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Synthesis and Properties of Al doped SnO₂ Nanoparticles

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Abstract: Undoped and 5 mol% of Al doped SnO₂ nanoparticles are synthesized by chemical co-precipitation method at room temperature. The morphology and structural properties of SnO₂:Al³⁺ nanocrystals are studied by X-ray diffraction (XRD), scanning electron microscopy (SEM), Energy-dispersive X-ray spectroscopy (EDS), and Raman spectroscopy (RS) techniques. Decrease in particle size from diffraction data indicates the incorporation of Al³⁺ in SnO₂ nanocrystal lattice. A small amount of Al³⁺ dopant favours the formation of stoichiometric SnO₂ nanoparticles. By the addition of Al into SnO₂, there is no shift towards higher or lower 2θ values, which is in agreement with the results of Raman spectra.

Keywords: SnO₂ nanoparticles, Structural properties, EDS, and Raman spectra.

1. Introduction and Experimental:

Recently, semiconductor materials have been extensively investigated because of their unique size dependent electronic, magnetic, optical and electrochemical properties [1-4]. As an n-type semiconductor with a wide energy gap, SnO₂ nanoparticles has a wide range of applications in gas sensors [3], transistors [4] and transparent conducting electrodes [5].

If the semiconductor particles becomes smaller than the Bohr's radius of excitation, quantum size effects occurs [1-5]. In recent years, considerable efforts have been paid on the synthesis of SnO₂ thin films or particles and exploration of their novel properties [6]. Different methods have been employed to synthesize SnO₂ nanoparticles [4, 5 and 7]. The synthesis of SnO₂ with one-dimensional (1D) nanostructures has been reported [8]. A considerable amount of recent work has been devoted to probe new properties of precipitation derived in organic nanoscaled materials. However, it is a challenge to find an efficient and simple way to prepare SnO₂ powders with particle size about several nanometers. In the present work, the pure and 5 mol% of Al doped SnO₂ samples have been synthesized using chemical co-precipitation method and their properties have been investigated.

Nanocrystalline SnO₂ particles are prepared by using a chemical co-precipitation method. Chemicals used in the experiment are analytic reagent (AR) grade and are used without any further purification. Stannous dichloride dehydrate (SnCl₂.2H₂O) is dissolved in distilled water to prepare 0.2 M solution. NaOH solution is then added in to the solution under constant stirring to form white precipitate of pure SnO₂. To prepare doped samples, the same process is repeated by adding of 5 mol% of Al from the source of AlCl₃. The precipitate is filtered out and washed several times to remove chloride ions completely from the precipitate. Finally, the product is dried at 70 °C for 9 hr, and then grounded and annealed in the furnace at 500 °C for 4 hr.

The structural properties of the samples are determined by Seifert 3003 TT X-ray diffractometer (XRD) using CuK_α radiation by applying voltage and current of 40 kV and 30 mA respectively. The surface morphology and chemical analysis of Al doped SnO_2 nanopowders are studied by SEM attached with Energy Dispersive Analysis of X-rays Spectra EDAX (model CARL - ZEISS EVOMA 15). Crystallinity of the nano structures is analysed by Micro Raman Spectroscopy.

2. Results and Discussion:

Typical XRD patterns of pure and Al doped SnO_2 are shown in Fig.1. The diffraction peaks of all samples are quite matching well with tetragonal rutile crystalline phases of tin oxide with a reference pattern (JCPDS 41-1445). No traces of impurity peaks are observed. It reveals that the substitution of Al does not disturb the tetragonal rutile crystalline structure of parent SnO_2 . This may be attributed to the limitation of XRD characterization that small amount of impurities cannot be detected. However, a careful analysis of the peak (110), (101), (200) (211) positions suggests that there is no shift toward higher or lower 2θ values with increasing Al doping as shown in Fig. 1, which indicates that the difference in ionic radii of Sn^{4+} and Al^{3+} is small, consequently small change in crystal micro structure. Average size of crystallites is approximately 21 nm for pure SnO_2 and 17 nm for Al doped SnO_2 sample. Decrease in particle size is due to Al doping is clearly observed and similar type of results have been reported by Kumari *et al.* [9]. From the XRD data, the observed decrease in particles size on doping, which is confirmed by SEM images.

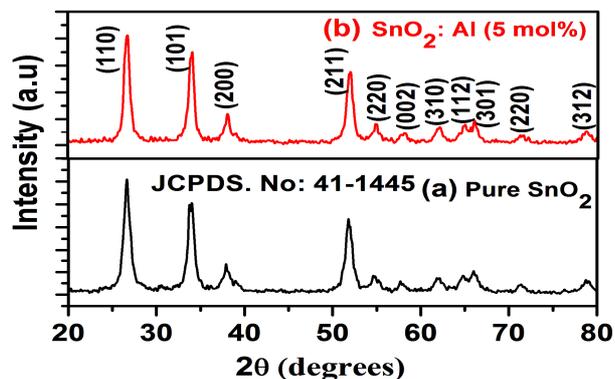


Fig. 1. XRD pattern of (a) pure (b) 5 mol% of Al doped SnO_2 nanoparticles.

The SEM micrographs of pure and Al doped SnO_2 nanocrystalline powders are shown in Fig. 2. The SEM micrographs revealed that the primary particles are weakly agglomerated, and several voids and pores are observed in the size range of $10\ \mu\text{m}$ to $2\ \mu\text{m}$ respectively.

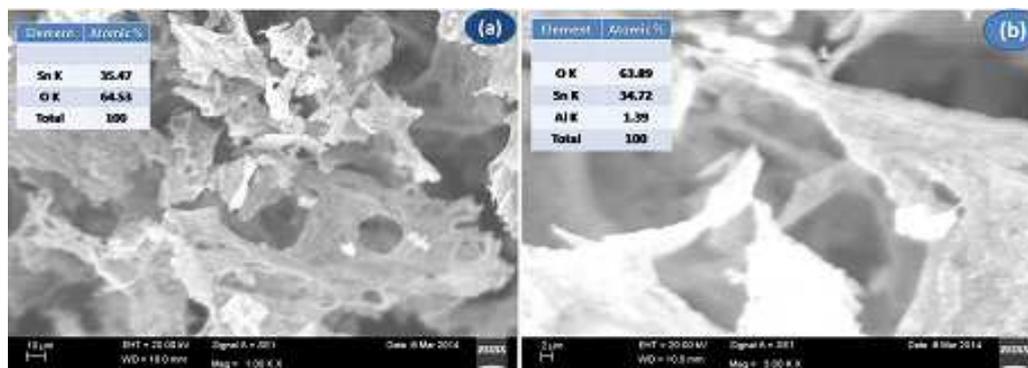


Fig. 2. SEM images of (a) pure SnO_2 (b) Al doped SnO_2 nanoparticles.

The elemental composition of pure and Al doped SnO_2 samples is given as an inset of Fig. 2a, and b. Therefore, the doped SnO_2 nano-powders are clearly consisting of Al elements in appropriate ratio, which are nearly comparable to the taken amounts of corresponding materials.

The Raman spectra of pure and Al doped SnO₂ nanoparticles are shown in Fig. 3. It shows that a little bit appreciable changes are appeared when compared the pure SnO₂ with the Al doped SnO₂ nanoparticles. Three fundamental Raman peaks at 469, 629, and 770 cm⁻¹, corresponding to E_g, A_{1g}, and B_{2g} vibration modes are observed respectively, and are in good agreement with those for the rutile SnO₂ (Fig. 3a). Thus, these peaks further confirm that the as-synthesized SnO₂ nanobelts possess the characteristics of the tetragonal rutile structure [10]. In addition to the fundamental Raman peaks of rutile SnO₂, the other weak Raman peak at about 689 cm⁻¹ is also observed. One weak Raman band at 689 cm⁻¹ seem to be correspond to IR-active E_u(3) TO and A_{2u}LO modes respectively.

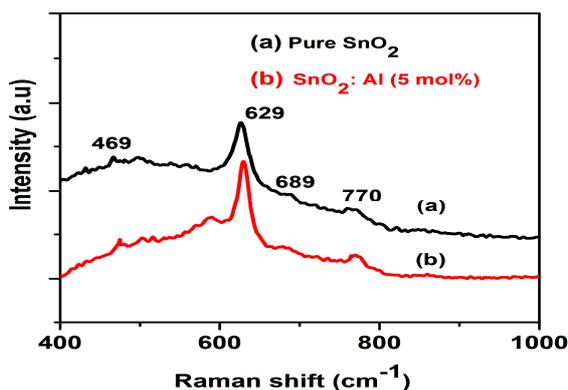


Fig. 3. Raman spectra of (a) pure (b) Al doped SnO₂ nanoparticles.

3. Conclusions:

Pure and Al doped SnO₂ nanopowders are successfully synthesized by chemical co-precipitation method. XRD results showed that the tetragonal rutile structure of SnO₂ nanomaterials, which are in good agreement with the Raman results. The SEM image size of Al doped SnO₂ is smaller than the size of pure SnO₂. Raman spectra of both undoped and Al doped SnO₂ nanopowder samples having nearly the same Raman modes.

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