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Structural, electrical and magnetic characterization of nickel-doped tin oxide film by a sol-gel method

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KEYWORDS

Crystal growth; Ni-doped SnO₂; Sol-Gel; Room temperature ferromagnetism; Bound magneton polaron. **Abstract.** Nickel (Ni) doped tin oxide $(Sn_{1-x}Ni_xO_2 x = 0.00, 0.02, 0.04, 0.06, 0.10, 0.20)$ thin films were deposited on glass substrates by a sol-gel method using an ethanol solution containing tin and nickel chloride. The structural and optical properties of Ni-doped SnO₂ transparent semiconducting thin films were investigated. X-ray diffraction patterns showed that all samples have tetragonal phases. The morphology of the films shows that they have a good surface and are very dense. The grain size was calculated between 4.4 and 5.3 nm by a transmission electron microscope. The electrical measurement showed that the resistivity increases as the Ni concentration increases. The optical properties of the films measured by UV-Vis showed that the films have transparency between 90% and 98%. The extinction coefficient is very small, and the refractive index is saturated at a wavelength > 400 nm. The VSM results showed that all the samples are ferromagnetic, except for the lowest Ni dopant. Moreover, the original ferromagnetism can be explained by the Bound Magneton Polaron (BMP) mechanism.

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1. Introduction

Transparent Conducting Oxide (TCO) materials, such as indium oxide (In_2O_3) , zinc oxide (ZnO), tin oxide (SnO_2) , and cadmium oxide (CdO), exhibit high optical transparency and electrical conductivity, simultaneously. These materials have a wide range of scientific applications, including optoelectronic devices, solar cells, electro-chromic devices, gas sensors, and flat-panel displays [1]. In order to enhance the application of these materials, many researchers have studied the effect of dopant material on their physical properties [2,3]. B. Thangarajuhave prepared F: SnO₂, Sb: SnO₂, and (F+Sb): SnO₂ film by spray pyrolysis at several temperatures (400°C, 350°C and 375°C). It was shown that the lowest sheet resistance, 5.65 Ω/\Box , was obtained from the F:SnO₂ sample [4]. It depends on the presence of interstitial Sn atoms and oxygen vacancies that leads to SnO_2 , and becomes an n type semiconductor [5]. Some researchers report that the physical properties, such as structure, and electrical and optical properties, could be improved by an improvement in the doping process. For example, Leite et al. reported that Sb plays an important role in reducing the resistivity of SnO_2 thin films prepared by soft chemical methods. The minimum resistivity was obtained around 10^{-3} Ω -cm for 6% Sb doped SnO₂, while the resistivity for pure SnO_2 is 3 Ω -cm [6]. Some researchers have studied the effect of transition metal dopants, such as Mn, Co, Cr and Ni, on the physical properties of SnO_2 . These elements act as an electron, acceptable at the tin oxide site, and change the type of semiconductors. Ghodsi et al. prepared Mn-doped

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 SnO_2 thin films using the sol-gel method, and reported that the n-type semiconductor changes to a p-type semiconductor as the Mn dopant is increased to more than 0.035 wt%. The transparency of their films was between 80-85%. [7].

Ni doped SnO_2 (film and nanoparticles) has attracted research interest since some experimental results have shown that Ni doped SnO_2 is a possible candidate for diluted magnetic semiconductor material [8-10]. Ahmad et al. prepared Ni-doped SnO₂ nanoparticles using the sol-gel method [11]. The XRD results show that all samples have tetragonal structures and the average crystalline size changes from 5 nm to 2 nm as the Ni dopant increases. The optical band gap decreases with the increment of the dopant level. Room temperature ferromagnetism (RTFM) of Ni doped SnO_2 is one of the interesting properties investigated by some research workers. Sharma et al. reported on Ni doped SnO_2 nanoparticles using a co-precipitation technique. The XRD result shows that Ni dopant has no significant effect on SnO₂ nanoparticle structure. The optical result revealed that Ni dopant decreases the band gap. Also, the highest magnetization is obtained from the $Sn_{0.97}Ni_{0.03}O_2$ sample by 0.028 emu/g [12]. Zhang et al. prepared Ni-doped SnO_2 thin films on silicon (111) substrate by a sol-gel method and studied the structure and magnetic properties of the films. The films with different nickel content have a rutile structure, and the sample annealed at 600°C has the highest magnetic properties at room temperature. The magnetic properties are explained using the Bound Magnetic Polaron theory (BMP) [8]. However, other important characteristics for Ni-doped SnO_2 deposited on glass substrates by a sol-gel method, such as electrical, optical and magnetic properties, have not been reported. SnO_2 is a famous transparent conductor, and, therefore, it is important to know its physical properties after doping with Ni.

This work reports the characterization of undoped and Ni-doped SnO_2 deposited on glass substrates by a sol-gel method. The objective of this study is the preparation and discussion of Ni effects on the physical properties of SnO_2 film.

2. Experimental procedure

SnCl₂ 2H₂O (tin (II) chloride dehydrate (Merk)) and NiCl₂ 6H₂O were dissolved in 10 ml ethanol (99.9%). The solution was stilled for 1 hour at 27°C and refluxed at 75°C for three hours. The obtained solution was filtered and spin-coated on a clean glass substrate for 30 seconds. The films were dried at 100°C for 30 minutes and then annealed at 600°C in the air for two hours. The structural pattern was characterized by a D6 Advance Bruker system using CuK α radiation. The surface morphology and cross-section of samples were studied using a Field Emission Scanning Electron Microscope (FESEM) model Zeiss supra 55 Vp. The electrical properties were measured using a Keithley source meter and a Keithley 2700 multimeter, via the Van der Pauw method. The optical transparency of the samples was studied by a Lambda 900 UV-Vis spectrophotometer, while the magnetic properties were studied by a Vibration Sample Magnetometer (VSM) (Model 7404 lakeshore).

3. Result and discussion

Figure 1 shows the TGA graph of the dried SnO_2 gel. The mass loss occurred at about 100°C to 120°C, which may be attributed to the evaporation of ethanol and water [6]. In addition, other mass losses were not observed until 600°C.

Figure 2 shows the X-ray diffraction patterns of the $\text{Sn}_{1-x}\text{Ni}_x\text{O}_2$ thin films that were studied at 2θ value from 20° to 60°. All samples are polycrystalline with tetragonal phases. The tetragonal phase of SnO_2 is refined by the P_{42}/mnm space group. The planes [(110), (011), (020), and (211)] have high intensity, and the ratios of the intensities are tabulated in Table 1. The



Figure 1. TGA curves of the dried SnO_2 gel.



Figure 2. X-ray diffraction scans of the $Sn_{1-x}Ni_xO_2$ thin film.

Samples	Ni (wt %)	I_{101}/I_{110}	I_{211}/I_{110}
SnNi00	0	0.69	0.29
SnNi02	2%	0.68	0.33
SnNi04	4%	0.50	0.19
SnNi06	6%	0.57	0.28
SnNi10	10%	0.56	0.26
SnNi20	20%	0.70	0.40

Table 1. Intensity of plans in $Sn_{1-x}Ni_xO_2$ thin films.

ratios of the intensities of these peaks changes with the increment of x content, indicating that the doping process affects the crystallinity of the film. Thus, the Ni ions replaced the lattice site of the tin oxide, then, produced a crystal defect [11]. Some reports have shown that the ratio of the intensity peaks was changed as the Cr dopant increased in the SnO₂ films [13,14].

The lattice parameters and cell volume were calculated for all samples using Rietveld refinement software and tabulated in Table 2. The RWP was 13.5 to 14.90. These values show that cell volume and crystalline size decrease as dopant percentage increases due to the replacement of Sn^{+4} (ionic radius 71 pm) with smaller Ni⁺² ion (ionic radius 69 pm) [11].

The $Sn_{0.98}Ni_{0.02}O_2$ and $Sn_{0.80}Ni_{0.20}O_2$ film were analyzed by X-ray Photoelectron Spectroscopy (XPS) instruments. The obtained binding energy from XPS spectra were calibrated to the adventitious C 1s peak (248.5 eV). Figure 3 shows the Sn 3d (a), Ni 2p (b), and O 1s (c) binding energy of the films. The obtained binding energy of SnNi02 film was analyzed and the result was tabulated in Table 3. The binding energy confirms Sn^{2+} and Sn^{4+} in the film but if the percentage of SnO in the composition is low, then XRD could not detecte it. And, also, the binding energy of Ni^{2+} and Ni^{3+} identify the oxidation state in the film.

The obtained energy of SnNi20 was analyzed and is given in Table 4. It has been identified as the binding energies for Sn^{4+} and Ni^{2+} in the sample. The above analysis confirms that tin and nickel ions in the films have a chemical valance of 4+ and 2+, respectively.



Figure 3. XPS data from $Sn_{1-x}Ni_xO_2$ (x = 0.02, and 0.20).

The binding energy of O 1s in the $\text{Sn}_{0.98}\text{Ni}_{0.02}\text{O}_2$ sample is 529.8, 531.1 and 532 eV, and for the $\text{Sn}_{0.98}\text{Ni}_{0.20}\text{O}_2$ sample is 529.7, 530.8 and 531.7 eV. It indicates that the oxygen in the film is -2 (Figure 3(c)). The binding energies of 531 and 529.8 eV belong to Sn-

Sample	$egin{a} a \ (\mathbf{A}^{\circ}) \end{array}$	$egin{array}{c} c \ (\mathbf{A}^{\circ}) \end{array}$	${f Thickness}\ ({f nm})$	Crystalline size (nm)	Cell volume (nm)	Average grain size (TEM) (nm)
SnNi00	4.7357	3.2038	73.69	4.70	71.85	4.37
$\mathrm{SnNi02}$	4.7290	3.1925	100	3.79	71.08	5.27
SnNi04	4.3368	3.1977	106.3	4.92	71.65	5.29
SnNi06	4.7451	3.2050	63.28	4.63	72.08	4.89
SnNi10	4.7347	3.1965	67.73	3.96	71.63	4.60
SnNi20	4.731	3.181	84.11	3.06	71.21	4.36

Table 2. Characterization of films was calculated.

Obtained	Standard			
binding	$\mathbf{Spectral}$	binding	Composition	
\mathbf{energy}	line	\mathbf{energy}	Composition	
(\mathbf{eV})		(\mathbf{eV})		
485.6	3d5/2	485.6	SnO	
486.2	3d5/2	486.20	SnO_2	
494.5	3d3/2	494.20	SnO_2	
494.7	3d3/2	494.7	$\mathrm{SnO}_{1.15}$	
854.7	2p3/2	854.7	NiO	
856	2p3/2	856	Ni_2O_3	
857.2	2p3/2	857.2	NiO	
871.7	2p1/2	871.8	NiO	
873.4	2p1/2	873.8	NiO	
874.7	2p1/2	874.9	Ni	

Table 3. The obtained XPS result for SnNi02 film.

Table 4. The obtained XPS result for SnNi20	film.
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Obtained		$\mathbf{S} \mathbf{t} \mathbf{a} \mathbf{n} \mathbf{d} \mathbf{a} \mathbf{r} \mathbf{d}$	
$\mathbf{binding}$	$\mathbf{Spectral}$	binding	Composition
\mathbf{energy}	line	\mathbf{energy}	Composition
(\mathbf{eV})		(\mathbf{eV})	
485.9	3d5/2	486	SnO_2
494.5	3d5/2	494.5	SnO_2
495	3d3/2	495	SnO_2
854.9	2p3/2	854.7	NiO
856.3	2p3/2	856.3	NICl_2
872.5	2p1/2	872.4	$\mathrm{ClOH}_{24}\mathrm{H}_4(\mathrm{NiCl}_4)$
873.7	2p1/2	873.8	$\rm Ni(OH)_2$



Figure 4. Typical FESEM micrographs of film's cross-section.

O bonding in the SnO₂ system and Ni-O bonding in the NiO system, respectively.

Figure 4 shows the typical FESEM of the crosssection of $Sn_{0.90}Ni_{0.20}O_2$ samples, and the measured thickness values are tabulated in Table 2.

Figure 5 shows the typical TEM images of the undoped and the Ni-doped SnO_2 films. The grain sizes were measured and tabulated in Table 2. The grain

size changes irregularly from 4.4 nm to 5.3 nm. Our value is smaller than the value reported by Ahmad et al. who reported that the grain size for Ni-doped SnO_2 nanoparticles is between 9 nm to 15 nm [11].

The electrical result (Figure 6) shows that the resistivity of SnO₂ is much higher than tin oxide films prepared by other techniques. For example, Abdiet al. deposited undoped and Cr-doped SnO₂ thin film on a glass substrate byspray pyrolysis technique and obtained the resistivity of undoped tin oxide at about $1.3 \times 10^{-2} \ \Omega$ -cm [15]. Also, the resistivity of the SnO₂ thin film prepared by electron beam evaporation is $2.65 \times 10^{-2} \ \Omega$ -cm [16]. The high resistivity obtained in this study is related to the low deviation from the stoichiometry of the film.

Figure 6 shows the resistivity and carrier concentration of Ni-doped SnO_2 at room temperature (25°C), wherein the resistivity increases with the increment of Ni content, due to the increment of the degradation in the crystalline site (as shown in the XRD results). Another factor that affects the resistivity can be explained by the fact that the replacement of Sn^{+4} ions with Ni²⁺ ions creates carrier traps that can affect film resistivity. This phenomenon also leads to reduction of carrier concentration, as shown in Figure 6. Carrier concentration was calculated using this formula:

$$n = -\frac{IB}{qV_H t},\tag{1}$$

where I is the applied current, B is the applied magnetic field, q is the electron charg, V_H is the Hall voltage and t is the film thickness.

The optical transparent spectra (T%) for all samples are shown as a function of wavelength in Figure 7. The transparency of all the films was between 90% and 98% in the visible region (300 nm to 850 nm). The transparency of the films had slight changes with the increment of Ni dopant. The small crystalline size (increased boundary) leads to the scattering of light, thereby, decreasing the transmission.

The absorption coefficient (α) , as a function of wavelength for all samples, is shown in Figure 8. The highest dopant of Ni provides the highest absorption coefficient in the visible region.

The presence of Ni dopant in the SnO_2 site causes disorder (mismatch) in its lattice, and increases the band gap. The direct optical band gap of tin oxide and Ni-doped tin oxide was calculated using the following relation:

$$A = \log T,\tag{2}$$

A: Sample absorption, and

T: Sample transmission,



Figure 5. Transmission electron micrograph of (a) SnNi00, (b) SnNi02, (c) SnNi06, (d) SnNi10, and (e) SnNi20 thin films.

$$\alpha = \frac{1}{d} \log \left(\frac{I}{I_o} \right),\tag{3}$$

 α : Sample absorption coefficient and

d: Sample thickness,

$$(\alpha hv) = A(hv - E_g)^n.$$
(4)

The details of the procedure to determine the correct value of n was explained earlier [13]. The direct band gaps for $\operatorname{Sn}_{1-x}\operatorname{Ni}_x\operatorname{O}_2$ (x = 0.00, 0.02, 0.04, 0.06, 0.10, 0.20) thin films were determined from the plot $(\alpha hv)^n (n = 2)$ versus hv. The plot shows that the optical band gap increases with the enhancement of Ni concentration, except for the SnNi02 sample that has the smallest band gap (see Figure 9). There are two possibilities to explain this behavior. First, some reports have suggested that impurities such as the NiO phase is responsible for the increment of the energy band gap [17].

However in this study, none of the NiO phases can be detected in the films. Furthermore, the band gap energy of the NiO was small (3.54 eV) compared with the obtained value. The second possibility is the particle size of the films. The optical band gap decreases for 2% Ni dopant and increases for x > 0.04wt%. This is related to the particle size of samples (see Figure 9), consistent with the normal phenomena of quantum confinement. Although the measured grain size in this study is approximated around the Bohr radius for SnO_2 (2.70 nm), the effect of the grain size on the energy band gap can still be detected. Furthermore, regarding electrical properties, the Nidoped SnO_2 cannot generate a carrier in the SnO_2 lattice. In addition, the carrier decreases with the increment of Ni dopant, indicating that the increase in band gap cannot be attributed to the Burstein-Moss (BM) shift. Baqiah et al. obtained a similar result for Cr-doped In_2O_3 thin film via the sol-gel method [2].

The extinction coefficient 'k' was calculated using



Figure 6. Resistivity and carrier concentration against Ni dopant.



Figure 7. Optical transparency of Ni doped tin oxide.



Figure 8. The absorption coefficient of Ni doped tin oxide.

the following formula:

$$\alpha = \frac{4\pi k}{A},\tag{5}$$

where α and A are the absorption coefficient and the absorption spectra, respectively. Figure 10 shows the extinction coefficient versus wavelength in which the



Figure 9. Energy band gap of Ni doped SnO_2 thin films.



Figure 10. The extinction coefficient of Ni doped tin oxide.

extinction coefficient has a small value that changes irregularly with an increment of level dopant. The highest extinction coefficient value is given by the highest dopant of Ni, due to the high absorption coefficient.

The refractive index 'n' has a relationship with the extinction coefficient, and the reflection is explained by:

$$R = \frac{(n-1)^2 + k^2}{(n+1)^2 + k^2},\tag{6}$$

where R is the reflection spectra. According to extinction coefficient data presented by the above formula, the data is so small, and can, thus, be neglected without losing effective information. The refractive index can then be written as follows:

$$R = \frac{(n-1)^2}{(n+1)^2}.$$
(7)

Figure 11 shows the refractive index versus the wavelength of all the films wherein the refractive index saturates at wavelengths larger than 400 nm.

The k and n results coincide with the results



Figure 11. The refractive index of Ni doped tin oxide.



Figure 12. The magnetization versus magnetic field for different Ni concentration.

reported by H. Demiryont et al. prepared SnO₂ film by the spray pyrolysis method, and then calculated $n(\lambda)$ and $k(\lambda)$ using the transparency spectra [18].

The magnetic properties of $Sn_{1-x}Ni_xO_2$ (x = 0.00, 0.02, 0.04, 0.06, 0.10, 0.20 films are shown in Figure 12, where the diamagnetic contribution of the substrate was deduced. The films seem to have ferromagnetic properties at room temperature (RTFM), whereas $Sn_{0.98}Ni_{0.02}O_2$ is diamagnetic. These results are different from that of the Ni-doped SnO_2 thin films on Si(111) substrate prepared by Zhang et al. by the sol-gel method, because the highest moment for their result was from the low dopant [8]. In our results, the magnetization was changed by the increment of the Nidopant, while the highest magnetization was obtained from 10% Ni dopant by 8.8 emu/cm³. Our results matched those of Nguyen Hoa Hong et al. who measured the magnetic saturation of 2^{μ_B} /Ni for $Sn_{0.95}Ni_{0.05}O_2$ thin film deposited on LaAlO [10].

Although the origin of the RTFM is still unknown, the carrier-mediated exchange mechanism is a



Figure 13. The magnetic saturation and carrier concentration against Ni concentrations.

well-used mechanism for explaining RTFM [19]. In our films, the carrier concentration decreased with enhancement of the Ni dopant, and the large density of carriers had the lowest ferromagnetic momentum (Figure 13). These results indicate that the original RTFM cannot be explained by carrier-mediated exchange interaction, such as the RKKY (Ruderman-Kittel-Kasuya-Yosida) mechanism.

The original ferromagnetic of pure SnO_2 is called d^0 ferromagnetic. This phenomenon is related to the lattice defect in the crystal. To understand the d^0 ferromagnetic, a charge transfer ferromagnetic model was developed by Coey et al. [20]. According to this model, ferromagnetic order is from itinerant electrons associated with structure defects. These defects create an impurity band in the band gap. Based on the tight-bonding model, the density of states depends on the number of nearest neighbours. In the vicinity of defects, this number of nearest neighbours is reduced, resulting in an increase in the local density of states. This will lead to some sort of density state structure. Electrons can be transferred to the defect states from an electron reservoir (this electron reservoir occurs due to ions coexisting in different valance states). This electron transfer could raise the Fermi level up to the point where spontaneous stoner spin splitting of the impurity band occurs. Spontaneous ferromagnetic order exists if the energy gap from spin splitting exceeds the energy cost of the charge transfer [21].

The description of the other samples (x = 0.04, 0.06, 0.10, and 0.20) can be interpreted by the Bound Magnetic Polaron (BMP) model [22]. Oxygen vacancies were created in the SnO₂ to maintain the charge neutrality when the Ni⁺² ions substitute the Sn⁺⁴ ions. The oxygen vacancies cause the conduction of RTFM. Furthermore, a hydrogen orbital is created when an electron belongs to the oxygen trap in the oxygen vacancy. The trapped electron and coupled Ni²⁺ construct polaron, as well as the overlapping of the magnetic moment of the polarons, causes RTFM [8].

4. Conclusion

Transparent SnO_2 thin films were prepared successfully using the sol-gel method. The physical properties of these films were investigated with various nickel dopants. The structures of these films exhibited a slight change in the lattice parameter and the cell volumes. The resistivity for SnO_2 was 74 Ω -cm, which increased with the increment of Ni concentration until 843 Ω -cm. The optical properties showed that the samples have high transparency and their band gap changed between 3.85 and 3.94 eV. The presence of nickel reduced the transparency and increased the band gap. The extinction coefficient and refractive index were altered for different level content. The sample with different Ni dopant, except for the lowest dopant, exhibited RTFM, and the 10% Ni dopant had the highest magnetic moment.

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