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Fe-DOPED SnO_2 NANOPOWDERS OBTAINED BY MECHANOCHEMICAL ALLOYING AND THERMAL TREATMENT OF $SnCl_2$

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Abstract – The effects of milling time and starting Sn reactant on the degree of purity of $Sn_{1-x}Fe_xO_2$ (x=0, 0.05, and 0.08) nanopowders obtained by mechanochemical alloying and thermal treatment have been investigated. Results suggested that 3 hours of milling, instead of 12 hours, and the use of anhydrous $SnCl_2$, instead of $SnCl_2{\cdot}2H_2O$, are proper conditions to produce impurity free samples. The oxidation states for iron and tin ions were of 3+ and 4+ respectively, both occupying octahedral sites, thus suggesting that iron replaced tin.

The search for proper conditions to prepare impurity free samples by using mechanochemical alloying is an important subject in materials science. Particularly, the preparation of pure transition metal doped tin oxide is receiving growing attention, because of its potential use as a spintronic like material. $Sn_{1-x}Fe_xO_2$ (x=0, 0.05, and 0.08) nanoparticles were synthesized by mechanochemical alloying of anhydrous $SnCl_2$ (or $SnCl_2{\cdot}2H_2O$), $FeCl_3$ and Na_2CO_3, with NaCl added as diluent, followed by a thermal treatment. The mechanochemical processes was performed in a planetary ball mill Fritsch Pulverisette 5, using Cr-based stainless steel jars and balls of 12 mm in diameter. The rotation velocity of the disc was of 250 rpm, and the ball to powder ratio was of 20:1. Samples were milled for 3 and 12 hours in atmospheric conditions. The as-milled mixtures were subsequently heat treated at 600 °C during 3 hours, in air atmosphere, and finally washed with double deionized water to remove the NaCl. The purpose of the NaCl diluent is to improve the milling characteristics of the reactant powders [1]. The overall reaction is accounted for by: $SnCl_2 + Na_2CO_3 \rightarrow SnO + 2NaCl + CO_2$, and the heat treatment of the milled-product in oxygen is followed in order to oxidize SnO to SnO_2 [2]. The X-ray diffraction patterns of $Sn_{1-x}Fe_xO_2$ samples showed only peaks due to the cassiterite phase of SnO_2 for the samples obtained for milling time of 3 h (Fig. 1). No impurity phases were detected. In contrast, the presence of hematite for samples obtained for 12 h milling was observed. The ^{57}Fe Mössbauer spectra of $Sn_{1-x}Fe_xO_2$ samples milled for 12 h were composed of magnetic and paramagnetic signals, whereas for samples milled for 3 h, only doublets were observed. The ^{119}Sn Mossbauer spectra for all samples indicated only the presence of Sn^{4+} in octahedral sites (see Fig. 2). In contrast, the sample obtained by using $SnCl_2{\cdot}2H_2O$, showed the presence of hematite.

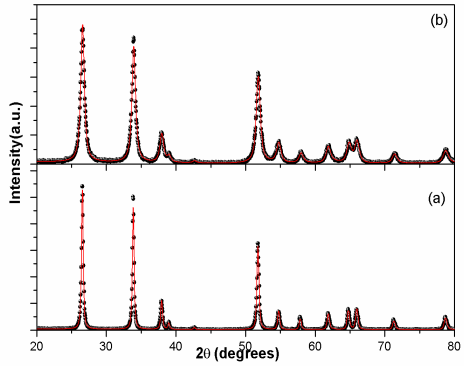

Figure 1: XRD patterns of $Sn_{1-x}Fe_xO_2$, with **a**) x=0 and **b)** x=0.08. The samples were milled for 3h. Solid lines are fits using the Rietveld method.

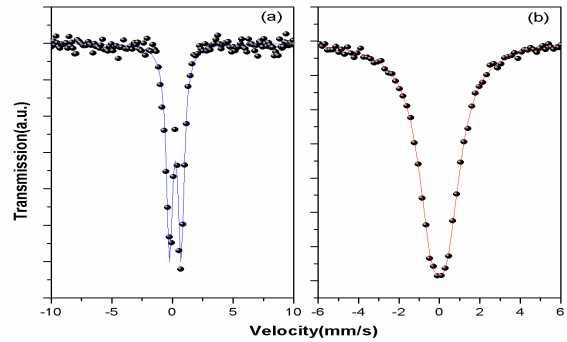

Figure 2: a) ^{57}Fe and **b)** ^{119}Sn room temperature Mössbauer spectra of sample $Sn_{0.92}Fe_{0.08}O_2$. The sample was milled for 3h.

[1] A. Dodd, A. McKinley, M. Saunders, and T. Tsuzuki, Nanotechnology 17 (2006) 692.
[2] H.M. Yang, Y.H. Hu, A.D. Tang, S.M. Jin, G.Z. Qiu, J. Alloys Comp. 363 (2004) 271.