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Study of Physical Properties of Nanocrystalline NiO Thin Films Prepared by Spray Pyrolysis Technique

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ABSTRACT

In this paper a systematic study has been carried out on the influence of thickness on the physical properties of nanocrystalline nickel oxide (NiO) thin films prepared on glass substrate by spray pyrolysis method. The prepared nanocrystalline NiO films were characterized using X-ray diffraction technique (XRD), field emission scanning electron microscopy (FESEM) and optical measurement techniques. XRD patterns reveal the cubic structure for all the samples and the crystallite size varies with the thickness. FESEM images confirmed that all the films are homogeneous, without any crack, dense and exhibit almost complete coverage of the substrate. The optical parameters such as transmittance, absorption coefficient and energy band gap of the films as a function of film thickness was investigated by UV-Vis spectrophotometer. The band gap of the films is found to be direct allowed transition and the variation of band gap values of nanocrystalline NiO thin films were found to be in the range of 3.48 eV to 3.53 eV.

1. Introduction

Metal oxides have been studied extensively over the last few decades due to their actual and potential applications in the science and technology. Due to their interesting optical and electrical properties, nickel oxide (NiO) is one of the metal oxides which has been investigated for applications in sensors [1], UV photo-detector [2], in polymer bulk-heterojunction solar cells [3], organic light emitting diodes [4], photo anode for O_2 evolution [5], perovskite heterojunction solar cells [6] etc. NiO is a p-type, semitransparent and wide band gap transition metal oxide material widely used as a nontoxic material showing antiferromagnetic behavior [1, 7].

Numerous techniques have been employed for the preparation of good quality NiO thin films. These include chemical bath deposition [8, 9], SILAR [10], sol-gel dip coating [11], potentiostatic electrodeposition [12], spin coating [13], electro-hydrodynamic atomization (EHDA) [14], spray pyrolysis [7, 15-17], e-beam evaporation [18], radio frequency magnetron sputtering [19], metal organic chemical vapor deposition (MOCVD) [20].

Depending on the application of interest, many efforts have been conducted to obtain the thin films with desirable physical properties with the above-mentioned thin film deposition techniques. The low-cost spray pyrolysis technique is a simple and versatile method for the preparation of NiO thin films with good optical and electronic properties [17]. Large area coating, by spraying the precursor solutions along with the carrier gas or by spraying the mixture directly on the heated surface of the substrate, becomes possible by using the spray pyrolysis technique [16]. The present work is focused on the preparation of good quality nanocrystalline NiO thin films using simple and economical spray pyrolysis technique. The effect of thickness on the structural, morphological and optical properties of nanocrystalline NiO films have been investigated and presented systematically in this paper.

2. Experimental Methods

Nanocrystalline NiO thin films were prepared on commercially available microscopic glass substrates (25 mm × 75 mm ×1.35 mm) using simple spray pyrolysis technique. Prior to deposition, the glass substrates were cleaned by using soap solution followed by chromic acid, acetone and deionized water as per the standard procedure. Finally, cleaned glass substrates were dried in oven.

An appropriate quantity of nickel chloride hexahydrate ($NiCl_2.6H_2O$) (Loba Chemie,~99% purity) as a precursor of Ni and deionized water as a solvent were used as a source material for preparation of nanocrystalline NiO films using spray pyrolysis technique. NiO films were prepared by spraying a homogeneous mixture of aqueous solution with 0.01, 0.05, 0.10 and 0.15 M $NiCl_2.6H_2O$ using compressed air as a carrier gas through a glass nozzle of 0.1 mm bore diameter on the pre-cleaned and pre-heated amorphous glass substrates kept at 350 °C ± 5 °C. The distance between the nozzle and the substrate was 28 cm. Film thickness was measured by using the weight difference method considering the density of the bulk nickel oxide. The structural, morphological and optical properties of the prepared nanocrystalline NiO films were carried out using X-ray diffraction (XRD) method in the 2θ ranging between 20° to 80° using Bruker AXS, Germany (D8 Advanced) X-ray diffractometer, field emission scanning electron microscope (FESEM, S-4800 Type-II, Hitachi High Technology Corporation Tokyo, Japan) and JASCO UV-VIS spectrophotometer (V-630) respectively.

3. Results and Discussion

The variation of nanocrystalline NiO film thickness studied as a function of Ni concentration in order to study the growth kinetics of the prepared films. Fig. 1 shows the variation of NiO film thickness versus Ni concentration.

It has been found that thickness of the films increases as the Ni concentration is increased to 0.1 M and then it is decreased when Ni concentration is 0.15 M. This shows that the maximum thickness exhibited by nanocrystalline NiO films prepared with 0.1 M concentration of precursor solution.

The XRD spectra of nanocrystalline NiO thin films of different thickness prepared by using spray pyrolysis technique are shown in Fig. 2(a-d). The XRD patterns clearly showed the influence of the film thickness on the crystallinity of the nanocrystalline NiO films. For all NiO films, the cubic structure characterized with (1 1 1) plane as preferred orientation, are identified with the standard JCPDS data [JCPDS card No. 73-1519]. Similar type of XRD pattern were found by Belahssen et. al. [21] for undoped NiO

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films prepared by using nickel nitrate as a Ni precursor. As a result of increase in film thickness, there is change in the intensity of the peak corresponding to the 2θ ≈ 37.26° were observed. It is observed that the crystallite size increases from 16.31 nm to 18.48 nm as film thickness increases from 120 nm to 210 nm and again it decreases to 16.44 nm for film thickness of 205 nm.

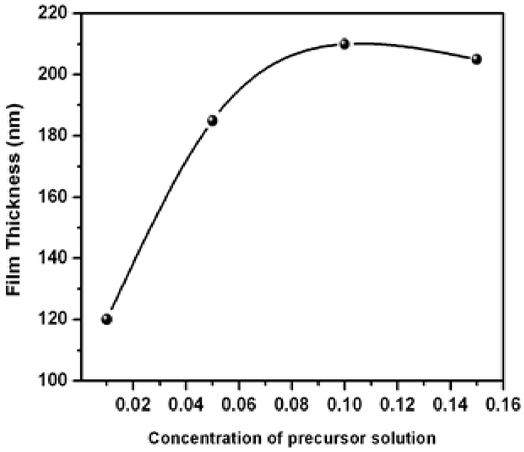

Fig. 1 Variation of NiO film thickness versus Ni concentration

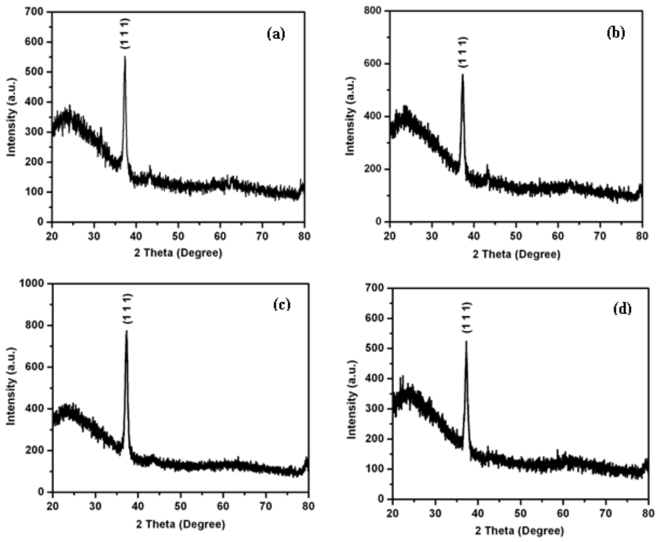

Fig. 2 XRD patterns of nanocrystalline NiO thin films deposited with different thickness (a)120 nm, (b) 185 nm, (c) 210 nm, (d) 205 nm

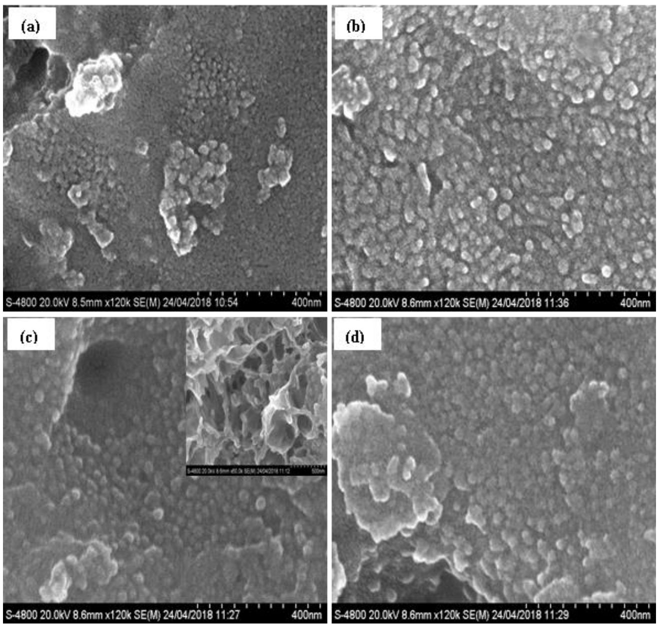

Fig. 3 FESEM images of nanocrystalline NiO thin films of different thickness (a)120 nm, (b) 185 nm, (c) 210 nm, (d) 205 nm

The topographical and elemental information of sprayed nanocrystalline NiO films was done by FESEM analysis. FESEM images of NiO films of different thickness are shown in Fig. 3(a-d). It can been seen that all the films are homogeneous and crack free. Also prepared material covering entire substrate surface area and microstructure consists of many round shaped grains, however there is a agllomeration in certain regions of the film which is clearly seen in the FESEM images. However porous surface morphology was observed in FESEM image of nanocrystalline NiO films with higher thickness as shown in inset of Fig. 3c. Such kind of morphology is very useful for gas sensing. The EDS spectra of nanocrystalline NiO thin films with higher thickness is as shown in Fig. 4. The analysis confirms the prsence of Ni and O elements with the ratio of 40:60. This confirms that the prepared NiO films is oxygen rich. It is very important to note that additional peaks attributed to Si are observed in EDS spectra which might be due to the glass substrate.

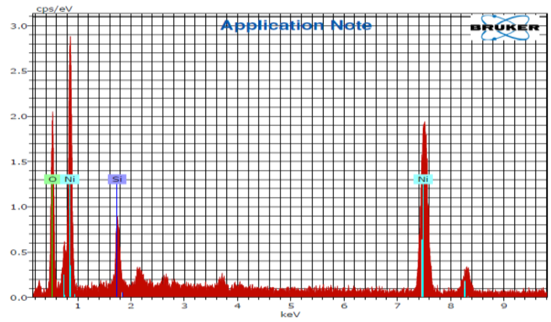

Fig. 4 EDS spectra of nanocrystalline nickel oxide thin films of thickness 210 nm

The optical transmittance and absorption spectra of the sprayed nanocrystalline NiO films was determined by UV-Vis spectrophotometer. The optical transmittance and absorption of the all samples are shown in Figs. 5 and 6 respectiely. From transmittance spectra it is observed that the prepared NiO thin films exhibit high trnasmittance, ranging between 65-80% in the near infrared wavelength region. Further, we found that the transmittance of NiO thin films is decreased with the increase of thickness upto 210 nm and for higher film thickness it is again increased. This is in good agreement with the XRD results where we have observed the change in crystallite size with the thickness. The absorption spectra were used to determine the band gap values of the nanocrystalline NiO thin films using the Tauc relationship [7, 22].

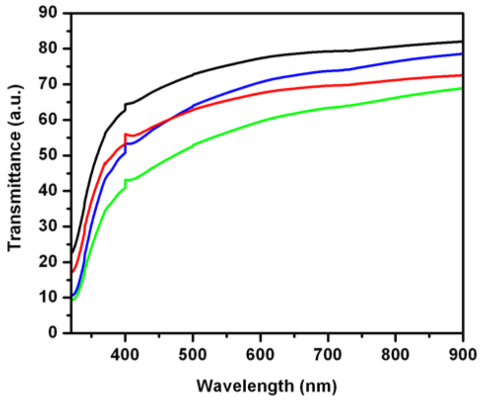

Fig. 5 Transmittance spectra for nanocrystalline NiO thin films of different thicknesses

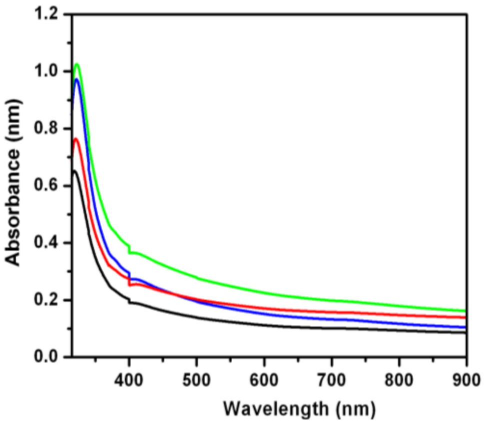

Fig. 6 Absorbance spectra for nanocrystalline NiO thin films of different thicknesses

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The band gap has been calculated by extrapolating the linear region of the plot of $(\alpha h\nu)^2$ versus hν on the energy axis. Fig. 7 shows typical plot of $(\alpha h\nu)^2$ versus hν for nanocrystalline NiO thin films with film thickness of 210 nm deposited using simple spray pyrolysis technique. The linear fit of the plot indicates the existence of the allowed direct band-gap transition. The band gap value of the NiO films is found to be 3.48 eV. The reported value of the band gap is in good agreement with the value reported previously [7]. Initially the band gap value is decreased with increase of thickness up to 210 nm and again increased for NiO film of 205 nm thickness.

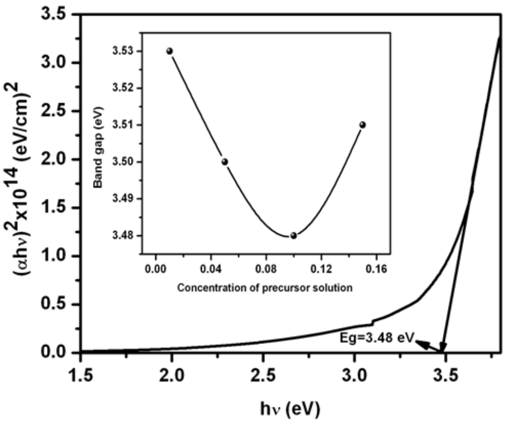

Fig. 7 Plot of $(\alpha h\upsilon)^2$ versus hυ for nanocrystalline NiO thin films with 210 nm thickness

4. Conclusion

NiO thin films with different thickness have been successfully deposited by the simple and cost-effective spray pyrolysis technique. Increase in the thickness of the NiO films increased the crystallinity as well as morphological properties. It is also observed from the transmittance spectra that for lesser thickness, the average transmittance in the visible region is found to be 80% and it varies with the film thickness. Direct band gap values of NiO thin films were found to in the range of 3.48 eV to 3.53 eV indicating that the properties of NiO films can be easily tailored by simply varying the film thickness.

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