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Processing of Tin Oxide Nanoparticles by Inert Gas Condensation Method and Characterization

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Abstract

Tin oxide nanoparticles (n-SnO and n-SnO_2) were synthesized by the inert gas condensation (IGC) method under dynamic gas flow of oxygen and argon at various conditions. Transmission electron microscopy (TEM) and X-ray diffraction (XRD) method were used to analysis the size, shape and crystal structure of the produced powders. The synthesized particles were mostly amorphous and their size increased with increasing the partial pressure of oxygen in the processing chamber. The particles also became broader in size when higher oxygen pressures were applied. Low temperature annealing at 320 ° C in air resulted to crystallization of the amorphous n-SnO particles to SnO_2.

Keywords : nanoparticles; tin oxide; inert gas condensation; oxygen partial pressure

1. Introduction

Tin oxide is a transparent conducting oxide, which has important applications such as gas sensing materials for reducing flammable gasses, ion-sensitive field effect transistors, and dye-sensitized solar cells [1, 2]. Recently, tin oxide nanoparticles have been synthesized and applied to sensor materials, as regard, higher surface are of nanoparticles improves the sensitivity of the sensors. Several methods are used to produce tin oxide powders, involving spray pyrolysis, pulsed laser ablation, chemical vapor deposition, inert gas condensation, rheotaxial growth and oxidation, sputtering, and sol-gel methods [3, 4]. Even though processing of nanoparticles in a dry and clean gaseous environment imposes a high production cost, the contamination of the nanoparticles can be minimized. Inert gas condensation (IGC) method has been widely used for processing of high purity metallic nanopowders [5]. Recently, the process has also been examined for production of metal oxides such as SnO [4, 6]. Within this work, the effects of oxygen partial pressure on the size, shape and structure of tin oxide nanoparticles synthesized by IGC method are studied.

2. Experimental and Results

Small amount of tin granulates with 99.9% purity was melted at 1400 ºC in a small tungsten boat after evacuating the chamber pressure to 10^{-4} mbar and filling with a high purity argon gas (99.99999%). Then, a mixture of oxygen and argon gas at various ratio was blown to the IGC chamber and the total pressure of the vessel was kept constant between 1-100 mbar using a rotary vacuum pump. The produced powder was examined by a high-resolution TEM (Phillips CM200) and XRD (X'pert, Phillips) equipment using a Cu anode.

Fig. 1 shows representative TEM pictures of the synthesized nanoparticles at the total pressure of 10 mbar. From these graphs, one may see the effect of oxygen partial pressure on the size and morphology of the particles. Apparently, the particles have spherical shape while they are slightly agglomerated. With increasing the oxygen partial pressure, coarser particles with broader distribution were produced. The particle size distribution was determined by image analyzing several of TEM pictures. The results are reported in Fig. 2. As it is seen, the mean particle size of the powders produced in the presence of oxygen is higher than that of pure tin (without oxygen blowing). The particles also become broader size distribution. However, under pure oxygen atmosphere, only slightly increase in the particle size was noticed.

Fig. 3a shows the XRD pattern of the powder produced at oxygen pressure of 5 mbar and the ratio of oxygen to argon of 1:1. As it is seen, the XRD peaks are very broad, but they are close to SnO. The peak broadening can be caused by the very small particle size (Fig. 1) and also due to the amorphous structure of the particles. Fig. 3b shows the SAD pattern of the particles taken under TEM. It appears that amorphous n-SnO nanoparticles were synthesized. When low temperature annealing in air was performed at 320 ºC for 24 hr, the XRD peaks revealed the formation of SnO_2 particles with relatively sharp peaks [7]. Therefore, it can be deduced that annealing in air resulted in oxidation of amorphous n-SnO to crystalline n-SnO_2.

3. Summary

Tin oxide nanoparticles were synthesized by the inert gas condensation (IGC) method. The results of TEM study

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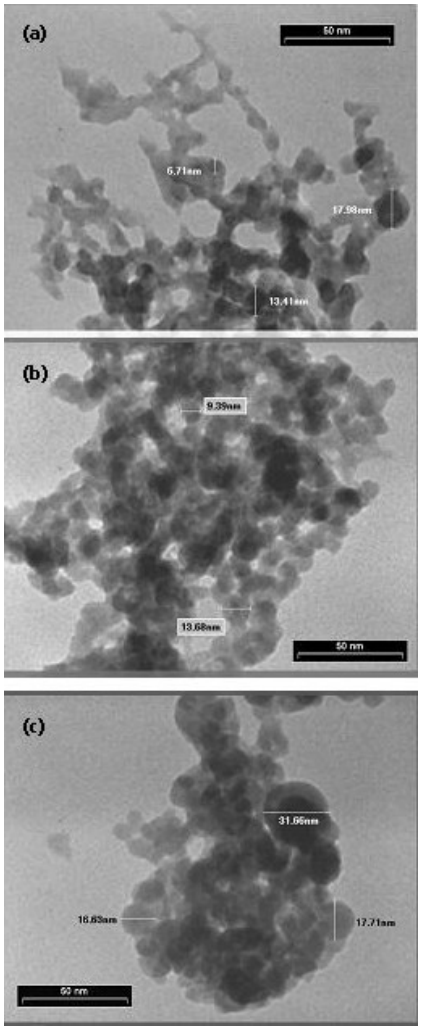


Fig. 1. TEM pictures of synthesized nanoparticles at oxygen ial pressure (mbar) of 0 (a), 5 (b), and 10 (c).

showed that the particles are almost spherical in shape with relatively narrow size distribution. The presence of oxygen in the IGC chamber increases the average particle size of the tin oxide powder and broader the particle distribution.

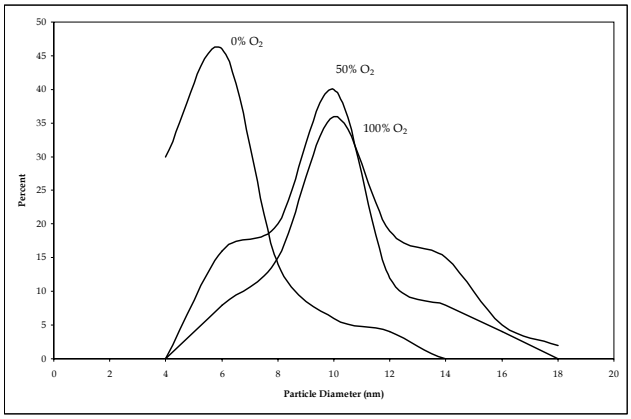
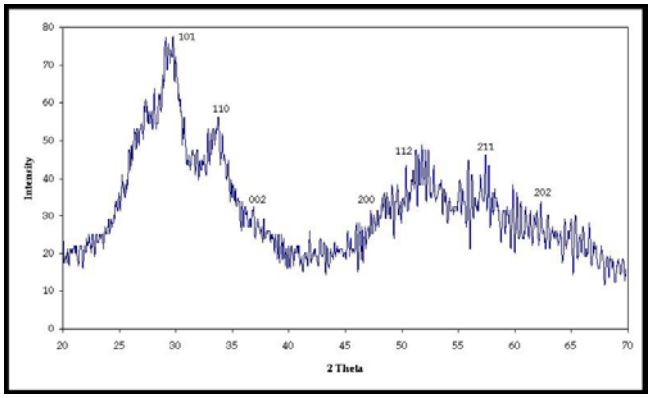
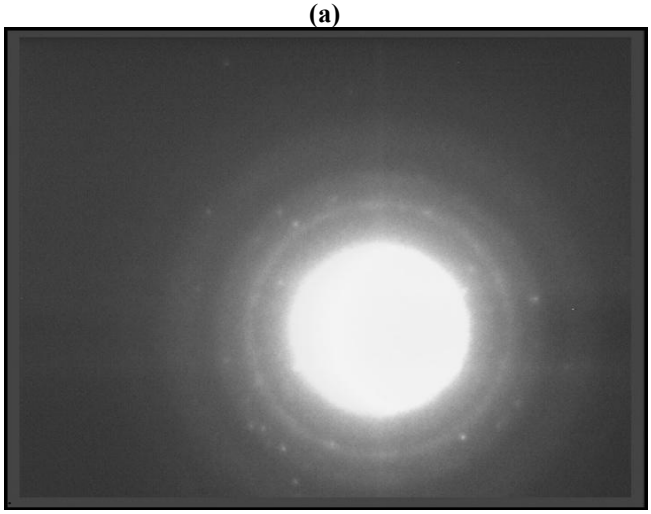


Fig. 2. Efect of oxygen partial pressure on the particle size distribution of tin oxide nanopowder.



(a)



(b)

Fig. 3. XRD (a) and SAD (b) patterns of tin oxide nanoparticles produced at 5 mbar oxygen partial pressure.

The X-ray pattern of the particles revealed that amorphous n-SnO was synthesized. Meanwhile, it was found that low temperature annealing at 320 ºC in air transforms the amorphous n-SnO to crystalline n-SnO_2.

4. References

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