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Thin Film Technology

Elixir Thin Film Tech. 82 (2015) 32620-32623

ISSN: 2229-712X

Sensing and Electrical Variation of Nickel Oxide Synthesis by RF-Reactive Sputtering

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ARTICLE INFO

Article history:

Received: 27 March 2015;

Received in revised form:

16 May 2015;

Accepted: 18 May 2015;

Keywords

Reactive Sputter,
Nickel Oxide,
Nitrogen Dioxide,
Hydrogen.

ABSTRACT

In this paper we synthesis various thickness of nickel oxide (NiO) nanostructure films deposited on si substrate. by using RF. Reactive magnetron sputtering technique rotating by 5 cycle/min with both Argon and Oxygen mixture gases .argon and oxygen partial pressure was(7.0×10^{-3}torr)and(1.89×10^{-2}torr) respectively. The gas sensing application and electrical properties investigated as a function to the thickness variation the dramatic change in the electrical properties shows strong dependence on the thickness variation. Conductivity, resistivity, gas sensitivity of hydrogen and nitrogen dioxide gases was (95 %) and (90 %) for 50 nm respectively, gas sensitivity for NiO films increasing as film thickness increasing.

Introduction

Nickel oxide can be formed with phases such as, Nickel (II) oxide (NiO) green, Nickel (III) oxide (Ni2O3) black. Nickel oxide (NiO) has excellent characteristic semiconductor material due to high electrical conductivity and chemical stability; it has advantage of low cast and available [1]. NiO can plays active role in may technological applications like solar cell, high sensitive gas sensing[2].p-type transparent conductive films[3].electro films[4] , NiO nanostructure(0-D), (1-D)and(2-D) can enhancing application strongly due increasing in the surface area and quantum confinement effects[5]. Metal oxide semiconductor sensors one of the most lowest cost for monitoring the emission of small amount of gases in the human activity gas sensors detection is enabled by a change in the electric resistivity through the adsorption of the target gas on to the surface of the semiconductor which allowing as to measure the target gas concentration it is important to gas sensor to have high sensitivity, short responds time. Nitrogen oxide gas (NOx) can be formed as (nitric oxide) NO, (nitrogen dioxide) NO2.(Nitrous oxide) N_2O, (dinitrogen trioxide) N2O3. Nitrogen dioxide is toxic gas inhalation it is easily can be detected by smell the mien risk of nitrogen dioxide appear to be effect on lunges. [6]. Hydrogen gas is very flammable and will burn in air at very wide range of concentration between (4% - 74%) by volume Hydrogen can react spontaneously and violently at room temperature with chlorine and fluorine to form the corresponding hydrogen halides, hydrogen chloride and hydrogen fluoride, which are also potentially dangerous acids. hydrogen forms with different phases compressed hydrogen, liquid hydrogen, slush hydrogen, solid hydrogen and metallic hydrogen [7]. NiO growing techniques can be chive by many ways such as vacuum evaporation, electron beam evaporation, DC and RF-magnetron sputtering, chemical deposition, sol -gel and spray pyrolysis techniques. [4, 5]. Film properties prepared by each technique depend on the deposition parameter then we can a jest the parameters to suitable the desired applications. one of the fine and delight technique is RF-sputtering because of the high control to the various parameters (pressure, gas mass full controller ,distance between the target and the substrate ,the angel between the target and the substrate surface , deposition rate controller ,argon and reactive gas partial pressure control ,temperature ,RF-power source leveling) all this parameters can be used to achieve any proper film for large range application. [10].Hao and et conclude that crystalline size increase as the thickness increase and the resistivity of the film increase

As the thickness increase. Young and et reported that the crystalinity and the resistivity of the film increase with the power increases A.Mallikarjuna and et all found transmittals and the band gab of the film was increase with increase in annealing temperature up to 573 K Hydrogen gas is an attractive candidate for future full and energy source, the concentration monitoring is very important for application of full cell as well as for the cases that hydrogen being an undesirable contamination

Experimental

TORR INTERNATIONAL, INC.CRC600 systematic system used in this work to deposits nickel oxide nanostructure film on glass substrate (Supe Rior w. Germany) by RF-reactive magnetron sputtering. The power chosen to be (200 W) which splayed by (SEREN, industrial power system) .the deposition rate and thickness controlled and measured by crystal sensor (FTM-2000) pure nickel target with purity (99.94) made by TORR INTERNATIONAL) the target dimension was (diameter =50 mm) (thickness = 3mm).

The ultimate pressure before sputtering was (6.77×10-6 torr) witch done by combination of molecular devotion and turbo. And the partial pressure for argon and oxygen was (9.43×10-3 torr) and (1.87×10-2 torr) respectively. Substrate rotating base used to rotate the substrate (5 cycle / min).the distance between the target and the substrate was (100 mm) and the angel between the target and substrate surface was (θ =55°).the chamber temperature kept around (T=150 C°).

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Film characterization

The crystal structure of deposited films was identified by the X-ray diffraction (XRD) (shimadzo 6-2006, with cukα radiation having Wave length λ=0.15406 nm) technique. The electrical properties including resistivity, done by Hall measurements in van der Pauw geometry the. Sensing test Dane by (physics department, College of Science Almustansiryh University).

Result and Dissection

Structural properties

Fig. (1) shows X-ray diffraction pattern of NiO thin film for different thickness deposited by RF- sputtering at 200 watt power (13.5 MHz) the crystalline structure and preferred orientation and crystalline size identified as 22.894 nm for 50 nm thickness and increases to 25.036 nm for 150 nm thickness and listed in table (1).

Table (1) Comparison between the lattice constant of NiO films deposited by RF-reactive sputtering and standard NiO values of ASTM stander card (NO.047-1049).

Thickness (nm)	(hkl)	FWHM	2θ		I/I0		d(A0)		Crystalline size L (nm)	Lattice Constant (nm)
			XRD	ASTM	XRD	ASTM	XRD	ASTM		
50	(200)	0.374	43.31	43.27	100	100	0.2087	0.2089	22.894	0.4174
100	(200)	0.350	42.98	43.27	100	100	0.2102	0.2089	24.438	0.4204
150	(200)	0.342	43.35	43.27	100	100	0.2085	0.2089	25.036	0.4170

All samples shows single crystallite of cubic phase (ffc) with strong reflection along (200) plane at 2θ=43.31°, 2 thaita and d (interplaner distance) shows very will agreement with ASTM stander card (NO.047-1049). The peak intensity increases with film thickness increases may be due to an increase in the crystallite size [12].also (2 thaita) position shifted to shorter 2 thaita due to microwstrian occur inside the film [11]. Crystalline size (grain size) of the preferred reflection peak determine by Debye-Scherer formula (1).

$$L = \frac{K\lambda}{\beta \cos\theta} \quad (nm) \qquad (1)$$

Where (λ) is the wave length of cupper X-ray source is equal to (1.5406 Å), β is FWHM in radian and θ is Braggs diffraction angel of (200) peak, the grain size increases from (22.894 nm) to (25.036 nm) as the thickness increases from (50 nm) to (150 nm)[12].

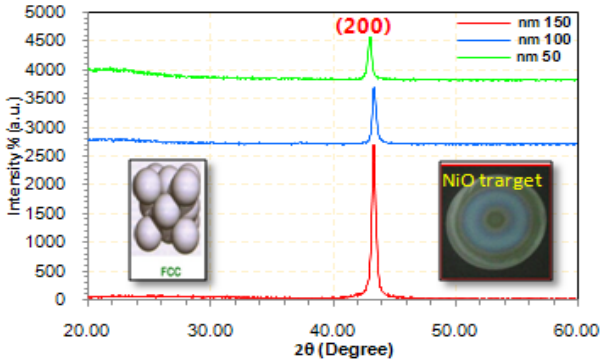

Fig 1. X-Ray diffraction pattern of NiO thin films deposited by RF-reactive sputtering

Electrical properties

The electrical resistivity of NiO thin films shows strong correlation to microstructure. Defects existing in crystal texture especially oxygen vacancy and defects [13]. Hall measurement revealed p-type NiO films at room temperature the little values of electric resistivity of prepared NiO films that compare to the bulk one is due to high crystallite of the film, electrical resistivity increases from (260.9 KΩ) to(550.14 MΩ) at 35 C° as the thickness of the film increases from (50 nm) to (150 nm) that could be explained as thickness increases led to imperfection or non stoichiometry of the NiO films[14]. While raising the temperature shows decrease in the resistance which revels semiconductor nature of the prepared film [15] Fig. (2) Shows the variation of the resistivity with temperature for the different prepared NiO films.

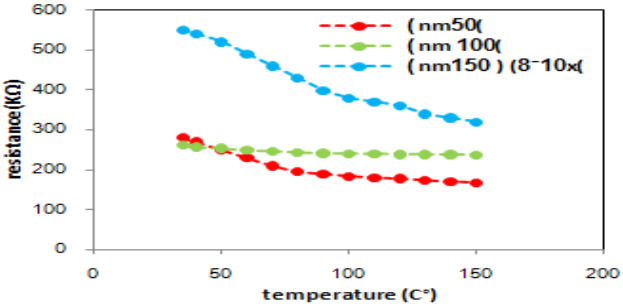

Fig 2. Electrical resistance of NiO thin films as function of temperature as deposited by RF-reactive sputtering

Sensing properties

In this study we use NiO thin film as gas sensor tester with different thickness (50,100,150) nm, to test nitrogen dioxide (NO2) and hydrogen (H2) gases with various concentration (150,200,250,300,350) ppm. At operating temperature is (150 C°). From Fig. (3- a&b), which represent the variation of resistance (KΩ) as a function to time(sec) for different thickness, from Fig(3-a),we can observe the increases of resistance of the NiO film as hydrogen gas in traduce to the chamber which Indicate that NiO is p-type semiconductor which (confirms hall measurement), the reduced gas (H2) react with the film surface to form water vapor and electrons transfer to the film (to conduction band) that's occur recombination of electrons and holls which represent the majority charge carriers in the p-type semiconductor that led to decreases in the number of majority of charge carrier then the resistance Of the film increases[16]. While the increases in the gas concentration from (150) to (350) ppm by 50ppm step for each case shows increases in the resistance differ (ΔR) (the difference between the gas resistance in the gas presence (Rg) and gas resistance in the air (Ra)).

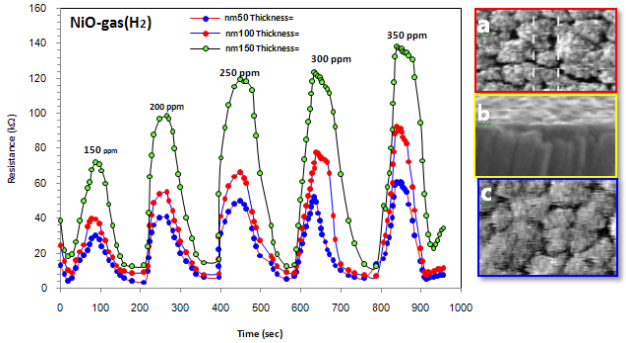

Fig 3a.resistance of NiO thin films deposited by RF-reactive sputtering varying with time and for different thickness and at operating temperature (150C°) .(a)50 nm thickness top view (b)100 nm thickness cross section view(c)150 nm top view of SEM (surface electron morphology)

Fig.(3-b),we can notes opposites behave of NiO film with oxidizing gas (NO_2),the resistance of the film decreases as the gas pumped in to the chamber, the oxidized gas capture electrons from conduction band introduce and that increases holls numbers in the conduction band which decreases the resistance of the film[17]. NiO (50) nm has the lowest resistance as the concentration increases that could led to syntheses high sensitivity sensor. Fig.(4 – a & b),represent the variation of sensitivity(S%) of NiO thin film as a function to (NO_2) and (H_2) gases concentration for different thickness at operating temperature (150C°) the sensitivity can defined by the resistance differ (ΔR) (the difference between the gas resistance in the gas presence(R_g) and gas in the air atmosphere (R_a) divided by the resistance of the gas(R_g),for p-type semiconductor react with oxidation gas[18].

$$S = \frac{(R_g - R_a)}{(R_g)} \qquad (2)$$

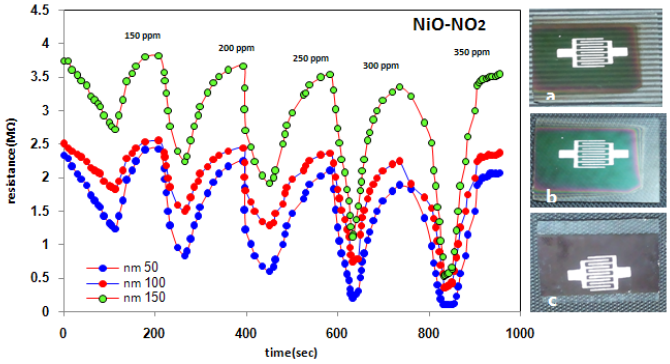

Fig 3b and Fig 3a resistance of NiO thin films deposited by RF-reactive sputtering varying with time and for different thickness and at operating temperature (150C°)(a)50 nm(b)100 nm (c)150 nm

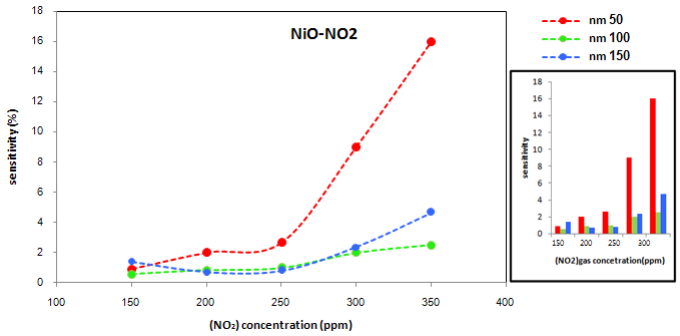

Fig 4a. Sensitivity of NiO thin films with different NO_2 gas concentration and thickness variation as deposited by RF-reactive sputtering and at operating temperature (150C°)

Fig.(4-a) , clearly shows that the sensitivity of NiO films p-type semiconductor increases as (NO_2) gas increases from (150 ppm) to (350 ppm) due to moor majority charge carrier (holes) generated in the conduction band as oxygen ions extracts electrons and also the conductance mechanism in metal oxide semiconductor is strongly related to the nickel vacancies and interstitial oxygen existing in the microstructure[19] also we observe that the sensitivity for the smallest thickness is higher than other thickness due to small grain crystalline size as calculated in scherr equation Fig.(4-b).describe to sensitivity of NiO thin film as deposited by different thickness for reduced gas (H_2) testing operating at room temperature , the sensitivity of the NiO film with oxidizing gas formed as the difference between film resistance in the air atmosphere (R_a) and in the gas presence (R_g)).divided by the resistance of gas resistance (R_g).equ.(2).[18],the sensitivity increase as the gas concentration is increases due to interact the reduced gas (H_2) with adsorbed oxygen at the film surface producing (H_2O) vapor and free electrons injected to the film, recombination operations between holls and electrons rising the resistivity of the film[20]. For (100,150) nm the sensitivity increases less than the (50)nm NiO film as the gas concentration increases due to the small grain size of the film which increase the surface volume ratio that increases adsorption action sites for oxygen or hydrogen molecules increases[21].

$$S = \frac{(R_a - R_g)}{(R_a)} \qquad (3)$$

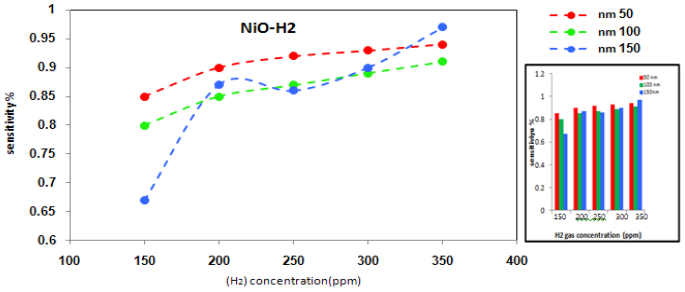

Fig 4b. Sensitivity of NiO thin films with different H_2 gas concentration and thickness variation as deposited by RF-reactive sputtering and at operating temperature (150C°).

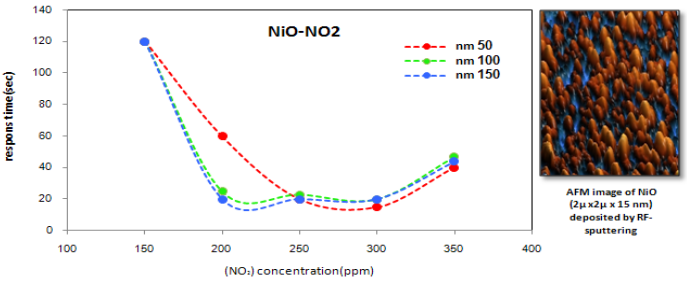

Fig 5a. Response time of NiO films deposited by RF-reactive sputtering to NO_2 gas with deferent concentration and at operating temperature (150 C°)

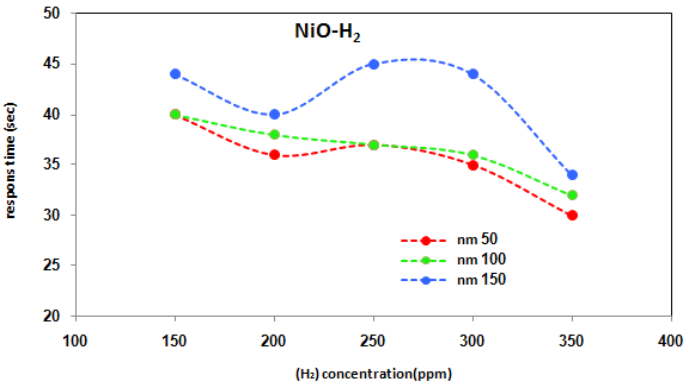

Fig 5b. Response time of NiO films deposited by RF-reactive sputtering to H_2 gas with deferent concentration and at operating temperature (150 C°)

In Fig.(5 a&b) The response time which we can defined it as (R_g\R_a), placed as variable to gas concentration at operating temperature (30C°) and for three different thickness (50,100,150) nm, Fig.(5-a) shows that response time of NiO thin film for NO_2 gas is decreases from (120 sec) to (20 sec) as the gas concentration is increases from (150 ppm) to (200 ppm) caused by increasing of injected electrons in conduction band and at the high gas concentration we can see response time increases with concentration increases[17] in this case we find all NiO films(50 ,100,150) have variation in response The response time for reduced gas (NO_2) and in Fig.(5-b)we fiend the response time decreases as the gas concentration increases

and the film with the smallest thickness have the lowest response time due to smooth surface with roughness (3.028 nm)and small grains confirm by atomic force micrograph(AFM) and X-ray diffraction (XRD)

Conclusions

In this work the researcher grub finish successful deposit NiO nanopartical film by RF-sputtering. Grain size determine by scherr equation shows very small grains (L=29.33 nm) and AFM measurement observe so smooth surface with roughness (average roughness) = 3.028 nm what led the NiO sensing both oxide and reduced gases with sensitivity around (90%) and at operating temperature (150 Cº).what makes this sensor very sotebul to work in stander atmosphere also we can rise the sensing efficiency by rising operating temperature and/or the gas concentration.

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