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Preparation of $In(OH)_3$ and In_2O_3 by the Microwave-Hydrothermal Method

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Abstract – In this paper, we report the preparation of crystalline $In(OH)_3$ single-phase by microwave-hydrothermal method used as precursor for the In_2O_3 synthesis, oxide obtained after heating using a conventional furnace (CF) and a domestic microwave oven (MO). The crystalline $In(OH)_3$ precursor was added into autoclave system and treated at 140 °C for 1 h, using a heating rate fixed of 25 °C/min. The powders were structurally characterized using X-ray diffraction (XRD), Raman spectroscopy and Field–Emission Scanning Electron Microscopy (FEG-SEM). The In_2O_3 single-phase was obtained for 5 min at 500 °C in a MO.

Indium oxide (In_2O_3) is a good n-type semiconductor with a direct band gap of 3.55–3.75 eV and has been widely in opto-eletronics industry devices. The hydroxide indium ($In(OH)_3$) is also an important semiconductor with a wide band gap (Eg) estimated in 5.15 eV. The physical and chemical properties of these nano or micron-sized powders are due to shape, size and size distribution of the particles, which depend on the characteristics of methods utilized [1-3]. In this paper, we report the synthesis of crystallines $In(OH)_3$ by microwave-hydrothermal and In_2O_3 in a conventional furnace (CF) and in a domestic microwave oven (MO). The reaction system was heat treated at 140 °C for 1 h, using a heating rate fixed at 25 °C/min. The resultant white precipitates were collected and dried in room temperature. The powders obtained were heat treated at 500 °C for 1h with a heating rate of 10 °C/min in a conventional furnace (CF) and for 5 minutes with a heating rate of 25 °C/min in a domestic microwave oven (MO). Fig. 1 shows the XRD patterns of the as-prepared products using microwave-hydrothermal process: (a) $In(OH)_3$ and (b) In_2O_3 calcined by: (i) (CF-1 hour) and (ii) (MO-5 min). X-ray diffraction of the $In(OH)_3$ powder (Fig. 1a) confirmed reflections of a crystalline single-phase. The experimental lattice constant was a = 7.96(4) Å, is in good agreement with (7.98 Å) from the standard card (JCPDS No. 85-1338). After annealing (Fig. 1b) at 500 °C, the XRD patterns showed all the peaks indexed as a single cubic phase with lattice constant: (i) 10.10(4) Å and (ii) 10.11(4) Å. These values are in according to (10.12 Å) the reported in the standard card (JCPDS No. 71-2194). Three Raman modes were observed in the region of 309, 358 and 392 cm^{-1} to $In(OH)_3$ and in the region of 132, 307, 367 and 495 cm^{-1} to In_2O_3. The Raman peak at ~307 cm^{-1} corresponds to the In-O vibrations of InO_6 structural units and is very sensitive to presence of oxygen vacancies. The size and morphology of the as-prepared product were investigated using Field–Emission Scanning Electron Microscopy (FEG-SEM). The In_2O_3 microcubes was obtained from of $In(OH)_3$ precursor annealing (Fig. 2) with the same morphology as was ascribed to chemical dehydration: $2In(OH)_3 \rightarrow In_2O_3 + 3H_2O$

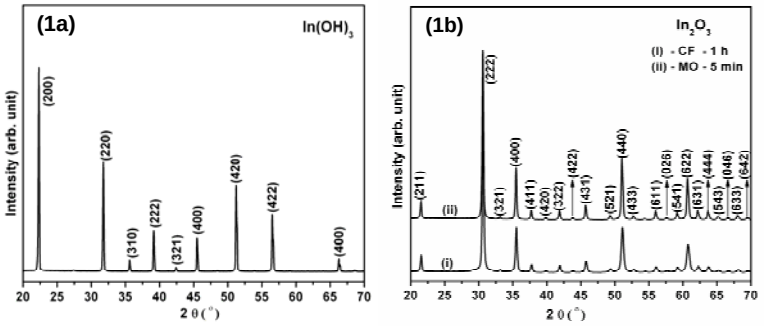

Figure 1. XRD patterns (a) $In(OH)_3$ as-prepared by microwave-hydrothermal process and (b) In_2O_3 annealed by: (i) (CF - 1 h) and (ii) (MO – 5 min).

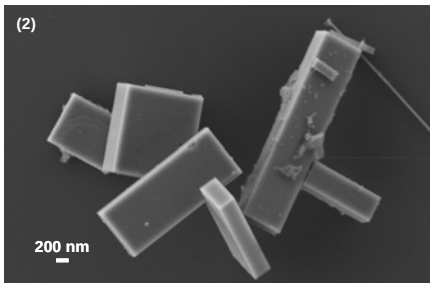

Figure 2. FEG-SEM micrograph of the $In(OH)_3$.

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