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Report:

Sn-doped In_2O_3, also known as ITO for indium-tin oxide is an n-type transparent conducting oxide extensively used in industrial applications. The desirable electrical properties of ITO depend on its microstructure, defect structure and Sn-doping level. The solubility limits of Sn in In_2O_3 have been reported by various authors at different temperatures but their results disagree [1-11].

For this *in situ* experiment, we had proposed to use single phase, nano-ITO powders with overdoped Sn concentrations to study the evolution kinetics of SnO_2 as a function of annealing time in the temperature range 1000 ^{o}C to 1300 ^{o}C. From the initial Sn doping content in ITO and the final fraction of precipitated SnO_2 phase, the Sn solubility limit in bixbyite In_2O_3 can be inferred for a particular annealing temperature. The diffraction experiments were performed at 90 keV with an x-ray beam size of 0.2 x 0.2 mm. The nano-ITO powders were contained in quartz capillaries of 1 mm in diameter with a wall thickness of 0.01 mm. A furnace heated the sample in the quartz capillary while a MAR image plate collected *in situ* 2D diffraction images up to a d-spacing of 1.1 Å. Because quartz undergoes a phase transition resulting in deformation and softening at ~1050 ^{o}C, the quartz capillaries could only be used to perform the *in situ* annealing experiment at 1000 ^{o}C. The 2D images were integrated to obtain x-ray diffraction patterns that were then analyzed with the Rietveld method. The results included sample composition, structural and microstructural information for each of the existing phases.

A region of the x-ray diffraction pattern is found in Figure 1 showing the second strongest SnO_2 peak next to a weak ITO bixbyite reflection. The precipitation of the tetragonal SnO_2 phase can be observed as a function of annealing time at 1000 ^{o}C. From the initial composition of the nano-ITO powder, the solubility limit of tin in In_2O_3 at 1000 ^{o}C is estimated to be less than one

cation percent. Using the Lorentzian and Gaussian broadening of the diffraction peaks, the grain size and strain of the phases were calculated. The grain size of bixbyite ITO and tetragonal SnO_2 are presented in Figure 2.
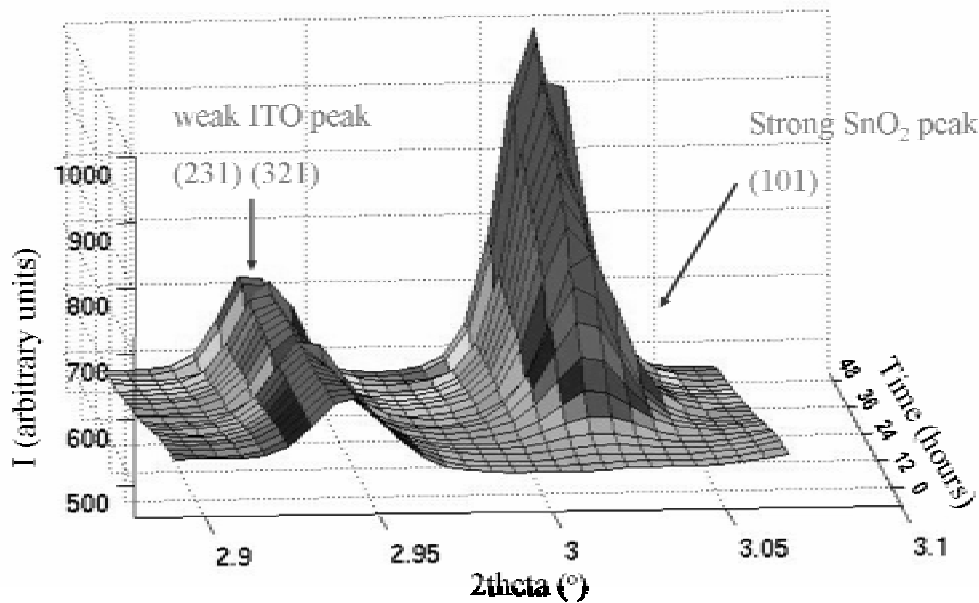

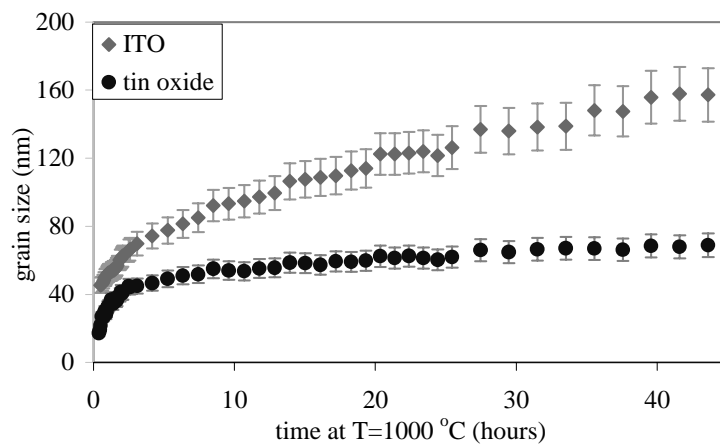Figure 1. Precipitation of SnO_2 as a function of annealing time at 1000 ^{o}C

Figure 2. Grain size of bixbyite ITO and tetragonal SnO_2 during isothermal annealing at 1000 ^{o}C

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