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Influence of concentration on properties of spray deposited nickel oxide films for solar cells

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Abstract

Spray pyrolysis technique was used to deposit various concentration of nickel oxide films on glass substrate. The Effect of varying precursor concentration on elemental, morphological and structural properties was investigated on the deposited NiO films. Nickel (II) acetate tetrahydrate precursor was used at substrate temperature of 350 ^{o}C. Precursor concentrations were 0.025, 0.05, 0.075 and 0.1 M. Scanning Electron Microscope (SEM) surface morphology revealed nanostructured films with particles densely distributed across substrates surface. Increased in surface grains was observed as the precursor solution increased. Elemental composition of NiO films revealed presence of Ni and O element. There was reduction in oxygen concentration as precursor solution increases. Amorphous structure was observed at concentration of 0.025 M while polycrystalline with cubic structure was observed at higher concentrations. Preferred orientation was along (1 1 1) peak with small intensity along (2 0 0) peak. XRD patterns have peak diffraction at (2θ = 37 ^{o} and 43 ^{o}) for (1 1 1) and (2 0 0) planes respectively and 64 ^{o} for (2 2 0) plane for 0.1 M. Film thickness grew with increase in precursor concentration. Film micro strain was observed to have compression for all precursor solution conspicuously revealing the effect of varied concentration on NiO films properties.

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Keywords: NiO; solar cells material; annealing, low income

1. Introduction

About one-fourth of earth's inhabitants lacks access to electricity with little or no changes in absolute terms since 1970s (Ahuja & Tatsutani, 2009). Most developing countries still struggle with affordable stable electricity (Ebhota,

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Eloka-Eboka, & Inambao, 2014). Renewable energy especially solar energy is one of envisaged solution. Solar energy is one of the best sources of renewable energy. Hourly solar influx on earth surface surpasses annual human energy needs (Lewis, 2007). Solar energy is environmentally benign. About 40 % million tons of CO_2 emissions is saved per year when 1 % of world electricity demand is supplied by solar grid (Gardner, 2008). However, cost is militating against successful deployment of solar technology worldwide because, converting solar energy into electricity occurs at a price comparable with fossil fuel. Solar cells are integral part of solar energy (Green, 1982). Large scale production and affordable cost is still researched into in fabrications of solar cells (Eslamian, 2014). This is attributed to difficulty in scaling up existing methods or expensive nature and complexities associated with vacuum environment fabrication. However, nanostructure metal oxide offers promises. Nanostructures materials offers potential improvement on efficiency of photovoltaic (PV) solar cells, reduction in manufacturing and electricity production costs (Serrano, Rus, & Garcia-Martinez, 2009). It is achievable by increased surface area to volume ratio of nanoparticles. This enhances solar energy collection and efficiency by exposing more conducting surfaces to sunlight. Nanostructures materials have unique characteristics that cannot be obtained from conventional macroscopic materials (Hussein, 2015). Conventional materials have weaknesses in the absorption properties of the conventional fluids which can lead to reduced efficiency of solar cells devices. Inorganic semiconducting materials are economical, environmentally friendly and viable sources for solar cells (Joshi, Mudigere, Krishnamurthy, & Shekar, 2014). In recent years, fabrication of nanostructured metal oxide films is attracting interest in terms of technological applications (Drevet et al., 2015; Rahal, Benhaoua, Jlassi, & Benhaoua, 2015; Shaikh, Inamdar, Ganbavle, & Rajpure, 2016; Zhang et al., 2006). They have been studied due to their vast usage (Soonmin, 2016). They have found applications in solar cells, UV detectors, electrochromic devices, anti-ferromagnetic layers, p-type transparent conductive thin films and chemical sensors (Li & Zhao, 2010; Magaña, Acosta, Martínez, & Ortega, 2006; Nam et al., 2015; Park, Sun, Sun, Jing, & Wang, 2013; Wu & Yang, 2015; Zhu et al., 2014). Nanostructured metal oxides often express n-type conductivity with few displaying p-type. Nickel Oxide (NiO) is a p-type semiconductor with wide band gap from 3.5 to 4.0 eV (Boschloo & Hagfeldt, 2001). Nickel oxides exist in various oxidation states (Subramanian et al., 2008). NiO has rhombohedral or cubic structure and possesses pale green color. NiO have excellent durability and electrochemical stability with a large range of optical densities. It is a promising material for various applications because of its better optical, electrical and magnetic properties. Nickel oxide thin films have been deposited using different methods; sputtering (Keraudy et al., 2015), sol–gel (Jlassi, Sta, Hajji, & Ezzaouia, 2014), electron beam deposition (El-Nahass, Emam-Ismail, & El-Hagary, 2015), laser ablation (Wang, Wang, & Wang, 2012), chemical bath deposition (Vidales-Hurtado & Mendoza-Galván, 2008). Spray Pyrolysis Technique is simple, low cost and feasible for mass production (Ismail, Ghafori, & Kadhim, 2013). Spray Pyrolysis is method that allows coating on large area by films of very thin layers with uniform thickness (Gowthami, Perumal, Sivakumar, & Sanjeeviraja, 2014). This study aims to optimize the precursor concentration of NiO films with motivation for efficient and affordable application in solar cells development. The scope involves: the preparation of a nanostructured NiO thin films on a glass substrate using SPT for deposition of aqueous solution of nickel (II) acetate tetrahydrate and determine the effect of varying the concentration on different properties of NiO films.

2. Experimental Procedure

2.1. Spray Pyrolysis set up

Experimental setup for spray pyrolysis used is shown in Figure 1. The set up consists of heater, air compressor, temperature controller, exhaust fan and pipe, spray gun with attached container. The container was used to hold the precursor solution. Spray gun was connected to the air compressor using hose or pipe. Temperature of 350 ^{o}C was attained and read by thermocouple attached to the heater before commencing deposition. The carrier gas is compressed air at pressure of 1bar.

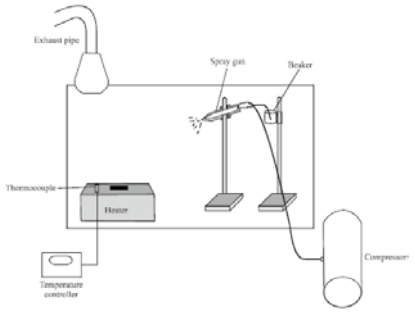

Fig 1: Experimental set-up of spray pyrolysis technique

2.2. Precursor preparation and deposition

Concentration of 0.025, 0.05, 0.075 and 0.1 M of nickel (II) acetate tetrahydrate ($\mathrm{Ni}(C_2H_3O_2)_2.4H_2O$) (Medicine, 2007) were used as precursor solution. It was dissolved in 50 mL distilled water and stirred for 10 minutes. The precursor solution was poured into spray gun container. Glass substrate was chemically and ultrasonically cleaned and thereafter heated at constant temperature of 350 ^oC on a heater. Other deposition parameters were maintained to obtain uniform film thickness. Optimum deposition parameters of spray deposited NiO films are shown in Table 1. Each droplet from the spray gun was less than micro sized particles. Sprayed solution on the preheated substrate glass undergoes evaporation. Solute precipitation and pyrolytic decomposition are as shown in Equation (1). The major end product is nickel oxide thin films.

$$\mathrm{Ni}(C_2H_3O_2)_2.4H_2O \xrightarrow{heat} NiO + 7H_2O \uparrow + 4CO_2 \uparrow \quad (1)$$

Colour of prepared thin films was observed to be gray, uniform and strongly adhered to glass substrate. Thermocouple was fixed to substrate's surface to record the temperature.

Table 1. Optimum deposition parameter of SPT NiO films

Deposition parameter	Value
Substrate temperature	350 ^oC
Height of spraying nozzle to substrate distance	20 cm
Spray rate	1 ml/min
Spray time	1 minute
Time between sprays	30 seconds
Carrier gas	Filled compressed air of 1bar

2.3. Characterization

Morphology of deposited NiO film was studied using ZEISS ULTRA PLUS Field Emission Gun Scanning Electron Microscope (FEGSEM). Elemental composition was done with Energy Dispersive X-ray Spectrometer (EDS or EDX: "AZTEC OXFORD DETECTOR"). Structural properties of deposited NiO films were investigated using EMPYREAN (PANalytical) X-ray powder diffractometer for a range of 5 º to 90 º 2θ angles.

3. Results and Discussion

3.1. Morphological studies

Figures 2 and 3 show the FEG SEM micrographs. It reveals homogeneous, smooth, well adherent films devoid of pinholes and cracks. It becomes grainier with bigger flakes as precursor concentration increased from 0.025 M to 0.1 M. This is an improvement on results observed by (Bari, Patil, & Bari, 2013; Saadati, Grayeli, & Savaloni, 2010).

This confirms that varying the concentration of the precursors affects the NiO films morphology.

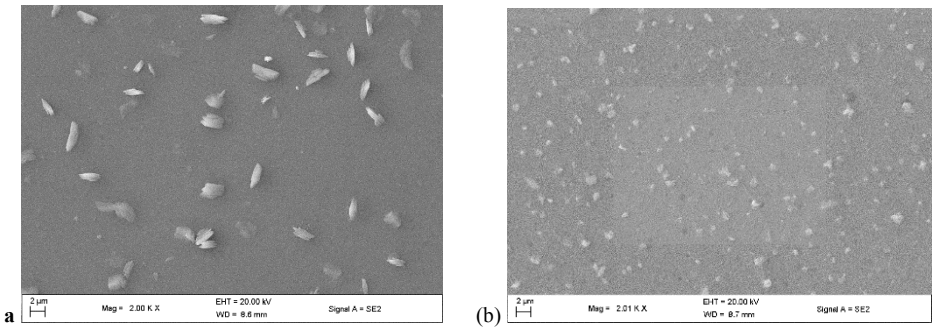

Fig 2: SEM micrographs of nickel oxide (NiO) film on glass substrate at (a) 0.025 M and (b) 0.05 M

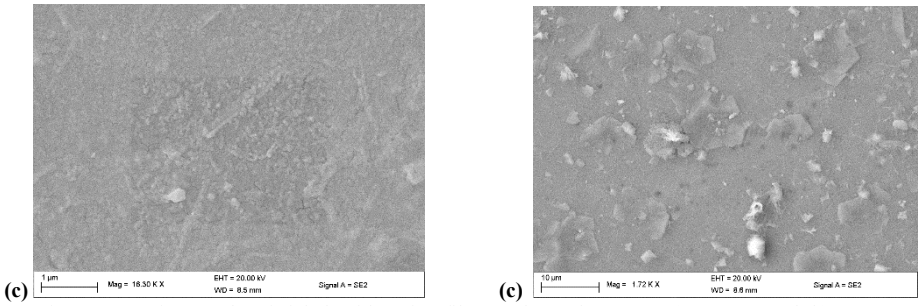

Fig 3: SEM micrographs of nickel oxide (NiO) film on glass substrate at (a) 0.075 M and (b) 0.1 M

3.2. Elemental composition Analysis

Figure 4 shows the EDX for the different concentration of the NiO thin films thereby confirming presence of Ni and O elements in NiO thin films. There was reduction in oxygen concentration in the deposited NiO films as precursor concentration increased as seen in Figure 4. This may be due to increase in film growth on the glass substrate thereby making less of the glass (oxygen) to be seen. Reguig et al. (Reguig et al., 2006) also reported presence of Ni and O elements. Additional Si element was also observed. This is because Silicon (Si) is present in soda-lime glass or soda-lime-silica glass substrate (de Jong, 1989).

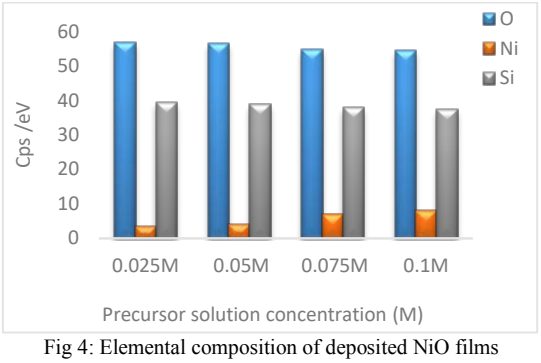

Fig 4: Elemental composition of deposited NiO films

3.3. Variation of the film thicknesses with precursor solution concentration

Films thickness was investigated as a function of the precursor concentration ranging between 0.025 M and 0.1 M. The measured data are graphically represented in Figure 5. Using the weight difference method, film thickness was calculated using the relationship in Equation (2) (Godse et al., 2011):

$$t = m/A \quad (2)$$

Where t denotes the film thickness while m is the actual mass deposited onto the substrate, A is the area of the film and ρ is the density of material.

The calculated film thickness is shown in Figure 6. From Figures 5 and 6, it was seen that the measured and calculated values are in good agreement. It was found that film thickness grew with increasing precursor concentration except for 0.025 M. This is an improvement of results by Boyraz and Urfa (Boyraz & Urfa, 2015). This is as result of accumulation of deposited NiO on substrate. This was collaborated by EDX results in Figure 4. The kinetics of the NiO forming reaction increased with precursor concentration. During the deposition, nozzle to substrate height and the deposition time were kept constant to control the thickness of NiO thin films. Average thickness range of the NiO thin films was found between 6.277 and 11.57 µm.

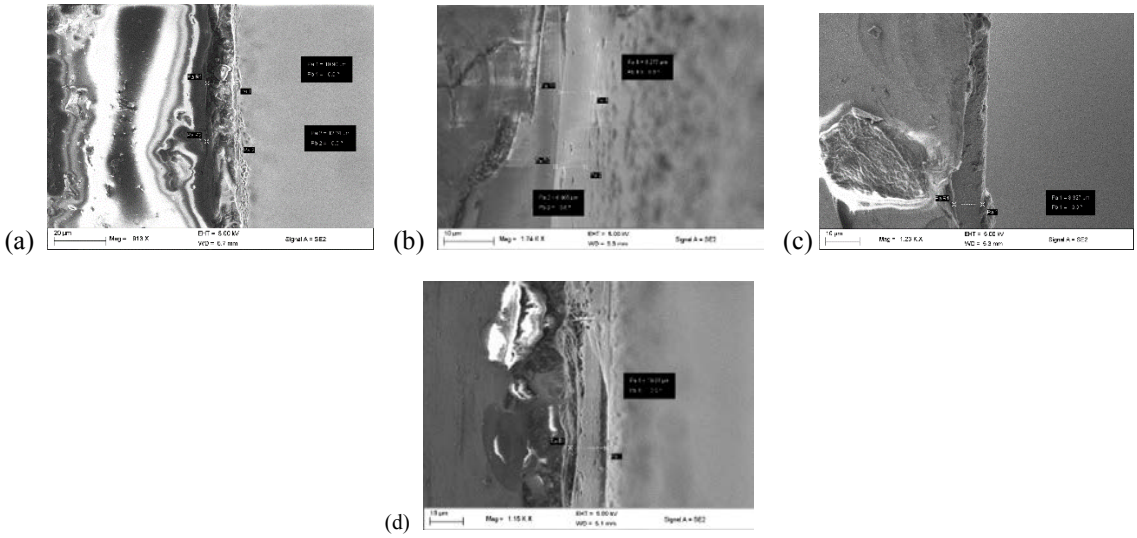

Fig. 5. Measured NiO film thickness at (a) 0.025 M; (b) 0.05 M; (c) 0.075M; (d) 0.1M

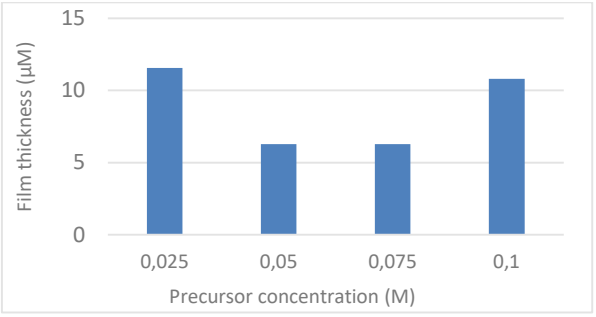

Figure 6: Calculated film thickness of NiO films

3.4. Structural studies

The phase present and preferred orientation of deposited nanostructured NiO films was determined using X-ray diffractometer (XRD). Figure 7 shows the XRD patterns of deposited nanostructured NiO films at different precursor concentration. The patterns have peak diffraction at (2θ = 37 º, and 43 º) for (1 1 1) and (2 0 0) planes respectively and 64 º for (2 2 0) plane for 0.1 M. This agrees with Joint Committee on Powder Diffraction Standards—International

Centre for Diffraction Data, JCPDS 04- 0835 for Bunsenite (NiO) (Gabal, 2003). Highest intensity was recorded for (1 1 1) having a strong peak when 2θ = 37 º for precursor solution of 0.05 M, 0.075 M and 0.1 M which is equal to (Bakr, Salman, & Shano, 2015). This maybe as a result of increase in grain growth caused by larger film thickness. It can also be due to increase in crystallinity as precursor solution concentration increases; thereby confirming polycrystalline with cubic crystalline structures of deposited NiO films similar to reported structure by Fadheela (2015). Lower intensity peak of (2 0 0) increases gradually as precursor solution increased from 0.05 M to 0.1 M with emergence of third peak (2 2 0) for 0.1 M. Average crystallite size was obtained using Debye Scherer formula (Barrett & Massalski, 1980; Scherrer & Nachr, 1918) in Equation (3) as shown in the following section.

$$D = \frac{k\lambda}{\beta \cos\theta} \quad (3)$$

Where; B denotes full width at half maximum (FWHM) intensity of the peak (in Radian), λ is wavelength, θ is Bragg's diffraction angle and k is 0.89 respectively. Grain size for (1 1 1) and (2 0 0) planes are found to be 22 nm and 63.77 nm. Lattice constant was found to be 4.1905, 4.1856, 4.1852, 4.1850 Å for 0.025 M to 0.1 M respectively. This agrees with standard lattice constant of NiO film value of 4.176 Å (Pistorius, 1963).

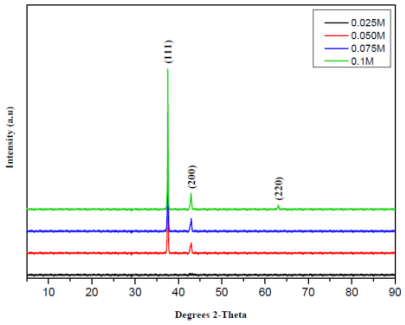

Fig 7: XRD patterns of nanostructured NiO films at different precursor concentration

Micro strain was produced through growth of thin films and was calculated using the formula in Equation (4) (AL-Jabiry, 2006).

$$\delta = (d\_(ASTM) - d\_XRD)/d\_ASTM \times 100 \quad (4)$$

Where "d" is the lattice constant and δ is micro strain.

A plot of NiO film micro strain against precursor solution is shown in Figure 8. It shows that there is an increase in micro strain as precursor concentration increases. Micro strain represents compression as seen in Table 2 which gives detail result of micro strain, lattice constants and 2θ values for deposited NiO films for precursor solution concentration of 0.025 M to 0.1 M.

Table 2: Calculated parameters from XRD data

Parameter		0.025 M	0.05 M	0.075 M	0.1 M
2θ	hkl		37	37	37
	(1 1 1)				
	(2 0 0)	x	43	43	43
	(2 2 0)	x	X	x	63
Lattice constant d (Å)	recorded XRD	4.1905	4.1855	4.1852	4.1850
	ASTM	4.1684	4.1684	4.1684	4.1684
Micro strain (δ) %		-0.5301	-0.4102	-0.4030	-0.3982

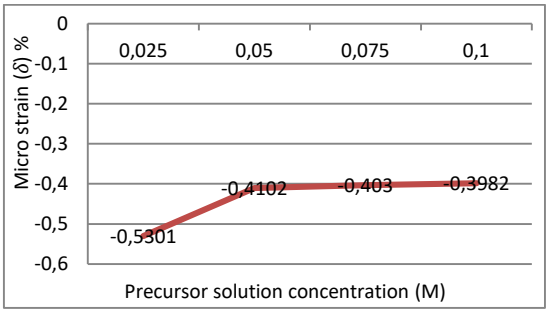

Fig. 8. Plot of Micro strain against precursor solution concentration for NiO films

4. Conclusion

In this study, nanostructured nickel oxide films were successfully deposited by spray pyrolysis of nickel (II) acetate tetrahydrate on glass substrate. The effect of varying precursor concentration of NiO films on elemental, morphological and structural properties were studied with a view to optimizing the material for solar cells application. This study contributed new results relating to surface morphology, structural, film thickness and micro strain of NiO films using SPT. The results clearly showed that varying the precursor solution concentration has effect on the morphological and elemental properties of nickel oxide thin films. The surface morphology is improved by increasing precursor solution concentration. Film thickness is improved as precursor solution concentration increases. Oxygen concentration reduces as precursor concentration decreases.

There is mark improvement on crystallinity with increasing precursor solution concentration. Leading to higher peak intensity and diffraction. New Peak diffraction was recorded at ($2\theta = 37^{o}$, and 43^{o}) for (1 1 1) and (2 0 0) planes for 0.05 M concentration and above and 64^{o} for (2 2 0) plane for 0.1 M. Lattice constant decreases from 4.1905 to 4.1850 Å for 0.025 M to 0.1 M which correlate 4.176 Å standard lattice constant of NiO. Micro strain of films shows compression and increases with precursor concentration.

Varying concentration of precursor solution has effect on overall properties of nanostructured nickel oxide thin films. Precursor solution 0.1 M outperformed others by showing good crystallinity and good film thickness. Therefore, NiO films from 0.1 M concentration can be further explored for solar cells application.

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