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Ni-Doped SnO_2 Nanoparticles Synthesized by Chemical Co-Precipitation Method

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Abstract: Ni-doped SnO_2 nanocrystalline powders have been synthesized by the co-precipitation method from $SnCl_2.2H_2O$ and $NiCl_2.6H_2O$. Nanoparticles crystallize in lower temperature (350°C) and shorter time (2hours) respect to other methods. The samples have been characterized by various advanced techniques such as, X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), Transmission electron microscopy (TEM) and Vibrating sample magnetometer (VSM). The X-ray diffraction reveals that all samples are pure rutile-type tetragonal phase and the average particles size was observed to vary from 32 nm to 24 nm as the nickel content was increased, The TEM images confirms the size of tin oxide particles in nanoscale and VSM measurements indicate that Ni-doped samples have superparamagnetic properties and they are single-domain nanoparticles.

Key words: Nanoparticles · Nickel · Tin oxide · Superparamagnetic

INTRODUCTION

In recent years, the interest in the physical properties of semiconducting metal oxide, such as SnO_2, TiO_2 and ZnO, has significantly increased due to their potential applications, in special when they are intentionally doped with magnetic elements. Tin oxide (SnO_2) with a wide-band-gap n-type semiconductor (3.6eV at 300°K), known as one of the most widely used semiconducting oxides due to its chemical and mechanical stabilities. It has been widely studied over decades because of its most applications in various optoelectronic devices, transparent electrodes and sensors [1-3].The properties of SnO_2 nanostructures can be enhanced by several ways like impurity doping [4], coating with surfactants [5] and annealing [6]. The final properties of impurity doped SnO_2 nanoparticles are related to both composition and processing method. Nanoparticles of tin oxide have been synthesized through different chemical routes, such as co-precipitation[7], hydrothermal [8] and sol gel [9] methods among others. In this work, we synthesized Ni-SnO_2 nanoparticles by chemical coprecipitation method because this method has some advantages such as precise control over the stoichiometry, low temperature synthesis, high purity and high chemical homogeneity.

MATERIALS AND METHODS

Ni doped SnO_2 nanoparticles have been synthesized by chemical co-precipitation method, $SnCl_2.2H_2O$ (98% Merck, Germany) and $NiCl_2.6H_2O$ (98% Merck) have been used as starting materials for the synthesis of Ni doped SnO_2 nanoparticles. The required amounts of $SnCl_2.2H_2O$ and $NiCl_2.6H_2O$ were added to double-distilled water and dissolved. Then, an aqueous ammonia (1M) was dropped into the mixture solution with constant stirring at 30°C until a pH of 5 reached. The resulting precipitate were collected, washed with distilled water and then dried at 100°C for several hours. Heating treatments of the synthesized nano-powders were conducted at 350°C for 2 hours. The preparation process of Ni-doped SnO_2 nanocrystalline powders is shown in Figure 1. Crystallinity, structure and particle size of Ni-doped SnO_2 nanoparticles were determined by X-ray diffraction (XRD) using Rigaku-Miniflex X-ray diffractometer with CuKα radiations (λ =0.15406 nm) in 2θ range from 10° to 70°.

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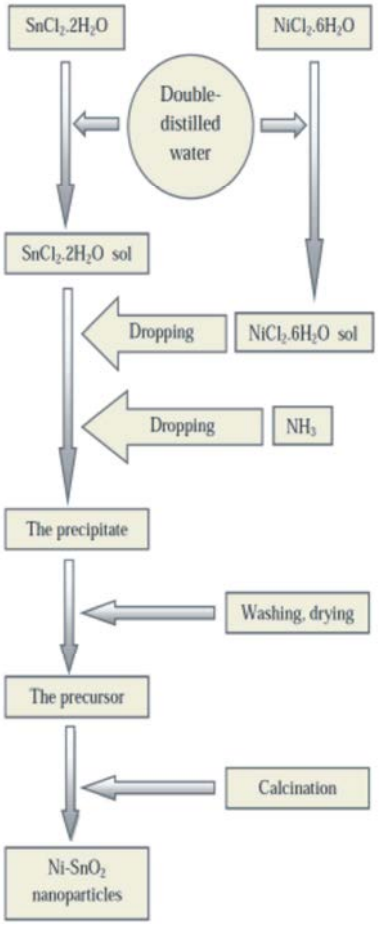

Fig. 1: The preparation process schema of Ni-SnO_2 nanoparticles

Morphologies and particle sizes of the samples were observed with a Hitachi H-800 transmission electron microscope (TEM), FTIR spectrum was recorded in the range 400-4000 cm^{-1} on a FTIR 460-plus spectrophotometer using KBr pellets. The magnetic measurements were obtained using a Vibrating Sample Magnetometer (VSM) in the room temperature.

RESULTS AND DISCUSION

XRD patterns of $Sn_{1-x}Ni_xO_2$ (with x = 0, 0.01 and 0.02) annealed at 350°C for 2 h are shown in Figure 2, in which S_1, S_2 and S_3 correspond to x = 0, 0.01 and 0.02, respectively. All diffraction peaks are well assigned to tetragonal crystalline phase of tin oxide (with the reference pattern JCPDS 880287) and the Ni doping does not change the tetragonal structure of SnO_2. From Figure 2, it is noted that the intensity of the $Sn_{1-x}Ni_xO_2$

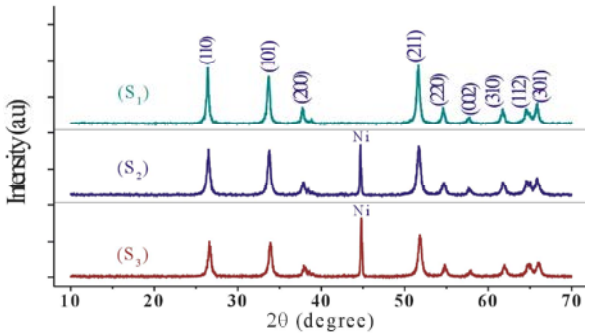

Fig. 2: XRD patterns of (S_1) SnO_2, (S_2) $Sn_{0.99}Ni_{0.01}O_2$ and (S_3) $Sn_{0.98}Ni_{0.02}O_2$ nanoparticles

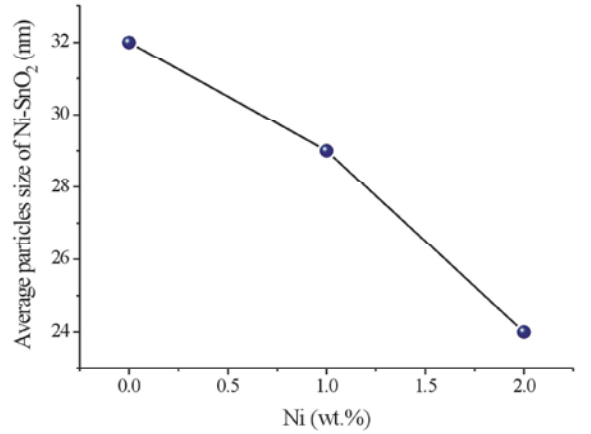

Fig. 3: The effect of the Ni-doping quantities on the average particle size of Ni-SnO_2 nanocrystalline powders

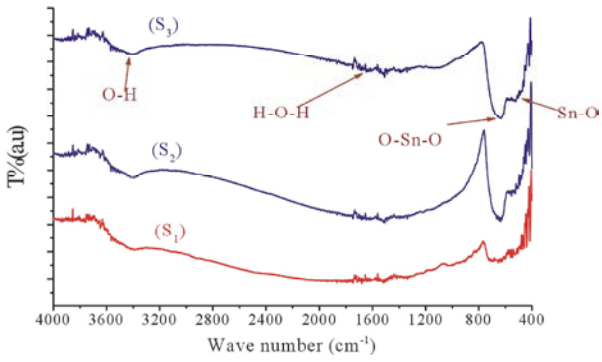

Fig. 4: FT-IR spectra of (S_1) SnO_2, (S_2) $Sn_{0.99}Ni_{0.01}O_2$ and (S_3) $Sn_{0.98}Ni_{0.02}O_2$ nanoparticles

peaks decreases with increasing Ni content and the full-width at half-maximum (FWHM) of the peaks increases with increasing Ni content, as well indicates that average particle size of samples decreases gradually as the Ni content increases (Figure 3). The average particle size of samples are calculated using Scherrer's formula,

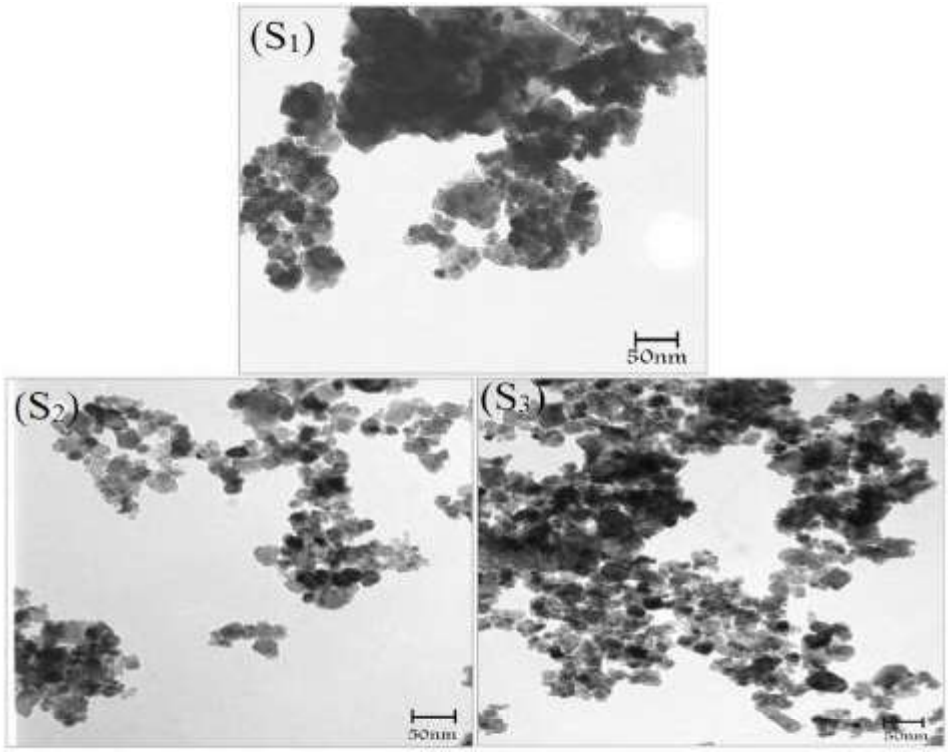

Fig. 5: TEM images of (S_1) SnO_2, (S_2) $Sn_{0.99}Ni_{0.01}O_2$ and (S_3) $Sn_{0.98}Ni_{0.02}O_2$ nanoparticles

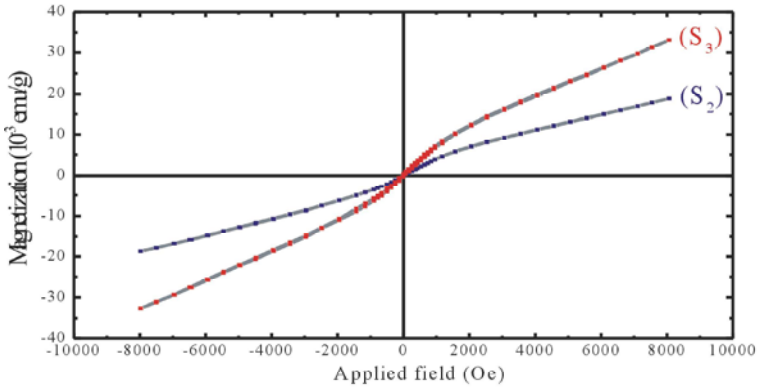

Fig. 6: Room temperature hysteresis curves of (S_2) $Sn_{0.99}Ni_{0.01}O_2$ and (S_3) $Sn_{0.98}Ni_{0.02}O_2$ nanoparticles

$$D = \frac{0.9\lambda}{\beta\cos(\theta)}$$

where λ is the wavelength of CuKα radiation (= 0.154056 nm), β is the full width at half maximum (FWHM) of the (*hkl*) peak at the diffracting angle 2θ [10], all the peaks are used to calculate the average particle size of samples.

The average particles size of samples are found to be 32nm for sample S_1, 29 nm for sample S_2 and 24nm for sample S_3 by using scherrer's formula.

The FT-IR transmittance spectra of different samples are given in Figure 4, in which the band located at 3398 cm^{-1} is owing to the vibration of O-H and the band located at 1636 cm^{-1} is due to the H-O-H vibrating mode of the absorbed water, while the bands at around 528 and 643 cm^{-1} can be attributed to the Sn-O stretching vibration and the O-Sn-O blending vibration in SnO_2.

TEM images of (S_1) SnO_2, (S_2) $Sn_{0.99}Ni_{0.01}O_2$ and (S_3) $Sn_{0.98}Ni_{0.02}O_2$ nanoparticles calcined at 350°C for 2 hours are shown in Figure 5. It is shown that the powders are uniform in size and the particle size of samples decreases with increasing doping quantities of Ni. The particle shape of the samples is spherical with an average diameter

of 28 nm for pure SnO_2, 25 nm for 1% Ni-doped SnO_2 and 21 nm for 2% Ni-doped SnO_2. The results of average particle size measurement from TEM observations are having good agreement with the XRD line broadening method.

To probe the magnetic properties of $Sn_{0.99}Ni_{0.01}O_2$ and $Sn_{0.98}Ni_{0.02}O_2$ nanoparticles, room temperature magnetization is performed on the samples and is shown in Figure 6. It shows no hysteresis loops and the magnetization curve exhibits zero remanence and coercivity (the coercive field is the applied magnetic field that needs to be applied in the direction opposite the initial magnetic field, to bring the magnetization back to zero), which proves that nanoparticles have superparamagnetic properties and they are single-domain nanoparticles [11, 12]. The superparamagnetism enables the nanoparticles to respond to an applied magnetic field without any permanent magnetization and redisperse rapidly when the magnetic field is removed. In the Figure 6, it can be also observed that magnetization of samples increases with increasing doping quantities of Ni.

CONCLUSIONS

Ni-doped SnO_2 nanocrystalline powders were synthesized by the co-precipitation method from $SnCl_2.2H_2O$ and $NiCl_2.6H_2O$. XRD results show that the average particles size is in the range from 24 to 32 nm, it is shown that the average particles size of the nanoparticles decreases with increasing amounts of doping Ni and nanoparticles have pure rutile structure, TEM images of SnO_2 and Ni-SnO_2 powders shows that the particle shape of the samples is spherical with an average diameter of 28 nm for pure SnO_2, 25 nm for 1% Ni-doped SnO_2 and 21 nm for 2% Ni-doped SnO_2, confirming the reduction in particles size as a result of Ni doping in SnO_2. Particles size obtained from TEM analysis is comparable with average particles size calculated from XRD spectra and VSM measurements of nickel doped SnO_2 nanoparticles indicates that both samples $Sn_{0.99}Ni_{0.01}O_2$ and $Sn_{0.98}Ni_{0.02}O_2$ have superparamagnetic properties and they are single-domain nanoparticles.

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