Optical and morphological investigation of aluminium and nickel oxide composite films deposited by spray pyrolysis method as a basis of solar thermal absorber

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Abstract. Applications of alumina and nickel oxide in various fields specially in solar energy conversion technology encouraged us to study physical properties of such materials. Hence after the deposition of the thin films on glass substrate by spray pyrolysis, using X-ray diffraction (XRD), scanning electron microscopy (SEM), and UV–visible spectrophotometry, various physical properties were investigated. Different optical quantities such as optical band gap, refractive index, extinction coefficient, dielectric constants, volume and surface energy loss functions and optical conductivity were determined. Within this paper for different nickel to aluminium ratio (from 20/80 to 80/20 ratio) at specific substrate temperatures (300°C), decrease of optical transmittance with nickel content was notable. Using the transmittance data, other optical quantities were achieved by a numerical approximation method. We also observed an increase in the volume energy loss (VELF) more than the surface energy loss (SELF) and simultaneously a decrease trend prevailed according to nickel amount. On the basis of the XRD results, the amorphous phase changed by the presence of more nickel and according to SEM, more obvious nanosized spherical grains at higher nickel ratios can be observed.

Keywords. Optical properties; surface properties; composite materials; Urbach energy; electron microscopy.

1. Introduction

The optical properties of thin films are very important especially for their applications, e.g., antireflection coatings, integrated optics, solar power engineering, microelectronics and optical sensor technology depending on the reflectance and transmittance properties of the films.¹ Therefore, preparing a mixture of semiconductor and dielectric materials together and investigating their optical properties may be interesting.

Aluminium oxide, commonly known as alumina, is a widely used material in the family of engineering ceramics. Alumina as a dielectric material possesses excellent properties,² such as high thermal conductivity, low permeability of alkali ions, high hardness,³ high chemical and thermal stability⁴ and high radiation resistance.³ Alumina thin films have a wide range of applications such as refractory coatings, antireflection coatings, anticorrosive coatings,⁵ microelectronic devices, capacitance humidity sensors, VLSI applications,⁶ water-repellent coating,⁷ waveguide lasers, buffer layers for superconductors,² organic light-emitting devices (OLED), solar selective coatings, bar code readers, optical lenses and windows.⁸

Nickel oxide (NiO) is a semitransparent *p*-type semiconductor material.⁹ It has an energy gap of 3.4–3.8 eV with

grey colouration. It represents attractive optical, magnetic,¹⁰ electric,¹¹ electrochromic,¹² thermo-electric¹³ properties as well as high chemical resistance¹⁴ among various applications like as gas sensors,¹⁵ smart windows,¹⁴ UV detection,¹⁶ anti-ferromagnetic materials,¹⁷ fuel cell,¹⁸ solar thermal absorber,¹⁹ transparent electrode,²⁰ catalyst for oxygen evolution²¹ and photoelectrolysis.²²

Alumina and nickel oxide thin films can be deposited by several techniques such as chemical vapour deposition (CVD),^{23,24} spray pyrolysis,^{25,26} electron beam evaporation,^{27,28} magnetron sputtering,^{29,30} anodization,^{31,32} sol–gel,^{33,34} pulsed laser deposition,^{35,36} atomic layer deposition (ALD).^{37,38} Among the different methods for film deposition, spray pyrolysis is suitable for the preparation of efficient, time-resistant and inexpensive thin films.² It is very simple, low cost³⁹ and does not require vacuum or exotic gas. In this method, fine droplets react on the heated substrate, owing to the pyrolytic decomposition of the solution. The hot substrate provides the thermal energy for the thermal decomposition and subsequent recombination of the constituent species. The phenomenon for the preparation of a metal oxide thin film depends on surface hydrolysis of metal salt on a heated substrate surface.⁴⁰ Thus, the substrate temperature, carrier gas flow, substrate rotating speed, number of spraying sequences, spraying distance, solution flow rate and molarity play an important role in forming the structure of the films ranging from amorphous to crystalline.^{41,42}

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In this report we have prepared aluminium and nickel oxides composite thin films in different molarities onto a glass substrate by spray pyrolysis (SP). The optical and morphological properties, e.g., transmittance, band gap, refractive index, extinction coefficient, dielectric constants, surface and volume energy loss and optical conductivity were investigated for obtaining a greater understanding of such structures.

2. Experimental

Our SP device is an experimental apparatus (SCS 90, made by Modern Technology Development Co., Iran) which consists of a spraying unit including a swivel stainless steel (ss) plate over the heater for rotation and heat treatment of substrate. It also has a moving (up and down and rotative) nozzle unit with its controlling tools (e.g., velocity, distance, pressure, aperture diameter, etc.), solution time controller and compressor.

Our optimized values are as follow: The nozzle–substrate distance ($H = 34.5$ cm), the carrier gas pressure (air, $p = 1.5$ bar) and the substrate temperature 300°C , these conditions as well as the substrate rotation speed and nozzle aperture diameter are fixed.

Aqueous alcoholic solutions of AlCl_3 (98%, Merck) and $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (97%, Chem-Lab) were used as precursors for the alumina and nickel oxide thin films in preparation. Deionized water (W) and absolute ethanol ($\text{C}_2\text{H}_5\text{OH}$, 99.9%, Merck) were used as solvents (water: ethanol = 1:1, in volumes). The main precursor solutions had 0.25 mol l^{-1} concentration and nickel content (mole percentage of nickel ions to metallic ions) were altered as 20%, 40%, 60%, 80%. Finally, the solution was sprayed perpendicularly onto preheated glass substrates at 300°C which was previously immersed in HNO_3 10% solution for 24 h and then cleaned with acetone and dry air.

Optical transmission for samples measured with a Varian Cary100 UV/visible spectrophotometer. Thickness measurement of films was done by Dektak system. The optical constants of the films were calculated using the pointwise unconstrained minimization approach (PUMA).⁴³ The surface morphology was observed using a scanning electron microscope (SEM) HITACHI S4160.

For easier refer to the samples, they are abbreviated as shown in table 1.

3. Results and discussion

To investigate the optical properties of thin films, transmittance spectra for all films were analysed. A good similarity

exists between the measured spectra and PUMA calculations (figure 1). The PUMA software implements an unconstrained formulation of the nonlinear programming model using a method based on repeated calls to a recently introduced unconstrained minimization algorithm. This method implements the complex optical equations, derived and formulated by Heavens⁴⁴ and Swanepoel.⁴⁵ By PUMA, important parameters such as refractive index n , extinction coefficient k , the film thickness d and an optimized transmittance can be obtained. Although an initial guess of thickness should be considered until a reliable results is achieved. We have acquired them by Dektak Profilometer as mentioned before and listed in table 2. By above information, other parameters such as band gap E_g , Urbach energy E_U , dielectric constants ε_1 , ε_2 , volume and surface energy loss function and optical conductivity can be estimated.

According to figure 1, by increasing the nickel content, the transmittance of the films reduced. Although alumina is known as a good absorber by itself,⁴⁶ but it seems that two factors affect the transmittance and make the reduction trend justifiable. In one hand, thickness and packing density of the films have an inverse relationship with transmittance⁴⁷ and on the other hand, the presence of the nickel particles would increase absorptance.⁴⁸ Our reflectance data (not reported here) had also such trend (decrease form by nickel increase)

Figure 1. Experimental and estimated optical transmission spectra for thin films (geometric shapes and solid lines for simulated and measured values respectively).

Table 2. Measured and estimated values of thickness for thin films and their transmittance.

Sample	Measured values of thickness (nm)	Estimated values of thickness (nm)	Transmittance (%)
S1	349	346	60
S2	957	955	52
S3	1151	1151	35
S4	1432	1430	24

Table 1. Samples names.

Nickel to aluminium ratio	20/80	40/60	60/40	80/20
Sample name	S1	S2	S3	S4

that verify our claim. This is suitable for special applications such as solar-selective absorber.

The optical energy gap values depend in general on the structure of the films. The absorption coefficient and the incident photon energy are related by the equation

$$(\alpha h\nu) = A(h\nu - E_g)^N, \quad (1)$$

where α is the absorption coefficient, A the constant, E_g the band gap of the material and the exponent N depends on the type of transition. $N = 1/2, 2, 3/2, 3$ corresponding to allowed direct, allowed indirect, forbidden direct and forbidden indirect, respectively.⁴⁹ There are several relations to evaluate absorption coefficient,^{45,50} such as

$$\alpha = -\frac{1}{d} \ln \left(\frac{(n+1)^3(n+s^2)}{16n^2s} T \right), \quad (2)$$

where d , n , s and T are the thickness, the refractive index of the film, the refractive index of the substrate and transmittance spectrum, respectively.

The determination of E_g was made by extrapolating the linear portion of the curves until they intercept the photon energy axis. The allowed direct transition band gap values (are shown in figure 2) are varied between 3.65 eV (for S1) and 3.93 eV (for S4), hence an increase trend is prevailed by the nickel content.

In the region between the weak absorption tail and the strong absorption region (Tauc region), the optical absorption coefficient follows Urbach rule.⁵¹ The absorption coefficient shows an exponential energy dependence, referred to as the Urbach tail.⁵²

$$\alpha = \alpha_0 \exp \left(\frac{h\nu}{E_U} \right), \quad (3)$$

where α_0 is the constant and E_U determines the width of the tail. The Urbach energy, is generally attributed to disorder in the material that leads to a tail in the valence and conduction bands. Figure 3 shows the variation of $\ln \alpha$ vs. $h\nu$ for the

Figure 2. Determination of the optical band gaps from the plots of $(\alpha h\nu)^2$ vs. photon energy $h\nu$.

thin films. From the inverse of the slope of the diagram, the values of E_U can be obtained. The relevant values are given in table 3.

It is obvious from the results that the E_U values change inversely with direct optical band gaps of the films. It can be related to formation of defects during thin films preparation. These defects produce localized states in the films. Thus, the increase in width of the localized states in the optical band gap decreases the optical band gap.⁵³

The width of the edge E_U , is also related to another parameter which is the slope of the Urbach edge,⁵⁴ and is obtained by

$$\sigma_U = \frac{K_B t}{E_U}, \quad (4)$$

where K_B is Boltzmann's constant and t the temperature. The steepness parameter σ_U , value was calculated using $t = 300$ K and is brought in table 3.

Optical constants (i.e., refractive index and extinction coefficient) are important parameters since they determine how a material responds to an electromagnetic field. In the present study, the refractive index, the extinction coefficient and their relevant quantities (such as dielectric constants) of the films have been calculated by PUMA. Of course a fitting of the data to Cauchy's equation was performed after the determination of refractive index and extinction coefficient. This method is highly reliable even in the case of absorbing films or of films that show no fringe pattern in their optical transmission.⁵⁵

Figure 3. The plots of $\ln \alpha$ vs. $h\nu$ for thin films.

Table 3. The optical band gap E_g , Urbach tail E_U and steepness parameter σ_U .

Sample	E_g (eV)	E_U (eV)	σ_U
S1	3.65	0.780	0.033
S2	3.85	0.531	0.049
S3	3.90	0.393	0.066
S4	3.93	0.256	0.101

The influence of the compositional ratio of nickel on the refractive index and the extinction coefficient of the films are presented in figure 4. By comparing the refractive index and considering the incremental trend with nickel increase, there would be a reason for the presence of more nickel oxide contribution than the alumina in higher nickel content, if our criterion is proximity to the bulk value or values which obtained by other researchers.^{1,56} The variation of the refractive index presents some interactions between photons and electrons in the material. The refractive index for long wavelengths is a smoothly decreasing function with increasing of wavelength without peaks, as shown in figure 4. This decrease in the refractive index can be associated with fundamental band gap absorption.⁵⁴ Meanwhile the refractive index of the films varies inversely with transmission data. The high (low) transmittance spectra have low (high) refractive index.⁵⁷

Extinction coefficient reflects the absorption of electromagnetic waves due to inelastic scattering events such as the Compton effect, photoelectric effect, pair production effect and so on.⁵⁸ Despite an initial decrease, an increase trend is observed in extinction values. Although the differences is not significant but they are significantly higher than the pure alumina films^{4,42,43} which shows the role of nickel particles in absorbance enhancement.

It was assumed that materials with a higher extinction coefficient are better absorbers, as the extinction coefficient of pure alumina is trivial and the difference between their values in our samples is tiny, it seems absorbance depends on higher refractive index⁵⁹ in agreement with our transmittance spectra and absorption coefficient relation, Relation (1).

The values of refractive index and extinction coefficient at 550 nm are listed in table 4.

The polarizability of any solid is proportional to its dielectric constants and their variation with photon energy is an indicative of the interactions between photons and electrons in the films in the energy range.

The real and imaginary parts of the complex dielectric constant are expressed as⁶⁰

$$\varepsilon_1 = n^2 - k^2 \text{ and } \varepsilon_2 = 2nk, \quad (5)$$

where ε_1 and ε_2 are the real and imaginary parts of the dielectric constant, respectively. The variation of ε_1 and ε_2 values of the samples with photon energy are shown in figure 5 and listed in table 4. The ε_1 and ε_2 values are analogous to corresponding complex refractive index (real part n and imaginary part k), since according to Relation (5), ε_1 depends on the square of n (subtraction is negligible), whereas ε_2 is related to multiplication of n and k . For this, the real parts of the dielectric constant are higher than that of the imaginary parts of the dielectric constant.

According to table 4, by increasing the nickel content, the relative increase would be observable, so that a minimum value of 2.89 and a maximum value of 9.89 (for real part of dielectric function) are appeared between them. The real part is important, especially in microelectronic applications.⁵

The inelastic scattering process on a surface, where energy quanta are transferred to or from the topmost atomic layers of solid, yields information about possible excitation of a

Table 4. The refractive index, extinction coefficient and dielectric constants values of the samples at wavelength of 550 nm.

Sample	Refractive index	Extinction coefficient	Real part of the dielectric constant	Imaginary part of the dielectric constant
S1	1.70	0.049	2.89	0.16
S2	1.79	0.023	3.21	0.08
S3	2.52	0.029	6.36	0.14
S4	3.14	0.031	9.89	0.19

Figure 4. The dependence of the refractive index and extinction coefficient of the thin films on the wavelength.

surface or interface, both electronic and vibronic ones. The spectral response of the solid in an inelastic electron scattering experiment can be described within the framework of dielectric theory named energy loss function.⁶¹ Energy loss function has a valuable role to investigate different aspects of materials. It has the advantage of covering the complete energy range including valance interband transitions and core level excitations.⁶² Two forms of energy loss function are usually interested, volume energy loss function (VELF) and surface energy loss function (SELF)

$$\text{VELF} = -\text{Im} \left(\frac{1}{\varepsilon^*} \right) = \frac{\varepsilon_2}{\varepsilon_1^2 + \varepsilon_2^2} \text{ and}$$

$$\text{SELF} = -\text{Im} \left(\frac{1}{\varepsilon^* + 1} \right) = \frac{\varepsilon_2}{(\varepsilon_1 + 1)^2 + \varepsilon_2^2}. \quad (6)$$

These quantities were estimated using real and imaginary parts of the dielectric constant, and plotted against energy (figure 6). From the calculated results it is obvious that the energy loss of the charge carriers when traversing through the bulk material has fairly the same behavior as when they traverse the surface. Although, it is clear that the VELF has a higher value than the SELF.

The energy loss spectrum of the low energy loss region typically 0–100 eV reflects collective electron excitations (plasmons) and single valence-electron excitations into unoccupied states of the conduction band. Hence, electron energy loss in the low energy loss range is sensitive to the valence and conduction band structures.⁶³

What is noteworthy for both, is a decrease trend of volume and surface energy loss amount, namely with approaching

Figure 5. The variation of real and imaginary parts of the dielectric constant with wavelength.

Figure 6. Plots of VELF and SELF vs. energy for thin films.

the more nickel contribution the less loss is achieved which is favourable for special applications such as solar spectral selectivity.

The frequency dependence of dielectric reflects the fact that the polarization of a material does not respond to an applied field instantaneously. For this, the dielectric constant is often treated as a complex function of the frequency of the applied field. A perfect dielectric is a material that has no conductivity. However, the presence of dielectric loss inhibits the propagation of electromagnetic energy which aided conductivity.⁶⁴

Optical conductivity is an interesting tool for studying the electronic states in materials. In general, by exerting an external electric field to a system, in general, a redistribution of charges occurs and currents are induced. The real part of optical conductivity can be determined by using the following relation:⁶⁵

$$\sigma = \frac{\omega}{4\pi} \text{Im}(\epsilon) = \frac{\alpha n c}{4\pi}, \quad (7)$$

where c is the velocity of light, α the absorption coefficient and n the refractive index.

Figure 7 shows the variation of optical conductivity vs. photon energy for thin films.

Figure 8 shows XRD patterns of thin films as a function of nickel content. The dominant amorphous nature of thin films especially in lower nickel content are obvious. But by increasing the ratio of nickel incorporation, some weak diffraction peaks appear. According to JCPDS no. 22-0451, there is rather good agreement between standard diffraction peaks of nickel aluminium oxide and our films. The relevant diffraction planes are marked into the diffraction curves. The amorphous structure of alumina in the temperature range below 550°C is ordinary^{5,66,67} even with doping material.⁶⁸ Meanwhile the presence of nickel oxide characteristic peaks is reported at higher substrate temperature (>350°C)¹ and lower molarity (<0.1 M).¹⁰ Therefore, it seems alumina facilitate the presence of nickel oxide crystalline peaks at higher nickel content with lower substrate temperature and higher molarity. Although weak intensity peaks and polycrystalline structure is observed.

Figure 7. Plot of optical conductivity vs. photon energy.

Our electrical measurement also confirmed the role of alumina since at lower nickel content, there is not any electrical resistance whereas nickel oxide has a semiconductor property against the dielectric property of alumina.

The SEM micrographs of thin films in different nickel contents are shown in figure 9. Generally speaking, based on the micrographs, all samples have similar microstructures. Although all of the micrographs consist of nano-sized grains but by increasing the nickel content, rather obvious spherical grains are observed. According to our previous experiment,^{41,42} alumina seems to have a spongy structure that establishes an appropriate status to infiltrate other elements such as a nickel.

Figure 8. X-ray diffraction pattern of thin films.

Figure 9. SEM images of thin films.

4. Conclusion

The optical and morphological properties of aluminium and nickel oxide thin films deposited on glass substrate by spray pyrolysis have been investigated. The effects of different ratios of nickel content were compared. The optical transmittance of the films decreased from 60% (S1) to 24% (S4) at 550 nm for various nickel ratios and our observations also exhibited a relevant increase trend for absorptance values. The values of the optical band gap varied between 3.65 and 3.93 eV for different films which comprised an increase trend. The refractive index and the extinction coefficient of the samples were also determined. The refractive index values have been varied between 1.70 and 3.14 and the extinction coefficient have been changed between 0.049 and 0.031 for the various samples. The real and imaginary parts of dielectric constants had analogous behaviour with corresponding complex refractive index because of their physical relations. In addition to the higher values of volume energy loss function than surface energy loss, a relative decrease in both due to nickel increase was also observed. According to XRD data, despite the overcoming amorphous nature of alumina especially in lower nickel content, when increasing nickel content, some weak diffraction peaks were appeared which were in good agreement with nickel aluminium oxide structure. However, a polycrystalline structure was formed. Although, similar micrographs were obtained by the SEM analysis, rather obvious nanosized spherical grains were developed by increasing the nickel content.

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