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Development and characterization of thin films for application as ethanol sensors

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Abstract – SnO_2:Sb have been studied due to their important technological applications, including as gas sensors. This work reports the preparation of SnO_2 thin films doped with 5, 10 and 14 mol% Sb. Precursor solution was obtained by Polymeric Precursor method, and films were deposited by spin-coating. XRD revealed that films aren't presents additional phases, and images obtained by FEG shown spheric and nanometric grains. These properties are important for they allow the increase of the electric conductivity of the material. This way, they present potential for futures sensor tests.

Gases sensors based in semiconductors oxides presents change of resistance when exposed in some gases atmosphere. SnO_2 is a material used in some gas sensors. A sensibility of this material is due a reaction of oxygen chemisorbed in surface with gases as NO, ethanol and others. To improve efficiency, SnO_2 can be doped with other cations, for example, Pd, Nb, Sb and others. The literature report that Sb dopant in SnO_2 lattice impedes particle growth and resulting in a significant increase of the specific surface area [1]. For these reasons, Sb-doping has been used on the SnO_2 sensors in order to improve their conductivity and sensibility of the sensor. Besides, the effect of surface microstructure and dopant concentration is an important factor in sensor properties, as sensitivity, selectivity, response and recovery in the presence of gases. These factors need to study in details.

The present work reports the development and characterization of SnO_2 thin films pure and doped with 5, 10 and 14 mol% Sb_2O_3. These films were obtained by spin-coating of a polymeric precursor solution [2]. The Polymeric Precursor method consists in the polymerization of a metallic citrate with ethylene glycol. Tin chloride, citric acid and ethylene glycol was mixed in molar ration 1:3:1. Then, Sb_2O_3 was dissolved in nitric acid and added in solution. The mixture was heated at ~70°C to promote a polymerization reaction.

The viscosity of the solution was adjusted in 28cP, by the addition of absolute ethyl alcohol. To obtain thin films, the solution was deposited by the spin-coating technique onto glass. The rotation velocity and time were fixed in four steps, as follows: 500rpm / 20 s; 1000rpm / 20 s; 2500rpm / 20 s and 5000rpm / 15 s. After deposition, the films were dried on a hot plate (~100°C) for 2 hours, followed by a two-stage heat treatment: 300°C with a heating rate of 1°C/min during 4 h, and 450°C with a rate of 5°C/min, during 8 h. The crystalline structure of the films was studied by means of X-ray diffraction (XRD), and morphology by field-emission gun scanning electron microscope (FEG-SEM).

Diffractograms of SnO_2:Sb thin films, with different molar ratio of Sb, show Bragg re. ections belonging to the SnO_2:Sb polycrystalline phase. The films aren't trace of additional phases and we can assume that antimony formed at least a partial solid solution with SnO_2. This is a good result, because futures sensor tests will be accomplished by electric measures, and the presence of other phases results in higher electrical resistivity values, due to a decrease of the electronic mobility and an increase on the quantity of interfaces between the Sb_2O_3 and SnO_2 phases. FEG images reveled a spherical-type grains, with nanometric average grain size close. SnO_2 film present grain size of ~20 nm, while SnO_2:Sb films present grain size of ~13 nm. This decrease of grain size could be attributed at Sb substitution on the SnO_2 lattice, should increase the stresses in this matrix and therefore reduce the grain growth. The cross section images showed a well packed film, and the thickness of the films presents ~500 nm. These results came promising, taking to futures studies as application of these films as ethanol sensor, through accomplishments of electric measures, and verification of the conductivity variation and sensibility of the films.

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