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Optimization and Characterization of Spray Pyrolysis Synthesized Nanostructured Nickel Oxide Films

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Abstract: Using spray pyrolysis technique, crystalline nanostructured Nickel Oxide (NiO) films were synthesized on glass substrates with different molarities of hydrated nickel chloride salt precursors solution ($NiCl_2.6H_2O$) at various substrate temperatures. Characterizations of synthesized nanostructured NiO films were investigated by using X-Ray Diffractometer (XRD), UV-Visible Spectrophotometer (UV–Vis), and Field-Emission Scanning Electron Microscopy (FESEM) with Energy Dispersive X-ray Spectrometer (EDX). Based on XRD analysis, the molar concentration and substrate temperature of the prepared films were optimized as 0.2 M and 400 °C, respectively. The XRD result showed to the formed cubic polycrystalline structure films with preferred orientation along (111) direction. The UV–Vis spectrophotometry analyzing showed the band gap of NiO films varied from 3.630 eV to 3.85 eV. The surface morphology analysis revealed that the nanoparticles are extra-fine, with an average particle size of 68.838 nm. The stoichiometric elemental composition ratio of NiO was confirmed by EDX analysis. The overall results of this study suggested that the synthesized high quality crystalline nanostructured NiO films would be highly suitable for gas sensing applications.

Keywords: Spray Pyrolysis, NiO nanostructure, Molarity, Substrate Temperature, Characterization

1. Introduction

Compared with their bulk properties, recently nanostructured materials have received attracted extensive interest in various fields due to their unique physical and chemical characteristics which depend on their structure, shape, size, and specific surface area (Kate, Khalate, & Deokate, 2017). The high surface area to volume ratio in nanostructured materials leads to excellent chemical reactivity and strength (Chen, Lu, & Hwang, 2005). Due to these excellent properties, nanomaterials show great advantages over conventional materials in many applications in different life sciences (Chen et al., 2005). In general, semiconductors metal oxides such as V_2O_5, ZnO, TiO_2, ZrO_2, SnO_2, NiO…. have remarkable optical and electrical properties and they are extensively used for a different opto-electronic devices application (Vera et al., 2005). Most of the traditional metal oxides usually is n-type conductivity, while notwithstanding there is lack of p-type metal oxide. In contrast with other types of the available nanostructured semiconductors metal oxides, nickel oxide (NiO) is a promising p-type metal oxide with a properties of wide gap energy of (3.4–3.8) eV, good adsorptive, non-toxicity and excellent chemical stability (Desai, Min, Jung, & Joo, 2006). However, Nanosized NiO demonstrated

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excellent optical and electrochemical properties (Yang, Lai, & Chen, 2005).

NiO promising candidature for many applications such as gas sensors, solar thermal absorber, photocatalysis, UV photo-detector and electrochromic device (González-Leal, Prieto-Alcón, Angel, Minkov, & Márquez, 2002). A variety of physical and chemical techniques have been employed for synthesizing NiO nanostructure, including vacuum evaporation, electron beam evaporation, atomic layer epitaxy, sputtering, chemical deposition, sol–gel technique and spray pyrolysis method (Raut, Pawar, Chougule, Sen, & Patil, 2011). Among these techniques, spray deposition is suitable technique for the fabrication of NiO films (Tauc, 1968; Tauc, Grigorovici, & Vancu, 1966). Spray deposition has many advantages compared with the other techniques such as low cost, simple deposition equipment, and the films can be coated for large area (Jlassi, Sta, Hajji, & Ezzaouia, 2014).

However, due to their good electrical conductivity and high optical transparency, in the past years NiO was used as a membrane in a gas sensing device (Wang, Zhou, Lu, Wei, & Zeng, 2019). In this work, nanostructures NiO thin films have been synthesized using spray pyrolysis method. The main target of the study is to obtain optimized NiO nanostructured NiO film with high quality used for sensing applications. Noteworthy, further work is underway to synthesis great sensitivity gas sensor based on the prepared NiO thin films, the study which will be reported in the future.

2. Experimental Details

2.1 Spray Deposition System

The spray pyrolysis system which was used for preparation the samples in this work mainly consists of an atomizer, a solution container, heater, and temperature controller an. Further, to improve the quality of the prepared samples, improvement of atomization and solution flow rate control added into the basic system. Schematic diagram of the spray pyrolysis system is presented in Figure (1).

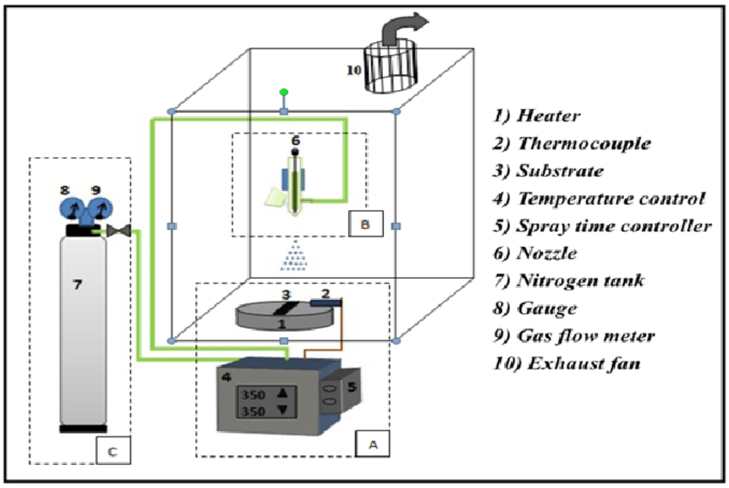


Figure 1: Schematic illustration of spray pyrolysis system

Moreover, moving arrangements were used to obtain uniform large area deposition, herein the substrate, the nozzle, or both of them are moving. The system was kept inside an aluminum vacuum and provided with a fan to remove the gases inside the vacuum, which was produced from the solvent vapor. The sprayed droplet undergoes pyrolytic decomposition and forms a single crystalline or a cluster of crystalline when reaching the hot substrate surface, while the other volatile escape as a vapor form. The hot plate provides the heat to the sample substrate for the thermal decomposition of the constituent and their recombination, and followed by sintering and recrystallizing which gives rise to a continuous sample with the final state.

2.2 NiO Thin Films Preparation

NiO nanostructure films were prepared onto chemically and ultrasonically cleaned and preheated glass substrates with the dimensions (76.2 mm × 25.4 mm × 1 mm) by spray pyrolysis technique equipment at a various substrate temperature of (300°C to 450°C). The glass substrate was put on a hot plate and provide by a heat through an electrical heater, where the substrate temperature was controlled by using a thermocouple. The spraying solution contains NiCl2.6H2O in addition to the deionized water which was used as the solvent. First, the spraying solution was mixed and heated at 30 °C for 20 min to prevent sedimentation. During the deposition processes, all the deposition parameters like molarity of the solution, nozzle to substrate distance (37 cm), spray rate of the solution (5 ml/min), spraying time (10 sec), and time interval between successive spraying (10 sec) were kept constant for all the experiment measurements. However, nitrogen was used as a carrier gas with a pressure of 2 bar for the solution spraying process. Furthermore, the thickness of the films was measured by using weight difference method considering the density of the bulk nickel oxide. The fine aerosols of aqueous NiCl2 solution was sprayed through an atomizer undergo evaporation, solute precipitation and pyrolytic decomposition, onto the preheated glass substrates thereby forming a NiO film according to the reaction below (Gowthami, Meenakshi, Anandhan, & Sanjeeviraja, 2014):

$$NiCl_2 .6H_2O \text{ (heat)} = NiO + 2HCl \uparrow + 5H_2O \uparrow \qquad [1]$$

2.3 Characterization Techniques

The structural, optical and morphology with chemical concentrations of the NiO samples obtained at optimized preparative parameters were investigated. The structural of the NiO nanostructures were analyzed by X-Ray Diffraction (XRD) by a PAN analytical X′ Pert PRO (Cu Kα =1.5406 A°). The optical measurements of the thins films are determined from the absorbance and transmittance spectra at normal incidence over the wavelength range (200 – 800) nm, by using UV-VIS spectrophotometer type (SHIMADZU) (UV-1600/1700 series). The surface morphologies of the samples were characterized by using Field-Emission Scanning Electron Microscopy (FESEM) type Quanta 450. The elemental analysis of NiO nanostructures thin films were determined by electron dispersive X-ray spectroscopy (EDX) type Quanta 450 (FEI, America).

3. Results

3.1. Structural Properties

In order to reach the optimum condition for formation of NiO nanostructure sample, first, different samples of NiO were prepared at a constant substrate temperature with different concentrations of NiCl2 precursor solution. In the next step, after the sample's characterization by XRD, samples were syntheses at various substrate temperatures with the optimized molarity value.

3.1.1 Prepared samples with Different NiO Concentrations

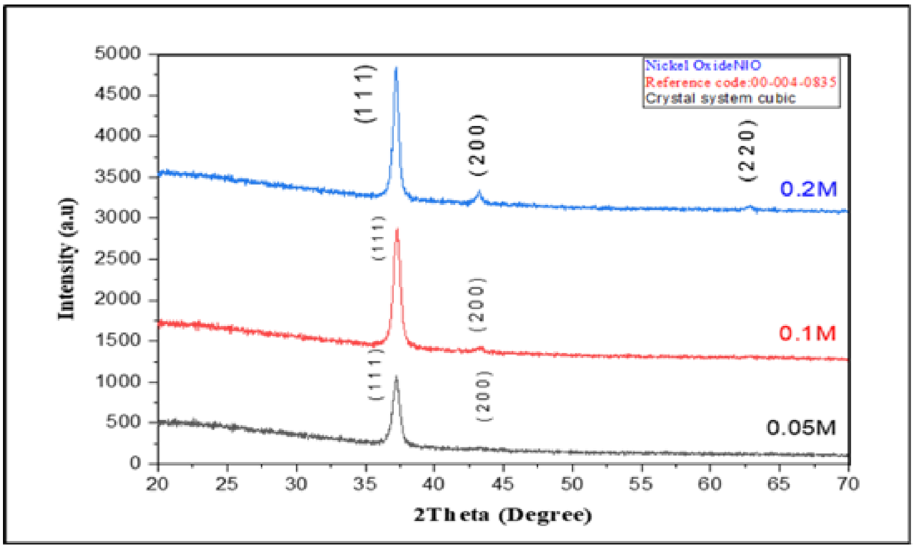


Figure 2: XRD patterns of NiO films prepared with various molar concentration at the same substrate temperature

The XRD spectra of sprayed NiO samples prepared with different molarities of (0.05, 0.10, 0.20) M at a substrate temperature of 400 °C are shown in Figure (2). All the patterns of the deposited films are polycrystalline in nature with cubic phase having a preferred orientation along (111) plane which was located at the position of Bragg angle 2θ = 37.3o, with two others weak reflection along the (200) and (220) planes located at the positions 2θ = 43.4o and 63o, respectively. There were no peaks related to any impurities in the spectra.

However, all the indexed peaks are consistent with the cubic crystalline structured of NiO (JCPDS 00-004-0835). It is clear from XRD pattern, with increasing the molarities the intensity of the peaks increased, while the full-width half-maximum (FWHM) value of the peaks decreased. The strong and sharp peaks indicated to the highly crystalline of NiO nanostructure. The results showed that the crystallization of NiO nanostructure was significantly improved at NiO concentration of 0.2 M, which means that 0.2 M is the optimum molar concentration result. Additionally, further information was obtained regarding the structural quality of prepared NiO thin film at optimum condition such as the interplanar spacing (d), the average crystallite size (D), microstrain (Є) and dislocation lines per unit volume (L) were calculated as below (Erdoğan, Kundakçı, Kasapoğlu, & Gür, 2018).

From the XRD spectrum, the interplanar spacing (d) which defines the crystalline, structure information was determined from the position of the peaks (Bragg peaks). The lattice constant (a) of the preferred orientation (111) plane for cubic NiO crystalline structure was calculated through the following relation (Akaltun & Çayır, 2015):

$$d = \frac{a}{\sqrt{(h^2+k^2+l^2)}} \qquad [2]$$

Where h, k and l are the miller indices of the planes.

$$a = d\sqrt{(h^2 + k^2 + l^2)} \qquad [3]$$

However, the average crystallite grain size (D) of the films was calculated using Scherrer's formula (Patterson, 1939).

$$D = K\frac{\lambda}{\beta \cos\theta} \qquad [4]$$

where λ is the wavelength of the used X-ray (λ = 1.5418 oA), K is the shape factor equal to 0.94 for the unknown shape of grain size, θ is the angle of diffraction peak (Bragg's angle), and β is the FWHM of the diffraction peak which has the maximum intensity. The microstrain structural defect parameter (Є) was estimated from the equation below (Erdoğan et al., 2018):

$$\epsilon = \frac{\beta \cos\theta}{4} \qquad [5]$$

Moreover, the dislocation (L) was determined using the relation below (Erdoğan et al., 2018):

$$L = \frac{1}{D^2} \qquad [6]$$

Variations of all the observed structural parameters prepared for NiO thin film at optimum condition are given in Table (1).

Table 1: XRD results identify the structure parameters for prepared NiO thin film at optimum condition

Concentration (M)	Pos. [°2Th.]	d-spacing [Å]	Height [cts]	FWHM Left [°2Th.]	Crystallite Size only (D) [nm]	Micro Strain only [%]	Dislocation L*10^¯3 (nm¯2)	Microstrain Є*10^¯3
0.05	37.260	2.411	805.27	0.2496	35.035	0.3444	0.8146	3.230
0.1	37.309	2.410	1382.01	0.2558	38.134	0.3160	0.6876	3.306
0.2	37.175	2.418	1525.02	0.2814	34.458	0.3509	0.8421	3.651

3.1.2 Prepared Samples at Different Substrate Temperatures

Afterward, to know the effect of substrate temperature on the crystalline properties of NiO films, the substrate temperature varied between 300 °C and 450 °C. XRD spectra of sprayed NiO prepared at various substrate temperatures of (300, 350, 400, and 450) °C with the optimized NiO concentration of 0.2 M is shown in Figure (3). All the prepared samples have a polycrystalline nature with cubic phase having a preferred orientation along (111) direction. The sample which was prepared at temperature of 400 °C exhibits highly intense peaks. The high intensity of the (111) plane and its narrowing with increasing the substrate temperature up to 400 °C indicates the improvement in the crystallinity of the film, and it can be assumed that at this temperature the atoms enable to move to the stable sites and in turn improve the crystallinity. Conversely, the degree of crystallinity decreased for the film was prepared at higher substrate temperature (400 °C). Accordingly, optimum condition for formation of NiO was found to be at a substrate temperature of 400 °C with NiO molarity of 0.2 M. The formed sample at this condition was the most suitable to be used for synthesis the final NiO sample.

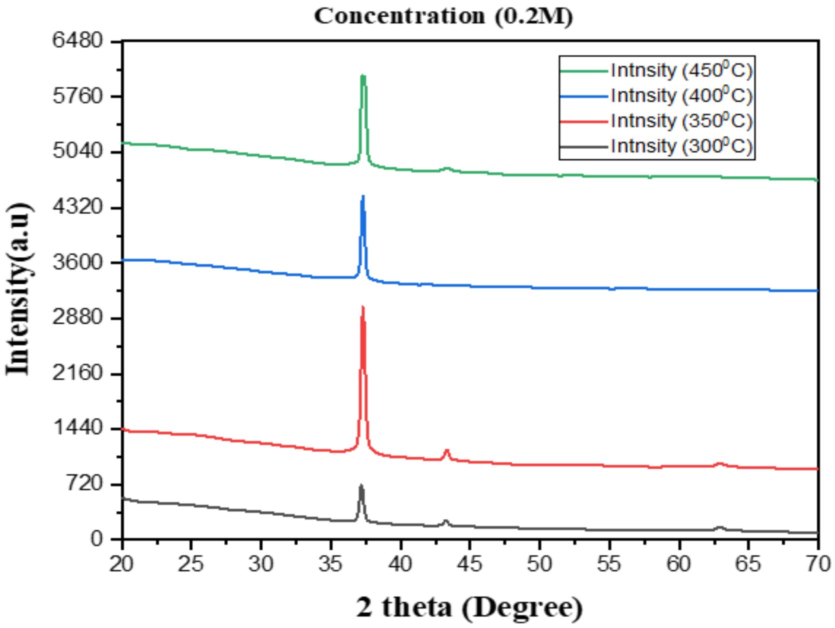


Figure 3: XRD patterns of NiO films prepared at various substrate temperatures with the same molar concentration (0.2 M)

3.2 Optical Properties

NiO films prepared at various temperatures between (300 to 450) °C with optimized molarity were investigated for studying the optical properties in the spectrum wavelength region (200 to 800) nm. Figure (4) depicts the optical spectra of the NiO samples deposited under optimized molarity condition of 0.2 M at different substrate temperatures. The optical spectrum of the prepared samples showed high dependence on the substrate temperature. Using the optical absorption spectra, the value of the energy band gaps (Eg) of the nanocrystalline NiO were determined and presented by the following equation for direct allowed transition (Ganjoo & Golovchak, 2008).

$$\propto (hv) = A\,(hv - Eg)1/2 \qquad [7]$$

$$where$$

$$\propto \text{ is the absorption coefficient, } hv \text{ is the photon energy, and } A \text{ is an energy} - \text{independent constant.}$$

To determine the band gap energy, $\alpha(hv)^2$ was plotted versus (hv). Then, by the extrapolation of the linear regions on the energy axis (hv) to zero absorption coefficient ($\alpha = 0$), the band gap energies were found as shown in Figure (4). The band gap values were found to be 3.630 eV, 3.60 eV, 3.375 eV and 3.85 eV for the samples deposited at substrate temperature 300 °C, 350 °C, 400 °C, and 450 °C, respectively. The lower value of the band gap was found for the sample prepared at 400 °C. The obtained minimum band gap value for the prepared sample at 400 °C can be attributed to the improvement in the crystal structure of the film.

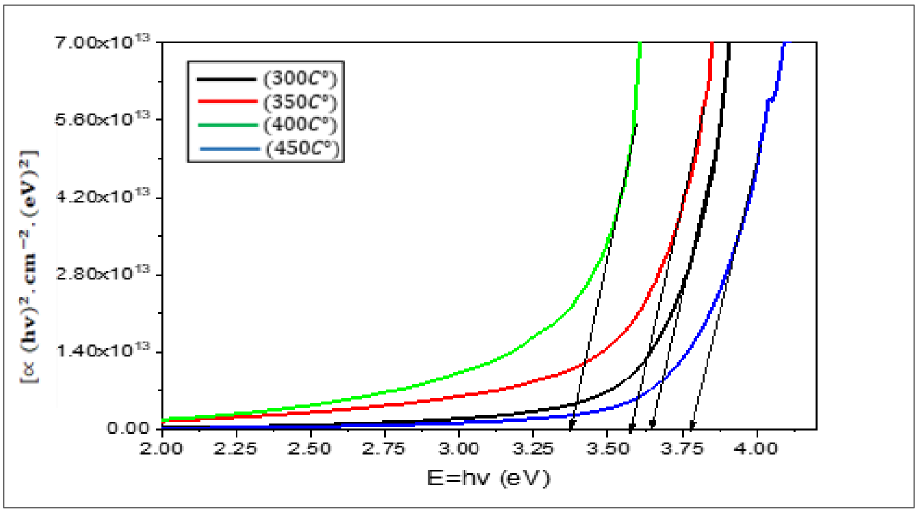


Figure 4: Variation of $[\alpha\,(hv)]^2$ versus (hv) of NiO samples prepared at various substrate temperatures

The determined band gap energies are found to be in good agreement with the values reported in literature (Akaltun & Çayır, 2015).

3.3 Morphological Properties

Investigation of the surface morphology of the prepared films is needed because the surface morphologies of the samples effect on their properties which are important in the applications for the sensing devices. The morphological study is a convenient method for knowing the shape, size and grain growth mechanism of the nanostructure's films. The morphology of the optimum NiO film grown with NiO concentration 0.2 M at 400 0C substrate temperature was investigated by the FESEM at different magnifications, as shown in Figure (5). The results show the homogenous and high-quality formation of nanostructures NiO thin film with different shapes and oriented randomly. Furthermore, the film is good adhered to the substrate with no cracks or aggregation of grain growth on the deposited layer surface. The size distribution of the particles is illustrated in the inset of Figure (5). From the results of FESEM analysis, the average particle size was found to be (68.838) nm.

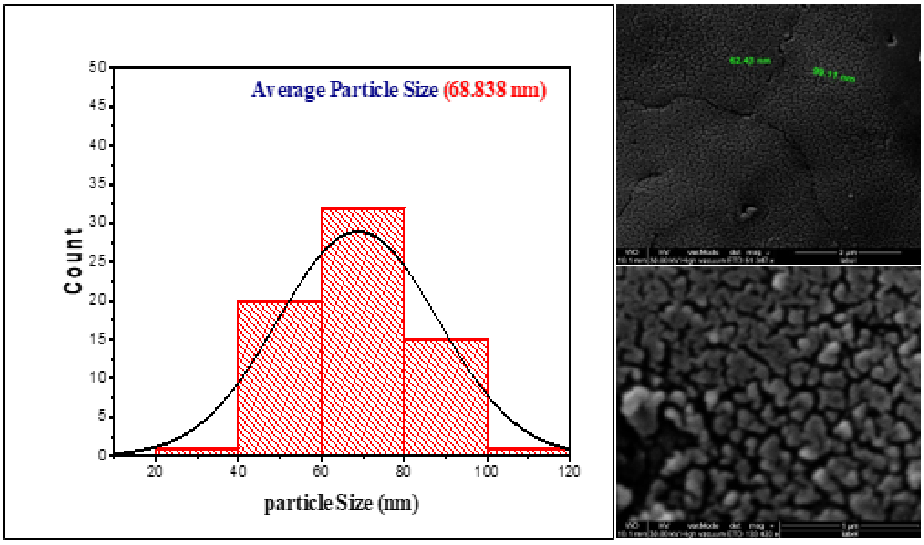
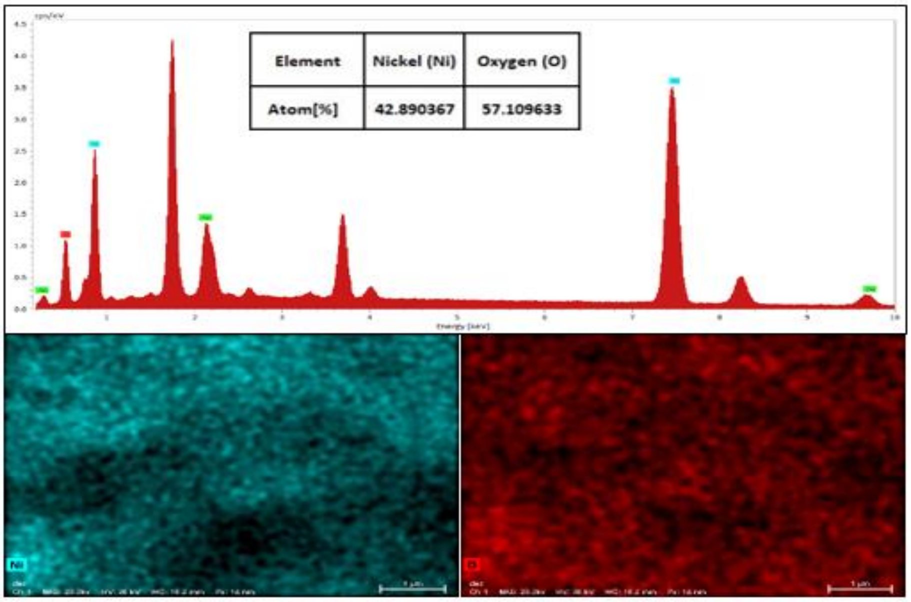


Figure 5: FESEM images of the optimized prepared NiO film and average particle size

3.4 Elemental Analysis (EDX)

The corresponding chemical composition of the elements presented (the atoms ratio of Ni to O) in the optimized NiO film was investigated by the EDX, as shown in Figure 6. The result revealed to the Ni and O elements are presented in the nanostructures NiO samples with no impurity. Besides that, some of Au element is existing in the EDX spectrum which may be due to coating the NiO film with a thin layer of gold to improve the morphology of the film. The measured atomic percentage for the elements Ni and O were found to be 42.890 % and 57.109 %, respectively.



Element	Nickel (Ni)	Oxygen (O)
Atom[%]	42.890367	57.109633

Figure 6: EDX spectrum with elemental atomic ratio of optimized nanostructured NiO film

The atomic percentage ratio of the optimized Ni to O film was found to be 0.75. This result indicates the stoichiometric ratio of Ni: O and the quantitative element distribution in atomic percentage is tabulated in the inset of Figure (6). Further, the two different colors of the EDS in Figure (6) spectrum confirm the stoichiometry of the nanostructured NiO. The EDX results indicated the successful preparation of the NiO thin films and supported the XRD data analyses.

4. Conclusion

In this study, nanostructured NiO films were synthesized successfully on glass substrate by using spray pyrolysis method. The influences of NiO molar concentration and substrate temperature on structural, morphological, and optical properties of the films were investigated. XRD analysis showed that a cubic polycrystalline NiO nanostructure films with preferred orientation along (111) plane were observed at all sprayed films and minimum crystallite size of 62.43nm was attained. The optimum condition for formation of NiO was found to be with NiO concentration of 0.2 M at 400 °C. The formed sample at this condition showed the best crystallinity structure. The optical studies showed the estimated band gap varies between 3.630 eV to 3.85 eV was infused by NiO molarity and substrate temperature. FESEM analysis revealed that the nanoparticles are extra-fine, with an average particle size of about 68.838 nm. The EDX analysis showed the elementals value of NiO is close to the stoichiometric ratio. From the obtained results of the present work, it can be suggested that the developed NiO nanostructured thin films is an excellent material promising for gas sensing applications.

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دروست کردنی باشترین توێژاڵی تەنکی ئۆکسیدی نیکل بەبەکارهێنانی تەنۆلکەی وردی شلە پرژێن

پوختە:

به بەکارهێنانی تەکنیکی بەگەرمی پرژاندنی کیمیایی، توێژاڵی تەنک لە هەریەک لە کلۆریدی نیکل وئاوێتەی ئاو (NiCl2.6H2O) پێکهێنانی گیراوەیەک بە ڕێژەی گیراوەی جیاواز وپلەی گەرمی جیاواز لەسەر بنکەی شووشە ئامادە کرا. لێکۆڵینەوە کرا لە سەر جۆری بلووری ومۆرفۆلۆجی وشیکردنەوەی ڕووی وسیفەتی بینایی بەهەرچوار تەکنیکی لادانی تیشکی ئێکس (XRD) ومایکرۆسکۆبی ڕوماڵکەری بواردەرکەر (FESEM) وپەرش بوونی وزەی تیشکی ئێکس (EDX) وسپێکترۆفۆتۆمەتەری UV-Visible (UV-Vis)، مایکرۆسکۆپی ئەلکترۆنی سکانکردنی فیلد-دەرچوون (FESEM). بە پێی ئەنجامەکانی شیکردنەوەی (XRD)، چڕی مۆلاریتی وپلەی گەرمی ماددەی کارتێکەر توێژاڵە تەنکە ئامادەکراوەکان باشترینن لە 0.2 مۆلاریتی و 400C° پلەی سیلیزی دا. ئەنجامەکەی ئێکس ئاڕ دی (XRD) نیشانی دا بۆ دروست کردنی توێژاڵی تەنکی پێکهێنراوی فرەشێوەبلووری پەسندکراو بە ئاڕاستەی درێژی (111) تەخت. شیکردنەوەی سپێکترۆفۆتۆمی UV–Vis نیشانی دا کە توێژاڵە تەنکە ئامادەکراوەکە گوێزەرەوەیەکی باشی ناوچەی بینراوە، وە کەلێنە وزەی دابڕاوی توێژاڵی تەنکی (NiO) لە 3.630 ئەلکترۆن-ڤۆڵت بۆ 3.85 ئەلکترۆن-ڤۆڵت دەگۆڕێت. شیکردنەوەی ڕووکەشی مۆرفۆلۆجی(SEM) دەریخست کە نانۆ گەردیلەکان زۆر باشن بە تێکڕای قەبارەی گەردیلەی (nm68.838). ڕێژەی ڕەگەزی پێکهاتەی ستۆیچیۆمتریکی (NiO) بە شیکردنەوەی (EDX) پەسەند کرا. ئەنجامە گشتیەکانی ئەم توێژینەوەیە پێشنیاری کرد کە توێژاڵە تەنکەکانی (NiO) کە بە کوالیتی بەرزی کریستاڵی دروست کراوە زۆر گونجاون بۆ ئامێرەکانی جێبەجێکردنی هەستکردن بە گاز.

تحسين وتوصيف أفلام أكسيد النيكل النانوية البنية النانوية المركبة للانحلال الحراري بالرش

الملخص:

باستخدام تقنية الرش الكيمياوي الحراري، تم تصنيع الأغشية الرقيقة ذات البنية النانوية البلورية من أوكسيد النيكل (NiO) على قواعد زجاجية بمولاريات مختلفة من محلول ملح كلوريد النيكل المائي (NiCl2.6H2O) عند درجات حرارة مختلفة للقاعدة. تم فحص توصيف أغشية NiO الرقيقة النانوية بواسطة مقياس حيود الأشعة السينية (XRD)، مقياس الطيف الضوئي بالأشعة فوق البنفسجية (UV-Vis)، المجهر الإلكتروني لمسح الانبعاث الميداني (FESEM)، ومطياف الأشعة السينية المشتت للطاقة. (EDX) بناءً على نتائج تحليل XRD ، تم تحديد افضل تركيزمولي ودرجة حرارة الركيزة للأغشية المحضرة لتكون 0.2 مولارتي و 400 درجة مئوية ، على التوالي. أظهرت نتائجلل XRD الأغشية ذات هيكل متعدد البلورات مكعبة مع اتجاه مفضل على طول مستوى (111). أظهر تحليل القياس الطيفي للأشعة المرئية وفوق البنفسجية الطبيعة الشفافة الجيدة في المنطقة المرئية، وتفاوتت فجوة النطاق لأغشية NiO من 3.630 الكترون-فولت الى 3.85 الكترون فولت. كشف تحليل البنية السطحي أن الجسيمات النانوية بالغة الدقة، بمتوسط حجم جسيمي (68.838) نانومتر. تم تأكيد نسبة التركيب العنصري المتكافئ لـ NiO عن طريق تحليل. EDX اقترحت النتائج الإجمالية لهذه الدراسة أن أغشية NiO الرقيقة ذات البنية النانوية البلورية عالية الجودة ستكون مناسبة للغاية لتطبيقات استشعار الغاز.