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Effect of Ethanolamine and HCl on structural and optical properties of Nickel oxide thin films

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Abstract: In the present work, Nickel Oxide thin films were prepared by spin coating technique. The effects of Ethanolamine and HCl on structural and optical properties of NiO thin films are studied. The structural properties of NiO thin films were examined by X-ray Diffraction (XRD). The amorphous nature of NiO thin films were observed from XRD pattern. FESEM studies of the surface morphology of NiO thin films exhibits smooth and uniform surface with average particle size of 200 nm for Ethanolamine doping sample and 300 nm for HCl doping sample. The functional groups and surface roughness of NiO thin films were investigated by FTIR and 3 D Laser Profilometry. The surface roughness of the prepared samples increases with increasing thickness from $4.49 \,\mu$ m to $6.81 \,\mu$ m. The optical characteristics of the samples were determined by UV Visible Spectrophotometer. This results shows that the prepared NiO thin films exhibits high optical transparency.

Keywords: Spin coating, Nickel oxide, Ethanolamine, HCl, XRD, FESEM.

Introduction

The researchers on thin films had led to a conclusion that every element in the periodic table has its own interest for different application. The researchers on nickel oxide thin films have been widely grown due to their various applications in all areas of science and technology. NiO thin films can be used for many applications including electrochromic and ion storage layer electrochromic devices, gas sensors and p-type semiconductor devices because of their wide band gap energy [1,2]. NiO can be used for self cleaning surface application to produce solar cells [3] and automotive glass[4].

Several methods have been used to prepare NiO thin films include spray pyrolysis[5,6],dip coating method [7,8] chemical vapor deposition, pulsed laser sputtering and

spin coating techniques [9,17]etc. Spin coating technique is the most important and commonly used technique among the various deposition techniques to prepare NiO thin films due to its ability to achieve dense, smooth and uniform coatings [10]. Duration and speed of the spin, viscosity and surface tension of sol, volatility of the solvent used in the formation of sol are the important parameters to achieve dense, smooth and uniform coatings.

The present work focused on the effect of Ethanolamine and HCl on structural and optical properties of Nickel oxide thin films.

Materials and methods Materials

Nickel (II) acetate tetrahydrate (≥98%), 2- Methoxy Ethanol (98%), Ethanolamine (98%) were used to prepare precursor solution. HCl GR (> 40% purity- Merck), HNO³ GR (>60% purity- Merck), distilled water and Extran were used to clean the microscopic glass slides. Glass substrates used for film coating are blue star glass slides with 25mm × 25 mm × 1mm dimension. The chemicals are in analytic grade so we used without any refinement.

Sample preparation

In this work, two samples were prepared Sol A and Sol B. The solution A (Sol A) was prepared by adding 0.125g Nickel acetate tetrahydrate with 20ml 2-Methoxy ethanol and stirred at 40 °C for 30 minutes. 0.15 ml Ethanolamine is added drop wise to the above solution and stirred at 50 °C for two hours.

The solution B (Sol B) was prepared by dissolving 0.9g Nickel acetate tetrahydrate with 25ml 2-Methoxy ethanol and added 0.1 ml of Hydrochloric acid to this solution and stirred at 50°C for one and half hours.

The prepared solution was deposited on the substrate surface by the help of spin coating unit (Apex instruments SCU2007A). The prepared solution of Nickel acetate tetrahydrate with Amine and HCl was deposited at the speed of 800 rpm for 30 seconds and 600 rpm for 45 seconds respectively. After coating the solutions on the substrates, the substrates were dried well by annealing at 120 °C with a rise in temperature of 1° C/min.

Characterization techniques

Synthesized samples were characterized by XRD, FESEM, FTIR, 3 D Laser Profilometry and UV- Visible spectroscopy. XRD (X Pert Pro Panalytical) analysis confirms the structural properties of the samples [11]. The morphological characterization was also performed using FESEM (Zeiss Sigma) analysis. The surface roughness of NiO thin films were obtained using 3 D Laser Profilometry (Zeta 20 apparatus). The FTIR (Shimadzu apparatus) is an analytic method to find the functional groups [12, 13] .Optical transmittance measurements were performed using UV Visible spectrophotometer (Jasco V 670) in the range of 200-700 nm [14].

XRD analysis

XRD analysis confirms the structural properties of the samples. The XRD patterns of Sol A and Sol B samples are shown in Fig. 1 and 2 respectively. The XRD patterns of all the samples show no peaks of crystallite phase of NiO thin films, and all the X-ray diffraction patterns exhibit amorphous structure [15].



50-0 10 20 30 40 50 60 70 80 90 2θ (degree)

Figure 2 XRD pattern of Sol B sample

FESEM analysis

Field Emission Scanning Electron Microscopy is used to analyze the surface morphology of the samples. The surface morphology of Sol A is shown in Fig.3 and reveals smooth surface with particle size in the range of 200 nm. The FESEM image of Sol B confirms smooth surface with particles size ranging of 300 nm is shown in Fig. 4.



Figure 3 FESEM image of Sol A sample



Figure 4 FESEM image of Sol B sample

FTIR analysis

FTIR Analysis is an analytic technique used to identify organic, polymeric and inorganic materials and also provide information regarding chemical and molecular structure of the materials. The FTIR spectrum is taken with the help of Shimadzu in the range of 4000-400cm⁻¹.

The obtained characterized peak of Sol A is given in the Fig. 5. The peak obtained at 1718 cm⁻¹ represent bending vibration and stretching vibration of O-H and 912 cm⁻¹ C-O and C-C stretches [12]. The peak at 560 cm⁻¹ confirms stretching vibration [6]. The characteristic peaks appeared at 458 cm⁻¹ and 408 cm⁻¹ is due to stretching bond between Ni and O [3].



Figure 5 FTIR spectrum of Sol A sample

Table 1	FTIR	peak	assignments	of Sol	А	sample
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Wavenumber (cm ⁻¹)	Peak assignments		
1718	Bending vibration and stretching vibration of O-H.		
912	C-O and C-C stretches.		
560,458, 408	Stretching bond between Ni and O		

The FTIR spectrum of Sol B is shown in the Fig.6. The characterized peak at 2914 cm^{-1} shows C-H stretching mode[16]. The peak obtained at 2347 cm⁻¹ indicates the vibration band of CO₂ and 915 cm⁻¹ represent C-O and C-C stretches[12]. The absorbed peak at 557cm⁻¹ and 467 cm⁻¹ confirms Ni-O stretching vibration [3].



Figure 6 FTIR spectrum of Sol B sample

Wavenumber (cm ⁻¹)	Peak assignments
2914	C-H stretching mode.
2347	Vibration band of CO_2 .
915 557, 467	Ni-O stretching vibration.

Table 2 FTIR peak assignments of Sol B sample

3D Laser Profilometry

The 3D laser Profilometry analysis is used to analyze the surface roughness of the samples. The surface roughness is measured by Zeta profilometer apparatus. The surface roughness of Sol A and Sol B are shown in the Fig.7 and Fig. 8 respectively. The thickness of ethanolamine doped NiO films has increased marginally while surface roughness has become

one fold for Sol B which also exhibits 80% transmittance in the entire visible region. This result becomes beneficial as the deposited NiO films can be applied in semiconductor devices.

Table 3 Surface roughness and film thickness of the deposited films

Samples	R. (μm) Surface roughness	R _P (μm)
_	-	Thickness
Sol A	3.41	4.49
Sol B	7.90	6.81







Figure 8 Surface roughness of Sol B sample

UV analysis

Optical properties of the samples have been analyzed using UV-Visible Spectrometer (JASCO V670) in the range of 200-700 nm. The transmittance spectra of all the samples are shown in the Fig.9. The transmittance of Sol A and Sol B is above 80 %.



Figure 9 UV spectra for Sol A and Sol B sample

Conclusion

NiO thin films were prepared by spin coating technique. The prepared samples are characterized by using XRD, FESEM, FTIR, 3D Laser Profilometry and UV Visible spectrophotometry. The transmittance of the visible spectra is above 80% for Sol A and Sol B.The functional groups are identified by FTIR spectra and it shows the presence of stretching bond between Ni and O. The smooth surface with particle size of 200 nm for Sol A and 300 nm for Sol B are observed from FESEM. Amorphous nature of NiO thin films are revealed by XRD pattern. The effect of ethanolamine and HCl leads to high transparency of the thin films which can be used for semiconductor devices.

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