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Synthesize of SnO_2, $SnSnO_3$ and Sr_2SnO_4 nanopowders by solide state-reaction

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ABSTRACT

The synthesis of nanoparticles has become a highly developed field owing to the scientific and technological interest due to the structural peculiarities and unusual physical and chemical properties. The one step solid-state reaction technique was employed to prepare nanopowders of SnO_2, Sr_2SnO_4 and $SrSnO_3$ through different weight ratio of Sn : Sr. This technique was found to give spherical products of uniform size and shape in over 90% yields. The materials have a variety of applications in ceramic dielectrics, gas-sensing materials and battery electrode bodies. Characterization of species was carried out using X-ray diffraction, SEM and TGA analysis. Investigation of samples revealed that, agglomerations of well connected, small grains lumped together with large number of pores have been formed. The XRD pattern analysis has shown that the change in crystalline sizes after calcining at various temperatures.

KEYWORDS

Technique;
Solid-state;
Nanopowders;
One step.

INTRODUCTION

The synthesis of nanoparticles has become a highly developed field owing to the scientific and technological interest due to the structural peculiarities and unusual physical and chemical properties they may lead to[1,2]. Nano-scaled particles are of great importance if the conformation of ceramics is considered; they have been found to enhance the mechanical, electrical, thermal, catalytic and optical properties of diverse ceramic materials[3,4]. Alkaline earth stannates have received more and more attention in recent years as components of ceramic dielectric elements[5]. Strontium stannate, $SrSnO_3$, has been reported to be used in humidity sensors[6]. Solid solutions of alkaline earth titanates and stannates are also used for the fabrication of ceramic boundary layer capacitors[7]. The phase equilibria in the SrO-SnO_2 system have been studied by several authors[8-10]. The existance of the two stable phases $SrSnO_3$ and Sr_2SnO_4 has been reported[8,9]. SnO_2, Sr_2SnO_4 and $SrSnO_3$ nanoparticles have been synthesized by soli state-reaction method. The produced sulfonamide nanoparticles were characterized by X-ray diffraction (XRD), infrared spectroscopy

(IR) and scanning electron microscope (SEM) and another techniques.

METHODS

Materials and general methods

$SnCl_4.5H_2O$, $Sr(OH)_2$ and $SrCl_2$ and solvents were purchased from Merck company. The infrared spectra of the compounds as KBr-disks were recorded in the range of 400–4000 cm^{-1} with a Mattson 1000 FT spectrometer. Powder X-ray diffraction (XRD) was carried out on a Philips diffractometer of X'pert Company with monochromatized Cu-K_α radiation. A multiwave ultrasonic generator (Sonicator_3000; Misonix, Inc., Farmingdale, NY, USA), equipped with a converter/transducer and titanium oscillator (horn), 12.5 mm in diameter, operating at 20 kHz with a maximum power output of 600 W.

EXPERIMENTAL

The powder of $SnCl_4.5H_2O$ (0.01 mol), $Sr(OH)_2$ (0.04 mol) and $SrCl_2$ (0.01 mol) were used as starting solid materials. The powders were weighed in three different preparative categories with Sn:Sr weghit ratio of 1:1, 1:2, and 2:1 to yield $SrSnO_3$, Sr_2SnO_4 and SnO_2, respectively. Each of this was ball-milled at room temperature for 30 minutes using zirconia's balls as a milling medium. According to relation (1), the mixing process accompanied by emission of water vapor from the surface:

$$SnCl_4, 5H_2O + 2\ Sr(OH)_2 \downarrow \rightarrow 2\ SrCl_2 + SnO_2, H_2O + 6\ H_2O \quad (1)$$

The product was washed, treated in an ultrasonic for ten minutes and then centrifuged (8000 rpm) for about 15 minutes. The yield was calcined at different temperature between 200-1000 °C. It was found that four hours calcinations led to better performance, where, the powder size of $SrSnO_3$, Sr_2SnO_4 and SnO_2 were obtained in the range of X-ray diffractometery (XRD) and the diameters of the resultant nanopowders were determined from the Scherrer's equation:

$$D = 0.89\lambda/\beta\cos\theta$$

The micro structural feature of nanopowders was studied by means of SEM. TGA of nanopowders was examined in O_2 at a heating rate 20 °C min^{-1} from room temperature to 1000 °C (Figure 1).

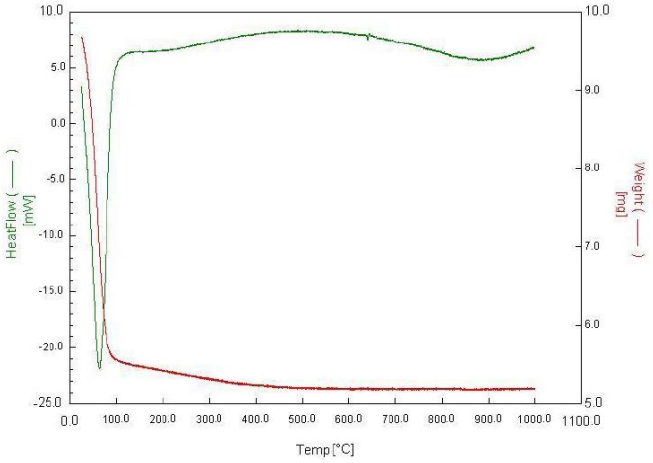

Figure 1 : TGA nanoparticles SnO_2

RESULTS AND DISCUSSION

The XRD pattern of nano tin oxide sample as prepared with 2:1 Sn to Sr ratio and calcined at temperature range of 200 °C to 1000 °C is given in figure 2.

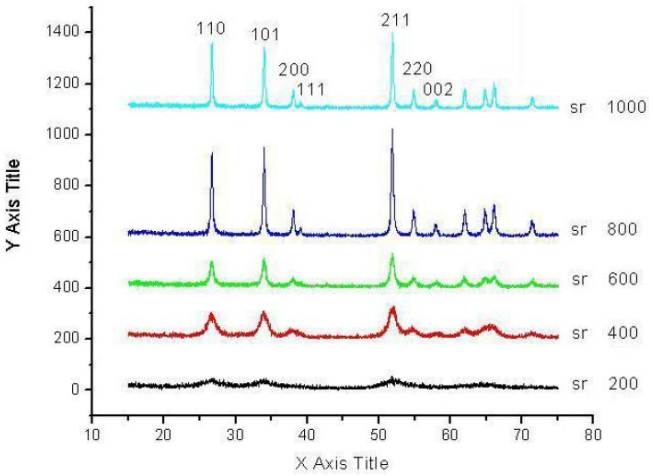

Figure 2 : X-ray diffraction patterns for nanoparticles SnO_2

TABLE 1 : Effect of calcinations temperature on powder size

Calcinations temperature (°C)	200	400	600	800	1000
d_{SnO2} (nm)	3.36	6.99	14.23	28.00	32.00
d_{SrSnO3} (nm)	16.59	35.60	49.80	66.35	66.37
$d_{Sr2SnO4}$ (nm)	14.00	24.00	39.00	49.80	36.17

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This figure also confirms phase purity and tetragonal rutile crystalline structure of SnO_2. The effect of calcinations temperature on powder size of the samples is summarized in TABLE 1.

The SEM image of SnO_2, sample is shown in figure 3. The agglomeration of 50-70 (nm) connected on grains lumped togeder with large number of pores could be observed.

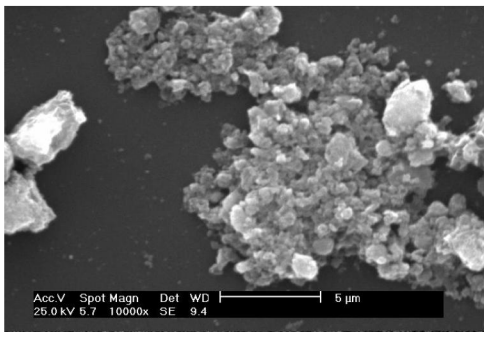

Figure 3 : SEM of SnO_2 prepared at 400 °C

The morphology of $SrSnO_3$ sample obtained via 1:1, Sn to Sr ratio is presented in figure 4. Deficiency of Sn with compare to SnO_2 sample is led to change particle shape from rutile to provskite structured. Coalescence of small grains together with their subsequent densification without much grain growth is seen this figure. It should be noted that due to loss of tin as volatile SnO_2 from the surface of the sample, the porosity of $SrSnO_3$, is interesting in the sense that, it contains well-connected porosity and still smaller grains. It has been documented that such structures with small crystalline size, well-formed intergranular connectivity and open channels are desired features of gas-sensing devices.

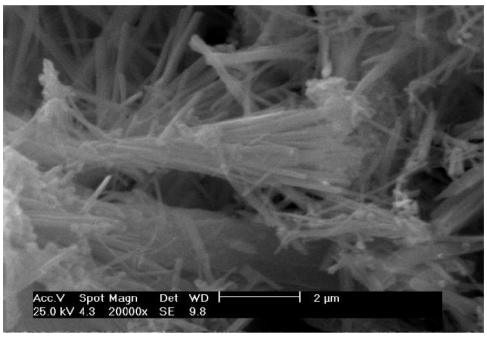

Figure 4 : SEM of $SrSnO_2$ prepared at 400 °C

Figure 5 shows the SEM micro structural images of Sr_2SnO_4 prepared by 1:2 Sn, Sr. Strong agglomeration accompanied with spherically shaped grains was formed. However, further geain growth was not seen.

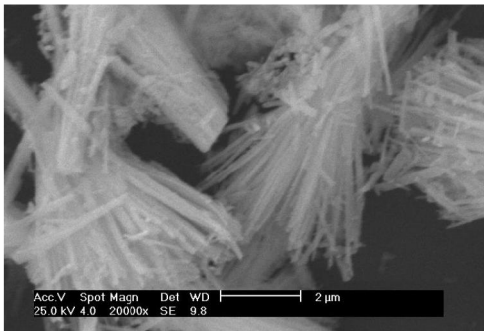

Figure 5 : SEM of Sr_2SnO_4 prepared at 400 °C

CONCLUSION

According to our observation, the $SrSnO_3$, Sr_2SnO_4 and SnO_2 powders obtained after calcinations (400 °C) revealed that the raw powder derived from solid-state reaction route were of very small (sub-micron) particles size.

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