flow of electrons. The increase in resistance is due to an increase in collisions between electrons and atoms or molecules in the material [56]. As shown in Table 5. Additionally, as the thickness of a thin film increases, its capacitance also increases. This is because as the thickness of a thin film increases, its surface area also increases, which allows for more charge to be stored on its surface, and the decrease in mobility can be attributed to an increase in dislocation density and lattice strain [45], as shown in Fig (10).

Table 5. Electrical Properties of MgO films at Different Thicknesses

Thickness (nm)	Concentration $(cm)^{-3}$	Hall Coefficient Rh(m^2/C)	Conductivity $(\Omega\cdot cm)^{-1}$	Resistivity ($\Omega\cdot$cm)	Mobility (cm^2/v·s)
200	0.2244×10^{11}	8.792×10^{6}	4.590×10^{-6}	2.178×10^{5}	1.276×10^{1}
400	-0.2163×10^{11}	-2.886×10^{6}	5.337×10^{-7}	1.874×10^{6}	1.54
600	-3.069×10^{11}	-2.034×10^{7}	3.369×10^{-6}	2.968×10^{5}	6.852×10^{1}

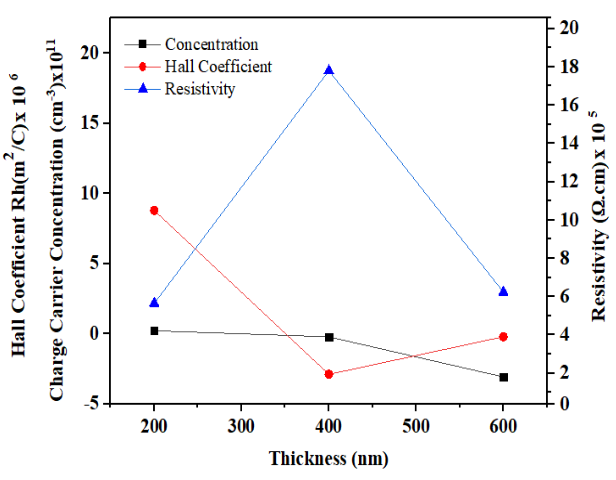

Figure 10. Electrical Properties of MgO films at Different Thicknesses

4. CONCLUSIONS

The thickness of the transparent MgO films prepared by chemical spray pyrolysis can be controlled by varying the deposition time. The films are composed of nanocrystalline grains with a size range from 10 to 50 nm, also The films exhibit good adhesion to the glasses substrate and The rate of roughness decreases with increasing the thickness of the film, The crystallite size decreases with increasing the thickness of the thin film, In the visible region, MgO films exhibit high transmittance with values up to 80%, The refractive index of the films increases with increasing film thickness, The films also exhibit low reflectance in the visible region, The energy gap (Eg) of the films increases with increasing film thickness, indicating that they can be used as optical coatings (color filters), and optical absorbers for applications such as photodetectors or solar cells. The electrical resistivity of MgO films decreases with increasing film thickness, indicating that they can be used as conductive layers for electronic devices such as transistors.

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ВПЛИВ ТОВЩИНИ НА ФІЗИЧНІ ХАРАКТЕРИСТИКИ НАНОСТРУКТУРНИХ ТОНКІХ ПЛІВОК MgO

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Технологія хімічного розпилювального піролізу осадила наноструктуровані тонкі плівки MgO різної товщини (200, 400 і 600 нм). Результати підтверджують, що товщина плівки вплинула на структуру, морфологію, оптичні та електричні властивості. Фізичні властивості плівок MgO досліджували за допомогою (XRD), (FE-SEM), (EDX), (AFM), (UV-Vis спектрофотометр) і ефекту Холла. Відповідно до структурного аналізу, плівки мають кубічну полікристалічну структуру оксиду магнію з переважною орієнтацією (002). Середній розмір кристала та ширина забороненої зони знаходяться в діапазоні (20,79-18,99) нм і (3,439-3,162) еВ відповідно зі збільшенням товщини плівок. Морфологія поверхні плівок показує, що вони вільні від кристалічних дефектів, таких як отвори та пустоти, а також гомогенні та однорідні. Карти EDS показують, що вирощені плівки містять магній і кисень. Ефект Холла вказує, що електропровідність зменшується з товщиною. Експериментальні результати показують, що товщина плівки впливає на фізичні властивості вирощених тонких плівок MgO. Більш товсті плівки можна використовувати як шар поглинача в сонячних елементах.

Ключові слова: *плівки MgO; хімічний розпилювальний піроліз; вплив товщини; структура; оптичні та електричні властивості*