	Experiment title: Studies on the In_2O_3-SnO_2 phase diagram	**Experiment number**: ME-904
Beamline: ID 15B	**Date of experiment**: from: 08/09/2004 to: 14/09/2004	**Date of report**: 23/11/2006
Shifts: 18	**Local contact(s)**: Gabriela GONZALEZ AVILES	*Received at ESRF:*

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Report:

In_2O_3, SnO_2 and Sn-doped In_2O_3 materials are transparent in the visible region and also exhibit electrical transport properties which are desirable for several commercial applications. The appealing electrical and optical behavior of these materials depend on their microstructure and defect structure. Since the electrical properties of In_2O_3 are enhaced when doped with Sn, knowing the solubility of tin in indium oxide is of great interest and has been studied by various authors. Nevertheless, there is disagreeement on the equilibrated solubility limits of Sn in In_2O_3 [1-11].

As in experiment ME-789, we proposed to anneal nano-ITO powders with overdoped Sn concentrations at temperatures ranging from 1000 ^oC to 1300 ^oC. The aim was to study *in situ* the precipiation kinetics and grain growth of ITO and SnO_2. Since we learned during ME-789 that quartz capillaries could not be used at temperatures higher than 1050 ^oC, due to a softening phase transition, we proposed to contain the powders in platinum tubes. The platinum tubes had a wall thickness of 0.04 mm and an exterior diameter of 0.65 mm. A furnace heated the sample while a MAR image plate collected *in situ* 2D diffraction images up to a d-spacing of 1.1 Å. The experiment was done using an x-ray energy of 90 keV in order to have deep penetration into the sample cell. Even at such high-energy, the absorption of platinum was very high. The diffracted signal coming from the ITO sample was significantly weaker than that of platinum, and we were not able to perform high-quality fits to the data. Therefore, we decided to remove the need of a sample container by pressing pellets with the nano-ITO powders. With this method, the diffraction patterns come only from the sample and they are stable at high-temperatures. We then performed an isothermal heating experiment at 1200 ^oC on a pellet sample. The diffraction 2D images were integrated and the resulting one-dimensional diffraction patterns were fitted using the Rietveld method.

An example of a Rietveld refinement is shown in Figure 1, where the sample has been annealed for 24 hours at 1200 ^{o}C and is biphasic. Figure 2 summarizes the precipitation behavior of the tetragonal SnO_2 phase as a function of annealing time at 1200 ^{o}C.

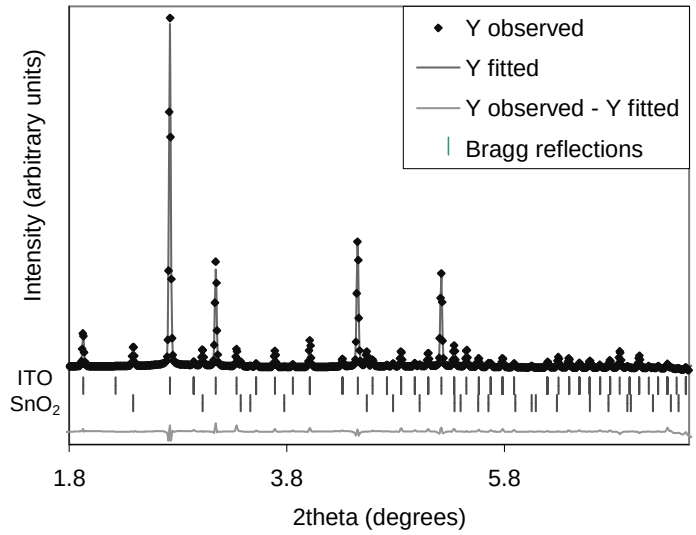

Figure 1. Rietveld refinement of an x-ray diffraction pattern after the sample had been annealed for 24 hours at 1200 ^{o}C

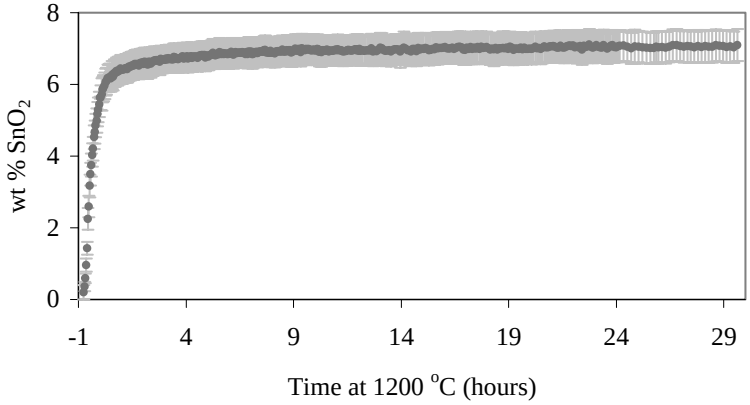

Figure 2. Fraction of precipitated SnO_2 as a function of annealing time at 1200 ^{o}C

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