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Synthesis and properties of (Fe, Al) co-doped SnO_2 nanoparticles

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Abstract

Un-doped and (Fe, Al) co-doped SnO_2 nano powders have been successfully synthesized by chemical co-precipitation method using $SnCl_2$ $2H_2O$, Fe $(NO_3)_2$ and $AlCl_3$ as raw materials and PEG (poly ethylene glycol) as stabilizer. The prepared samples have been annealed at 600^0 C for 4 hours under ambient condition in order to improve the crystallinity. Powder XRD results show that the prepared samples are in tetragonal rutile type SnO_2 phase. The average crystalline size of pure and (Fe, Al) co-doped SnO_2 are found to be around 20-27 nm. The crystal structure of the SnO_2 does not change with the co-doping of Fe and Al, but crystalline size increases by increasing from 3 mol % to 5 mol% of Fe at 5 mol % Al kept constant. These results have been confirmed by the transmission electron microscopy (TEM) studies. The crystalline size of the samples from TEM is 30-50 nm, which is in good agreement with the XRD results. The Raman studies reveal that the obtained peaks 300, 478, 633,779 cm^{-1} are good agreement with standard peaks of rutile phase SnO_2. The SEM micro graphs of the (Fe, Al) co-doped nanoparticles result in agglomeration with near spherical shape. The EDAX Spectra show the chemical composition of samples to be uniform. UV-VIS diffusion reflectance spectroscopy (DRS) studies show that the band gap energy is 3.7 eV for pure, and 3.6 and 3.5 eV for (Fe, Al) co-doped (3 mol% and 5 mol %) SnO_2 nanoparticles respectively. Photoluminescence (PL) has been studied with excitation wavelength of 350 nm. The un-doped sample exhibit one broad emission peak at 449 nm and relatively sharp emission peaks at 491 nm and 500 nm respectively. Co-doped SnO_2 samples also exhibit the broad emission peaks at 442 nm and 444 nm with decreasing intensity. The magnetic measurements reveal that all pure and co-doped nanoparticles exhibit room temperature Ferromagnetism (RTFM) and weak Ferro magnetism due to lattice defects such as O-vacancies and play an important role in activating the observed ferromagnetism based on the bound polaron model (BMP). As a result weak Ferromagnetism and Ferromagnetism is developed.

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Keywords: SnO2 nanoparticles, XRD, PL, TEM, RTFM.

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1. Introduction

Transition metal oxide nano materials such as SnO_2, TiO_2, ZnO and WO_3 possess good research interests in Nanomaterials. Among those SnO_2 have been attracted special interest due to their unique physical and chemical properties. The SnO_2 is an n-type semiconductor with wide band gap (E_g = 3.6 eV) and has large excitation binding energy (130 m eV) [1]. The novel potential applications of SnO_2 are due to the existence of intrinsic defects in its structural and optical properties. It has been widely used in many fields such as gas sensors, catalytic oxidation, solar cells, transparent conducting electrodes and photo catalysis resulting in outstanding structural and optical properties accompanied with mechanical and chemical stability [2]. Many methods of synthesis of SnO_2nanopowders such as hydrothermal [3], chemical vapor deposition (CVD) [4], sol-gel [5], chemical co-precipitation [6] and spray pyrolysis have been developed [7]. The SnO_2 evinces interest because it is a naturally non-stoichiometric prototype transparent conducting oxide (TCO). It has high transparency in visible region and high reflectivity in IR region. SnO_2 nanoparticles have been successfully doped with transition metal ions (Fe, Co, Mn, Cu) [8-10] and rare earth ions (Tb^{3+}, Eu^{3+}, Ce^{3+}) [11-14]. Thus the properties of impurity doped SnO_2 nanoparticles depend on the impurity position and processing method. Recent studies show that the co-doping of different TM ionssuch as Fe and Co co-doping [15] and Fe and Mn co-doping [16] is effective to enhance the saturation magnetization (M_s) With this motivation, we focus on Fe and Al co-doping into SnO_2 in order to elucidate the advantages of two TM ions. Both Fe and Al co-doping into SnO_2 enhance sustainment of the magnetic properties as ferromagnetic oxides.

In this paper, (Fe, Al) co-doped SnO_2 nanoparticles are synthesized and the structural, morphological, optical and magnetic properties are investigated by X-ray diffraction (XRD), Scanning Electron Microscopy(SEM), Transition electron microscopy (TEM), UV-VIS DRS, photoluminesces Spectroscopy and Vibration sample magnetometer (VSM).

2. Experimental Procedure:

2.1. Synthesis of SnO_2 nanoparticles

The pure and (Fe, Al) co-doped with SnO_2nanopowders are prepared by chemical co-precipitation method. $SnCl_2$ $2H_2O$ is synthesized using analytical grade Fe $(NO_3)_2$, $AlCl_3$ and aqueous NH_4OH as reactants. $SnCl_2$ $2H_2O$ is dissolved in de-ionized water to make 0.2M solution, and $Fe(NO_3)_2$ (3 mol% and 5 mol %) and $AlCl_3$ (5 mol % as constant) are co-doped into the above solution under vigorous stirring, then aqueous NH_4OH is added in to the solution drop wise until the pH of the solution reaches to 9 under constant stirring. After the completion of reaction, the above solution is washed several times with de-ionized water to remove the unnecessary impurities formed during synthesis and filtered. The precipitate is dried at 80 ºC for 12 hours and then ground. Finally the resulting nano powders are annealed at 600ºC for 4 hours under ambient atmosphere.

2.2. Characterizations:

The prepared powders are carefully subjected to the following characterization studies. Powder XRD pattern is recorded on Bruker diffractometer within the 2θ range of 20 to 80º using CuK_{α} as X-ray source (λ= 1.53906 Å). The structure of pure and co-doped SnO_2 areanalyzed by Micro Raman Spectroscopy (LAB-RAM HR, HORIBA JOBIN-YVON Spectrophotometer). The surface morphology and chemical analysis of (Fe Al) co-doped SnO_2nanopowders are studied by SEM attached with Energy Dispersive Analysis of X- ray Spectra (EDAX) (model CARL - ZEISS EVOMA 15).Transmission Electron Microscopy (TEM) and selected area electron Diffraction (SAED) are recorded on a Technai G20-Stwin High Resolution Transmission Electron Microscope (HRTEM) using an accelerating voltage of 200 kV. The optical properties are analyzed by UV-VIS diffusion reflectance Spectroscopy using CARY 5E UV-VIS-NIR Spectrophotometer in the wavelength range 200-2500 nm. The energy band gap is evaluated using Kubella-Munk model. The room temperature photoluminescence (PL) studies are carried out with PL Spectrometer. Magnetic investigations at the room temperature are carried out using a VSM magnetometer (Quantum Design, PPMS) at 15×10^3gauss.