Effect of Film Thickness on Structural and Electrical Properties of Sputter-Deposited Nickel Oxide Films

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This work studies dependences of resistivity, carrier concentration, mobility and structural properties on the thickness of nickel oxide (NiO) films deposited onto glass substrates by RF magnetron sputtering in a pure oxygen atmosphere at an RF power 200 W. The electrical properties were measured by Hall Effect measurements. The X-ray diffraction (XRD) and transmission electron microscope (TEM) analyses of nickel oxide films indicates that these films are polycrystalline when the samples are prepared with an unheated substrate ($T_s = 303$ K) and using a substrate at a higher substrate temperature ($T_s = 673$ K). The thickness of the films varied in the range from 50 to 300 nm. The variations of the microstructural parameters, such as crystallite size (L), dislocation density (δ), stacking fault probability (α), strain (ϵ) and density (D), with film thickness and substrate temperature were investigated. The results show the crystallite sizes increase as the thickness of the film increases. The variation of the dislocation density and the stacking fault probabilities and strain decrease as the thickness increases. The resistivity of NiO film is increased with an increase in film thickness, which is related to the decrease of carrier concentration with film thickness. The NiO film with a thickness of 200 nm has a minimum resistivity of $0.69 \times 10^{-2} \Omega\text{m}$ when deposited at substrate temperature of 303 K.

(Received December 7, 2004; Accepted March 1, 2005; Published April 15, 2005)

Keywords: nickel oxide films, thickness, hall effect, structural, electrical

1. Introduction

Nickel oxide (NiO) thin films with an NaCl-type structure have attractive interest recently because they have excellent chemical stability, favorable optical, electrical and magnetic properties. They have been used as an antiferromagnetic material,¹⁾ p-type transparent conducting films,²⁾ material for electrochromic display devices³⁾ and in functional sensor layers for chemical sensors.⁴⁾ These films have been fabricated using various physical and chemical vapor deposition techniques, such as spray pyrolysis,⁵⁾ plasma-enhanced chemical vapor deposition⁶⁾ and reactive sputtering.³⁾ Among these methods, reactive sputtering is the most extensively used. Researches^{6–10)} have been carried out on the dependence of film properties on sputtering parameters. Numerous reference data and previous studies^{6–10)} have demonstrated that superior electric and optical properties of NiO films can be obtained by reactive sputtering with a sputtering pressure in the range 0.1–1 Pa and in a pure oxygen atmosphere using a heated substrate.

The influences of film thickness on the structural, optical, magnetic and electrical properties of thin films are very important. Many reports on the effect of size on thin films of various materials have been published.^{11–15)} However, only a few works on the dependence of the properties of nickel oxide films on film thickness have been published.¹⁶⁾

Nickel oxide films obtained by sputtering onto glass substrate at 303 and 673 K were analyzed to understand the role of film thickness on the structural and electrical properties of the nickel oxide films. The thickness of the films was varied between 50 and 300 nm.

2. Experimental Methods

2.1 Film preparation

Nickel oxide films were deposited on a Corning 1737 with a 0.7 mm thick glass substrate by RF magnetron sputtering from an NiO target with a purity of 99.99% in an atmosphere of pure oxygen. The distance between the target and the substrate was approximately 80–85 mm. The chamber was evacuated to a pressure of under 4×10^{-4} Pa before deposition. Sputtering deposition was performed at a gas pressure of 1 Pa. The RF power was maintained at a constant 200 W. The substrates were kept at room temperature 303 and 673 K respectively. The sputtering time was changed to yield different film thicknesses.

2.2 Film characterization

A conventional stylus surface-roughness detector (Alpha-step 200) was used to measure the thickness of films. The electrical properties of nickel oxide films, including resistivity, carrier concentration and mobility, were measured using a Hall measurement system ("Lake Shore" model 7604 Hall Effect measurement systems). The crystal structure of the deposited films was identified by X-ray diffraction (XRD). XRD patterns of the films were obtained using a Rigaku D/MAX 2500 multipurpose X-ray thin film diffractometer with monochromatic high-intensity $\text{CuK}\alpha$ radiation ($\lambda = 0.15418$ nm). The microstructure was observed using an ultrahigh resolution analytical electron microscope (HR-AEM, Hitachi model HF-2000 field emission transmission electron microscope, Japan and FEG-TEM, Philips Tecnai F30 Field Emission Gun Transmission Microscope, Holland). The surface and in-depth profile of NiO films were analyzed by Auger Electron Spectroscopy (VG. Microlab 310D Auger Electron Spectroscopy).

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2.3 Structural analysis

The inter-plane spacings can be calculated given by the Bragg law.¹⁷⁾

$$d_{hkl} = \lambda / (2 \sin \theta) \quad (1)$$

where d_{hkl} is inter plane spacing (d -spacing); $\lambda = 0.15418$ nm is X-ray wavelength of CuK α radiation, and θ is the diffraction angle.

The full-width at half-maximum (FWHM) can be expressed as a linear combination of the contribution from the lattice strain and crystallite size.^{18,19)} The effects of the strain (ε) and crystallite sizes (L) on the FWHM can be expressed by the following equation (Williamson–Hall method):^{18,19)}

$$\beta(\cos \theta / \lambda) = 1/L + \varepsilon(\sin \theta / \lambda) \quad (2)$$

where β is the measured FWHM (in radians), θ is the Bragg angle of the peak, λ is the X-ray diffraction wavelength, L is the effective crystallite size, and ε is the effective strain. The strain value obtained from a Williamson–Hall plot serves to indicate the magnitude of the strain distribution of a non-uniform strain state. A crystalline size value can also be obtained from a Williamson–Hall plot through the intercept of the fitted line with the y -axis.

The dislocation density (δ), defined as the length of the dislocation lines per unit volume of crystal, is evaluated using the formula,^{14,15,20)}

$$\delta = (n/L^2) \quad (3)$$

where n is a factor that equals unity when the dislocation density is minimum and L is the crystallite size.

The stacking fault probability α is the fraction of layers that undergo stacking sequence faults in a given crystal, so fault is expected to be found in $1/\alpha$ layers.²⁰⁾ The occurrence of stacking faults gives rise to a shift in the peak positions of different reflections with respect to the ideal positions of a fault-free well-annealed sample or a standard sample. Warren and Warekoi^{20,21)} proposed a relation between α and the peak shift $\Delta(2\theta)$. The stacking fault probability α is given by

$$\alpha = [2\pi^2 / (45\sqrt{3} \tan \theta)] \Delta(2\theta) \quad (4)$$

The stacking fault probabilities were calculated from the shift of the peaks of the X-ray lines of the films with reference to the 2003 JCPDS database No: 89-7130, using eq. (4).

Nickel oxide is a cubic structure. The lattice parameter ‘ a ’ can be evaluated from the relation

$$a = d\sqrt{h^2 + k^2 + l^2}, \quad (5)$$

where h , k and l are Miller indices.

The density of nickel oxide films was estimated from²²⁾

$$D = M / (6.02 \times 10^{23} \times V) \quad (6)$$

where M is the total mass of the unit cell of NiO; 6.02×10^{23} is the Avogadro constant, and V is the volume of unit cell of the NiO.

3. Results and Discussion

3.1 Structural properties

Figure 1 displays the X-ray diffraction patterns for nickel oxide films, prepared at 200 W in an atmosphere of pure

Fig. 1 X-ray diffraction patterns for different thickness of NiO films that were deposited on substrate at temperature of (a) 303 K, (b) 673 K.

oxygen, with thickness in the range from 50 to 300 nm. Figure 1(a) shows the films deposited on substrates at 303 K. Figure 1(b) shows the films deposited on substrates at 673 K. The different peaks are indexed and the values of interplanar spacing d are calculated and compared with standard 2 theta and d values (2003 JCPDS database No: 89-7130). Table 1 and 2 present the calculated d values and the corresponding indices, respectively.

The peak intensity increases with the film thickness and the X-ray spectra are polycrystalline in nature. Also, the X-ray and electron diffraction patterns indicate that the films deposited on the glass substrates maintained at 303 and at 673 K have an NaCl-type structure. The XRD pattern of NiO films prepared at these two substrate temperatures are compared. Those films deposited at a substrate temperature of 303 K had (111) preferential planes and those films deposited at 673 K had (200) preferential planes.

The dependence of crystallographic orientation on the substrate temperature of 303 and 673 K for NiO films might be explained as follows. The crystal orientation of the film is determined by the method for growth of the nuclei. In the deposition of oxide films, the films initially nucleated in a

Table 1 Structural information obtained from XRD patterns for NiO films of different thickness deposited at room temperature (303 K) that compared with JCPDS-No: 89-7130. (Ref. 2003 JCPDS database)

Substrate temperature T_s/K	Thickness t/nm	hkl	2θ deg.	d -spacing d_{hkl}/nm	Lattice parameters $a = b = c/nm$	FWHM $W_{FWHM}/deg.$
JCPDS-No: 89-7130		111	37.10	0.24216	0.41944	
		200	43.10	0.20972	0.41944	
		220	62.59	0.14829	0.41944	
303 K	50	111	36.99	0.24283	0.42059	0.47
		200	42.94	0.21046	0.42091	1.3
		220	62.21	0.14911	0.42174	0.9
	100	111	37.04	0.24251	0.42004	0.42
		200	42.85	0.21088	0.42176	0.98
		220	62.28	0.14896	0.42131	0.65
	150	111	37.06	0.24238	0.41982	0.43
		200	43.01	0.21013	0.42026	0.82
		220	62.4	0.14870	0.42058	0.75
	200	111	37.16	0.24175	0.41873	0.46
		200	42.97	0.21032	0.42063	0.91
		220	62.52	0.14844	0.41986	0.74
	250	111	37.16	0.24175	0.41873	0.41
		200	43.22	0.20916	0.41832	0.96
		220	62.55	0.14838	0.41968	0.57
	300	111	37.06	0.24238	0.41982	0.42
		200	43.03	0.21004	0.42007	1.11
		220	62.46	0.14857	0.42022	0.64

Table 2 Structural information obtained from XRD patterns for NiO films of different thickness deposited at higher temperature (673 K) that compared with JCPDS-No: 89-7130. (Ref. 2003 JCPDS database)

Substrate temperature T_s/K	Thickness t/nm	hkl	2θ deg.	d -spacing d_{hkl}/nm	Lattice parameters $a = b = c/nm$	FWHM $W_{FWHM}/deg.$
JCPDS-No: 89-7130		111	37.10	0.24216	0.41944	
		200	43.10	0.20972	0.41944	
		220	62.59	0.14829	0.41944	
673 K	50	111	36.93	0.24321	0.42125	0.9
		200	42.87	0.21078	0.42157	0.74
		220	62.02	0.14952	0.42290	1.35
	100	111	36.78	0.24416	0.42291	1.03
		200	42.66	0.2117	0.42355	0.65
		220	61.87	0.14984	0.42383	1.47
	150	111	36.87	0.24359	0.42191	0.77
		200	42.62	0.21196	0.42392	0.66
		220	61.87	0.14984	0.42383	1.18
	200	111	37.07	0.24232	0.41971	1.07
		200	42.87	0.21078	0.42157	0.75
		220	62.42	0.14866	0.42046	1.60
	250	111	37.06	0.24238	0.41982	0.93
		200	43.1	0.20971	0.41942	0.72
		220	62.7	0.14806	0.41877	1.44
	300	111	37.02	0.24264	0.42026	0.69
		200	42.86	0.21083	0.42166	0.62
		220	62.24	0.14904	0.42156	1.11

random orientation. When the film grows from the initial nuclei, the crystal plane of the nuclei with minimum surface free energy may remain parallel to the film surface, because the growth rate of the crystal plane with minimum surface

free energy is lower than of the other crystal plane. In an ionic crystal of NiO with NaCl-type crystal structure, crystallographic orientation of the film is affected by the arrangement of O^{2-} when active species of nickel and oxygen, which are

produced by sputtering process, collide separately with the growing film surface.^{1,23)} This is because NiO has no directivity of combination between Ni^{2+} and O^{2-} , and the radius of O^{2-} (0.140 nm) is larger than that of Ni^{2+} (0.069 nm). In NiO with NaCl-type crystal structure, on the other hand, (111) plane is the most densely packed plane of O^{2-} for NiO crystal structure. It indicates that the (111) orientation minimizes the surface free energy of growing NiO film in this case. The (100) plane is the most densely packed plane among the planes composed of both Ni^{2+} and O^{2-} , indicating that the (100) orientation minimizes the surface energy of growing NiO films in this case. When the deposition is conducted in O_2 , it may be controlled by the arrangement of O^{2-} , resulting (111) orientation of the NiO films. When Ni atoms or NiO molecules are sputtered more efficiently than oxygen atoms, (111)-oriented NiO film maybe difficult to obtain because the growth layer is composed of a mixture of Ni^{2+} and O^{2-} under this condition. Fujii *et al.*¹⁾ prepared NaCl-type NiO films on glass substrates at 400°C by PE-MOCVD. The (111) and (100) preferred orientations of NiO films were controlled by changing the O_2 flow rates. The (111)-orientated NiO film was obtained at lower O_2 flow rates, and (100)-orientated NiO film was obtained at higher O_2 flow rates. Ryu *et al.*²³⁾ observed that the NiO films deposited on Si(100) substrate at room temperature in different ratios of O_2 to Ar by RF magnetron sputtering had (100)-oriented NiO film in an Ar atmosphere and (111)-oriented film in an O_2 atmosphere. Kang *et al.*²⁴⁾ prepared NiO films with $\text{Ni}(\text{C}_5\text{H}_5)_2/\text{O}_2$ at various temperatures and O_2 flow rates by chemical vapor deposition. The (111)-orientated NiO film was obtained at the low deposition temperature region (<548 K), low nickel content in nickel oxide, and low grain size. The (100)-orientated NiO film was obtained at the high deposition temperature region (>548 K), high nickel content in nickel oxide, and high grain size. These different methods indicate that the NiO film texture depends on the oxygen content as well as on the deposition temperature. In our study, nickel oxide (NiO) films were deposited onto glass substrates by RF magnetron sputtering in a pure oxygen atmosphere. The (111)-oriented NiO film was obtained at room temperature of 303 K, and (100)-oriented NiO film was obtained at higher temperature of 673 K. This result can be attributed to a minimum surface mobility of the nuclei at higher substrate temperature of 673 K. At higher substrate temperature, the sputtered atoms obtain more kinetic energy when they arrive at the substrate surface. Also extra thermal energy is provided by the heated substrate. The mobility of atoms and clusters, which is in proportion to their energy, on the substrate will be increased with increasing the substrate temperature. It may favor the growth of the films along some simple crystal planes such as (100) and (001). It is suggested that the NiO molecules are sputtered more efficiently than oxygen atoms. It is easier to obtain (100)-oriented NiO film because the growth layer is composed of a mixture of Ni^{2+} and O^{2-} under higher substrate temperature.

In Fig. 2, $\beta \cos \theta / \lambda$ is plotted versus $\sin \theta / \lambda$ for NiO films deposited on substrate temperatures of 303 and 673 K. The effective crystallite size that has taken the strain into account can be estimated from the extrapolation of the plot shown in

Fig. 2 Williamson-Hall plots of NiO films deposited on substrate at temperatures of (a) 303 K, (b) 673 K.

Fig. 2. The slope of the fitted line indicates the presence of strain in the ZnO crystal lattice. Transmission electron microscopy was also used to observe the microstructure of the NiO films. Figure 3 shows the transmission electron micrograph (TEM) and transmission electron diffraction patterns of NiO films deposited at room temperature (303 K) and at 673 K, with a thickness of about 200 nm. The crystallite sizes were found in nanoscale and estimated at about 20–30 nm. The crystallite sizes were estimated from Williamson-Hall plot, which is similar to that observed by TEM. Table 3 presents the crystallite size (L), density (D), strain (ϵ), dislocation density (δ) and stacking fault probabilities (α). Figure 4 plots the variation of the crystallite size (L) and strain (ϵ) with the thickness of the films. Figure 5 plots the variation of dislocation density (δ) and stacking fault probabilities (α) with thickness.

The crystallite size increases with the thickness of the films and strain decreases with the thickness of the films, as shown in Fig. 4. The dislocation density and the stacking fault probabilities, all decrease as the thickness increase as shown in Fig. 5. Figure 6 shown the nature of dislocations and stacking faults in NiO films were observed by high-resolution transmission electron microscopy (HR-TEM).

The increase in the crystallite size may be caused by a columnar grain growth in the structure. The crystallite sizes

Fig. 3 TEM photographs of NiO films deposited on substrate at temperatures of (a) 303 K, (b) 673 K, and a thickness of about 200 nm.

of NiO films deposited at 673 K were smaller than those deposited without heating (303 K) which fact may be attributed to the formation of new smaller crystallites on the larger grains. The FWHM was observed to decrease as the thickness of the film increased. The result reveals the crystallization approaches more perfect as the film was increased. This is due to decrease the internal micro-strain in the films and the increase in the crystallite size.^{15,25} Table 3 compares the crystallite size (L), the dislocation density (δ), the stacking fault probabilities (α) and the strain (ϵ) of the

NiO films of different thickness on glass substrates. The crystallite size increases but the dislocation density decreases as the film thickness increases. Increasing the thickness, both the micro-stresses and the dislocation density existing in the film decrease. The reliefs of the stresses built up in the films leads to reduction in the interplanar spacing, and thus minimize the stacking fault probability in the films.²⁶ During the increase of the film thickness, the dislocation density and the microstrain are reduced because stress is released in the films by simultaneously annealing during deposition. Also the crystallite sizes initially gradual increase with film thickness and finally to a saturation value. The cumulative effect of the decrease in the microstrain and the dislocation density can be used to explain the gradual reduction in the stacking fault of the films with increasing film thickness.²⁶

3.2 Electrical properties

Figure 7 shows the dependence of resistivity (ρ) and carrier concentration (N) on the thickness of the NiO films that were deposited on substrate at the temperatures of 303 and 673 K. Figure 7(a) shows the resistivity and carrier concentration of the as-deposited films prepared on unheated substrate (303 K). The resistivity is seen to increase from 0.84×10^{-2} to $2.2 \times 10^{-2} \Omega \text{ m}$ as the film thickness is increased from 50 to 300 nm. The carrier concentration is also seen to decrease from 15.3×10^{18} to $8.94 \times 10^{18} \text{ cm}^{-3}$ as the film thickness is increased from 50 to 300 nm. It can be explained with the fact that resistivity is known to be inversely proportional to the carrier concentration. Figure 7(b) shows the resistivity and carrier concentration of the film prepared on the heated substrate of 673 K. The resistivity is seen to increase from 1.63×10^{-2} to $11.09 \times 10^{-2} \Omega \text{ m}$ as the film thickness is increased from 50 to 300 nm. The carrier concentration is also seen to decrease from 10.6×10^{18} to $0.08 \times 10^{18} \text{ cm}^{-3}$ as the film thickness is increased from 50 to 300 nm. It can be seen that the film thickness has a much larger effect on the resistivity (10 times higher) as well as the carrier concentration (100 times lower) as the substrate is heated.

The conduction mechanism of the NiO film is believed to be related to the concentration of the electrical carrier, which is the oxygen vacancy existed in the structure. The electrical properties of NiO films are then associated with their microstructure and composition, and consequently, on the deposition environment.⁷⁻⁹ To clarify this phenomenon, the film composition was investigated by AES. Figure 8 shows the AES depth profile of the NiO films prepared at substrate temperature of 303 K with film thickness of 250 nm. For the AES profile, the atomic concentration near the surface is usually not a good indicator since it can be affected by the oxygen in the environment. The atomic concentration near the substrate is also not a good indicator since it can be affected by the silicon oxide existed on the surface of the substrate. For the middle region of the AES profile, it can be seen that oxygen concentration is decreased as the sputtering time is increased. The decrease in oxygen concentration indicates that oxygen vacancy is increased. However, the increase in sputtering time corresponds to a decrease in film thickness. Therefore, the AES profile does show that as film thickness increases, concentration of oxygen vacancy de-

Table 3 Microstructural parameters for different thickness of NiO films deposited at different substrate temperatures. (Theory density of NiO: 6.72 g cm⁻³)

Substrate temperature T_s /K	Thickness t /nm	Crystallite size L /nm	Density D g·cm ⁻³	Strain, $\varepsilon \times 10^{-3}$	Dislocation density, $\delta \times 10^{15}$ lin·m ⁻²	Stacking fault probability α
303 K	50	11.94	6.62	1.81	7.01	0.0795
	100	13.42	6.64	1.39	5.55	0.0416
	150	15.23	6.67	1.31	4.31	0.0264
	200	27.90	6.70	1.35	1.28	0.0490
	250	40.77	6.71	1.02	0.60	0.0490
	300	41.20	6.69	1.08	0.59	0.0264
673 K	50	11.17	6.56	1.91	8.01	0.1471
	100	13.53	6.52	2.09	5.46	0.2841
	150	15.00	6.52	1.72	4.44	0.3103
	200	25.35	6.68	2.41	1.56	0.1471
	250	36.13	6.76	2.20	0.77	0.0013
	300	40.39	6.62	1.72	0.61	0.1536

Fig. 4 The crystallite size and lattice strain of the NiO films at different substrate temperature of (a) 303 K, (b) 673 K.

Fig. 5 Dislocation density and stacking fault probability against film thickness for NiO films that were deposited at different substrate temperature: (a) 303 K, (b) 673 K.

creases. This leads to the conclusion that the dominant factor for the resistivity of the NiO films is oxygen vacancy. Hence the resistivity of NiO film is increased with an increase in film thickness.

4. Conclusion

The relationships between the thickness of NiO films and structural and electrical properties are studied. The thicker

(a)

(b)

Fig. 6 HR-TEM lattice images of NiO films prepared at different substrate temperature: (a) 303 K, (b) 673 K.

films of NiO develop more perfect microstructures. The crystallite sizes increase as the thickness of the film increases. The variation of the dislocation density and the stacking fault probabilities and strain decrease as the thickness increases. Stresses in the layers and the dislocation density decrease because the stresses that have built-up in the layers are released as the thickness increases. The resistivity of NiO film is increased with an increase in film thickness, which is related to the decrease of carrier concentration with film thickness. The NiO film with a thickness of 200 nm has a

(a)

(b)

Fig. 7 Dependence of electrical resistivity and carrier concentration on the thickness of NiO films were deposited on substrate at temperature of (a) 303 K, (b) 673 K.

Fig. 8 AES depth profile of NiO films prepared at substrate temperature of 303 K with film thickness of 250 nm.

minimum resistivity of $0.69 \times 10^{-2} \Omega\text{m}$ when deposited at substrate temperature of 303 K.

Acknowledgments

The authors would like to thank the National Science Council in Taiwan for financially supporting this research under Grant No. NSC93-NU-7-006-002 & NSC93-2216-E-168-006.

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